JOURNAL
OF THE
OIL AND COLOUR CHEMISTS' ASSOCIATION

Vol. 49 No. 8 **August 1966**

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Phenylmercury compounds as fungicides

New developments in the field of polyurethane lacquers

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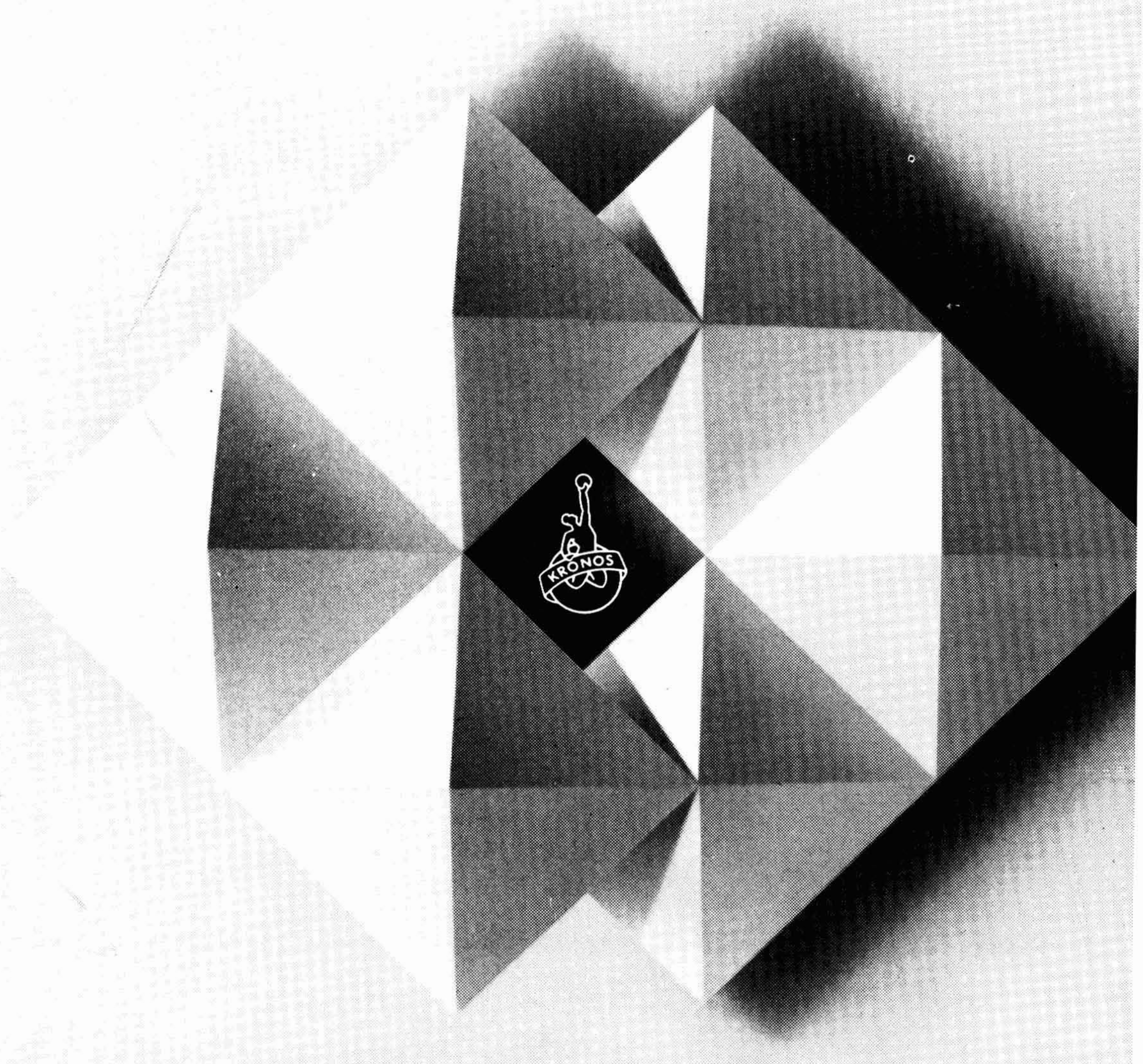
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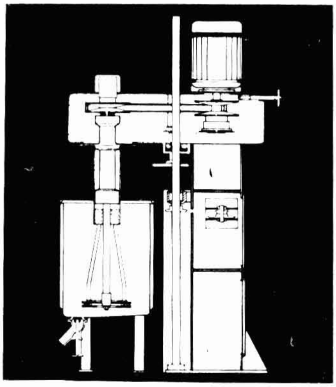

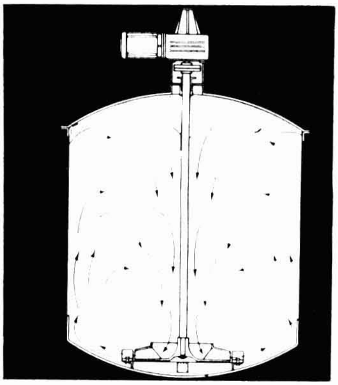

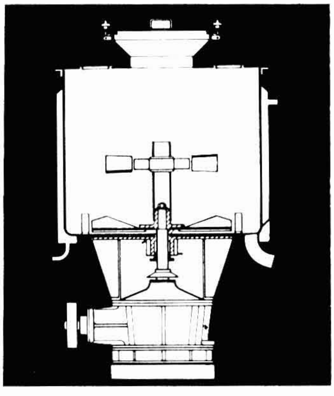

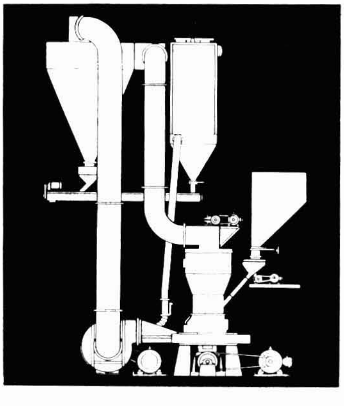

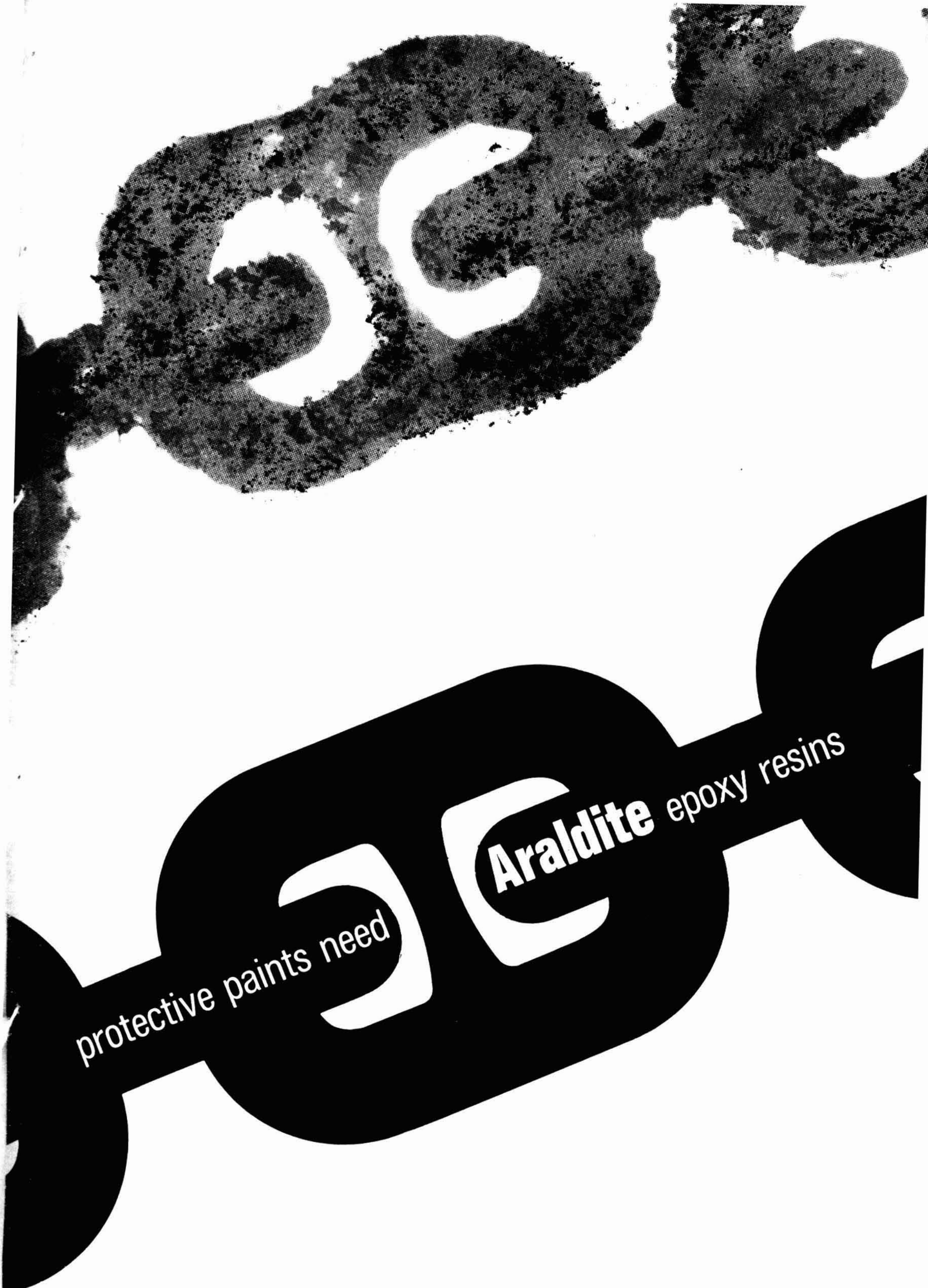
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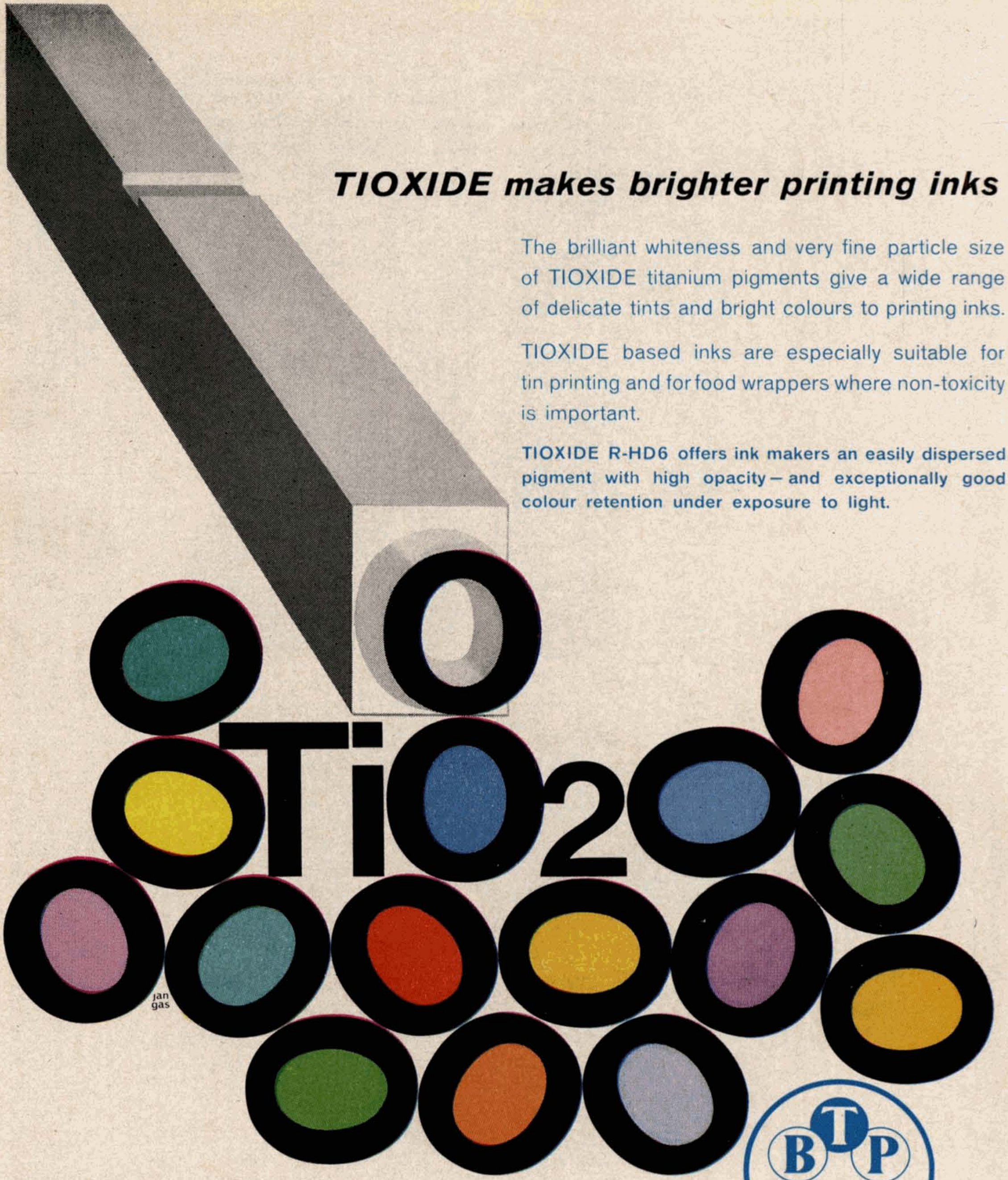
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Phthalocyanine pigments—their form and performance*

By F. M. Smith and J. D. Easton
Geigy (UK) Ltd., Simonsway, Manchester 22

Summary

The properties and preparation of various types of copper phthalocyanine pigments are described. Two types of " crystallisation " are recognised and shown to depend on the original crystal form of the pigment and on the rate of phase change. Pigment preparation may be by acid treatment or salt grinding ; the course of the latter process has been followed and mechanisms of producing α- and β-form pigments in this way are considered. A survey is made of flotation, flooding and flocculation and their control. A brief account is given of the physical and chemical factors affecting the properties of phthalocyanine green.

Pigments de Phtalocyanine—Leur Forme et Leur Rendement

Résumé

On décrit les propriétés et la préparation des types divers de pigments de phtalocyanine de cuivre. On reconnait deux types de " cristallisation " que l'on démontre se dépendent également de la forme cristalline originale du pigment et du taux de changement de phase. La préparation du pigment s'effectue soit par traitement acidique soit par broyage à sel ordinaire. Le progrès de ce dernier procédé fut suivi et quelques mécanismes pour produire en cette manière les formes α et β sont considérés. On examine ces pigments au point de vue de flotation, flocculation, et séparation du solde d'une peinture ainsi que le contrôle de ces défauts. Une description assez brève est fournie des facteurs physiques et chimiques qui affectent les propriétés du phtalocyanine vert.

Phthalocyaninpigmente—ihre Form und ihr Verhalten

Zusammenfassung

Die Eigenschaften und Herstellung verschiedener Arten von Kupferphthalocyaninpigmenten werden beschrieben. Zwei Arten von " Kristallbildung " sind erkennbar und, wie gezeigt wird, sind diese von der ursprünglichen Kristallform des Pigmentes und der Geschwindigkeit, mit der die Phase geändert wird, abhängig. Die Darstellung der Pigmente kann durch Säurebehandlung oder Salz-Vermahlung erfolgen ; der Verlauf des letztgenannten Verfahrens wurde verfolgt, und es werden Betrachtungen über den Darstellungsmechanismus von auf diese Weise gebildeten α- und β-Formen der Pigmente angestellt. Die Phenomena des fleckigen Aufschwimmens (flotation) und des einfarbigen Aufschwimmens (flooding), sowie des Ausflockens und deren Bekämpfung werden besprochen. Die physikalischen und chemischen Faktoren, welche die Eigenschaften von Phthalocyaningrün beeinflussen, werden kurz aufgezählt.

Фталоцианиновые грунтовые краски—их форма и действие

Резюме

Описываются свойства и подготовка различных типов грунтовых красок на базе фталоцианина меди. Два типа кристаллизации отмечаются, которые зависят от первоначальной кристаллической формы грунтовой краски и от скорости фазового изменения. Подготовку можно осуществить обработкой кислотой или соляным растиранием; последний процесс изучался и обсуждены механизмы получения α–и β–форм этих грунтовых красок. Сделан обзор флотации, неравномерного окрашивания и флоккуляции и их контроля. Дается краткое описание физических и химических факторов которые влияют на свойства зелёной грунтовой краски на базе фталоцианина.

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Introduction

It is one of the fundamental facts of pigment technology that the same chemical compound can give rise to a number of pigments differing in shade, strength and other aspects of behaviour in use. These differences are ascribed to the physical condition of the product, in particular to particle size and shape, and the state of its surface ; the technology of pigment manufacture is largely a matter of controlling these. Until recently this has been done empirically, but as knowledge increases it becomes easier to control results on a scientific basis. More work has probably been done on phthalocyanine pigments than on any other organic pigments and it is the object of this paper to use them to illustrate the importance of physical factors and methods in pigment processing.

The phthalocyanines occupy a unique place in pigment technology for they cover a shade range from blue to green where there are no other pigments with the same outstanding properties of brightness and strength combined with fastness to light, heat and chemicals. They have set a new standard of stability and consequently their manufacture is one of the fastest growing sectors of the industry. They find use in all organic pigment consuming industries such as paint, printing inks, plastics and mass coloured synthetic fibres.

Chemical types

Broadly speaking, there are four chemical types of phthalocyanines of pigmentary importance (Fig. 1). Copper phthalocyanine (a) shows outstanding stability, brightness and cleanliness of shade and is by far the most important member of the group ; so much so that phthalocyanine blue is practically

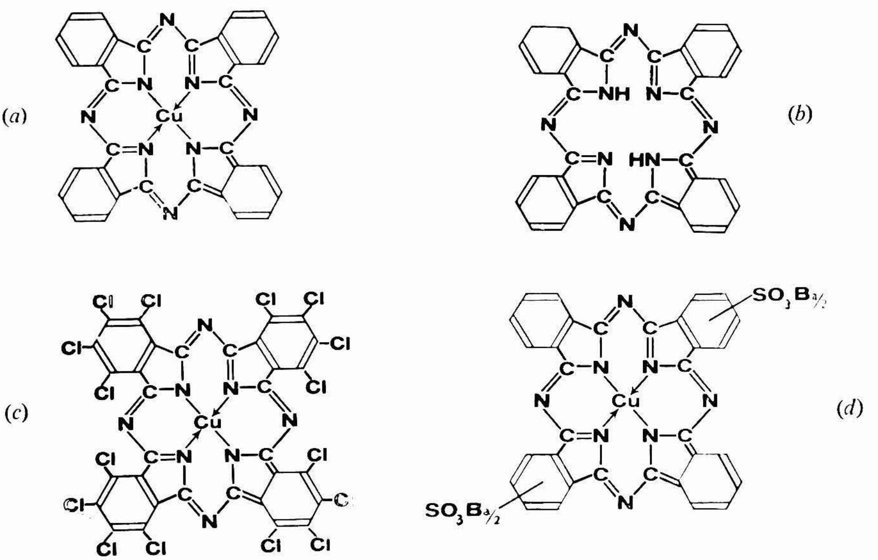


Fig. 1.

synonymous with copper phthalocyanine. Metal-free phthalocyanine (b) has a greener and somewhat duller shade and is of lesser importance as a pigment because of its higher cost and lower fastness properties. Substitution of halogen in the benzene rings of copper phthalocyanine gives a bright green pigment and at about 15 chlorine atoms per molecule gives the normal commercial phthalocyanine green (c). Ring substitution by a sulphonic acid group and formation of the heavy metal salt gives a blue lake pigment (d) which lacks the fastness to light and alkali of the parent compound. It found favour in the past since it was not subject to such defects as flocculation and crystallisation, but as these problems are being overcome the importance of this derivative is decreasing.

Distinction between these different compounds is not as easy as might be supposed, particularly if only a small portion of coloured material is available or if a mixture is present, and chemical methods as generally recommended are rather inadequate. In this situation infrared analysis has proved extremely useful. Very dilute solutions of phthalocyanines in concentrated sulphuric acid show characteristic sharp peaks just beyond the red end of the visible spectrum where there is little interference from other compounds and none from other organic pigments. These spectra (Fig. 2) are already known but their practical value seems so far to have escaped notice. The barium lake is not easily distinguished from copper phthalocyanine itself, but its alkali solubility is at once apparent. The shift in absorption maxima on replacement of the chlorine by bromine is worthy of note. This technique is of little quantitative value, but its speed and accuracy have made it very useful qualitatively.

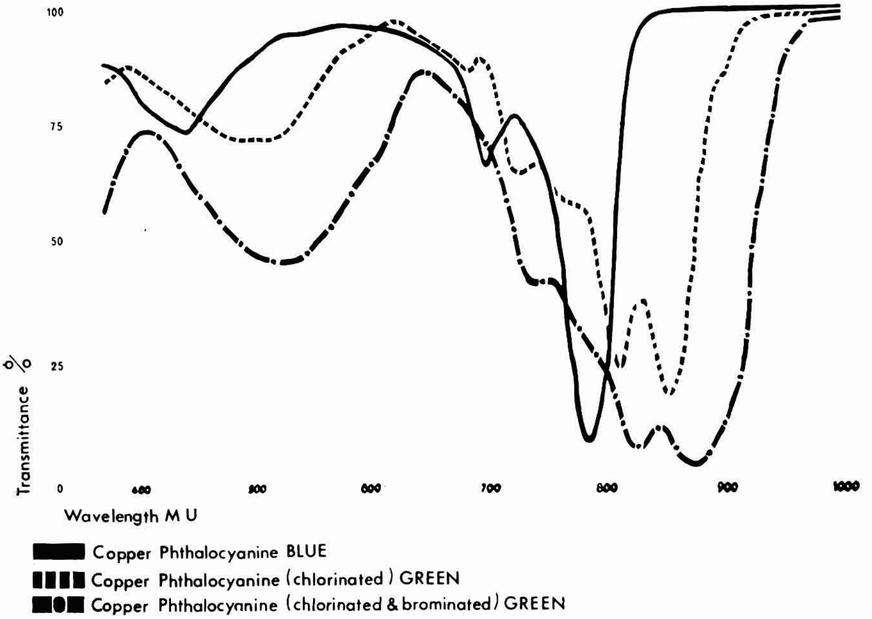


Fig. 2. Transmittance of phthalocyanine pigments in sulphuric acid solution

Polymorphism of copper phthalocyanine

Copper phthalocyanine is made commercially by two well-known processes starting from phthalic anhydride and phthalonitrile respectively. Both processes involve the use of high temperatures, possibly with organic solvents, and as a result the product is not immediately suitable for use as a pigment. It is necessary to bring it into the correct physical condition for pigmentary application and even for specific applications. Fundamental to this is the fact that copper phthalocyanine exists in several crystal forms, i.e. it exhibits polymorphism.

Different crystal forms are not merely differences of crystal shape, but arise from different arrangements of the molecules in the crystal lattice which give rise to differences in fundamental physical properties as well as in pigmentary performance. For example, their X-ray diffraction patterns and infrared spectra are quite different. Two forms of copper phthalocyanine designated α and β are of considerable practical importance ; other forms probably exist and will be mentioned later, but so far they have not appeared in commerce. The α-form is redder in shade, is almost always of very small particle size and tends to revert to the β-form under the action of heat and organic solvents ; thermodynamically the α-form is the less favoured. The β-form is greener in shade, particle size varying from very small which is suitable for pigmentary use, up to needles about 2 cm long ; this is the form favoured thermodynamically. The α- and β-forms are distinguished by their X-ray diffraction patterns and their infrared spectra and the proportions in mixtures can be quantitatively determined[1].

Two electron micrographs (Fig. 3) show the α- and β-forms as they are used as pigments. One notable feature is the remarkably small ultimate particle size in both cases—about one-fortieth of a micron—which is responsible for some of the characteristic properties of strength and transparency on one hand, and the tendency to poor flow properties on the other.

The mechanism of crystallisation defects

The well-known phenomenon of " crystallisation " of phthalocyanine blue derives from the existence of α- and β-forms. It can occur in many media, but is most often recognised as " can fading " of paints containing aromatic solvents. When such a paint is pigmented with unstabilised or " normal " α-form phthalocyanine blue and stored, particularly at tropical temperatures, colour strength is lost and microscopic examination (Fig. 4) shows that the pigment is transformed to comparatively large needle-like crystals. These are found to be of the β-form, and regrinding will not break them up and restore the original shade of the paint. This inability to regain an original strength on further grinding is vital in diagnosing the defect of crystallisation and distinguishing it from " flocculation " where the strength is restored by shearing forces, and from poor initial dispersion where the full colour strength was never developed.

The mechanism of crystallisation depends on the limited, but still significant solubility of copper phthalocyanine in solvents, particularly aromatic solvents. There is a dynamic equilibrium between the undissolved phthalocyanine

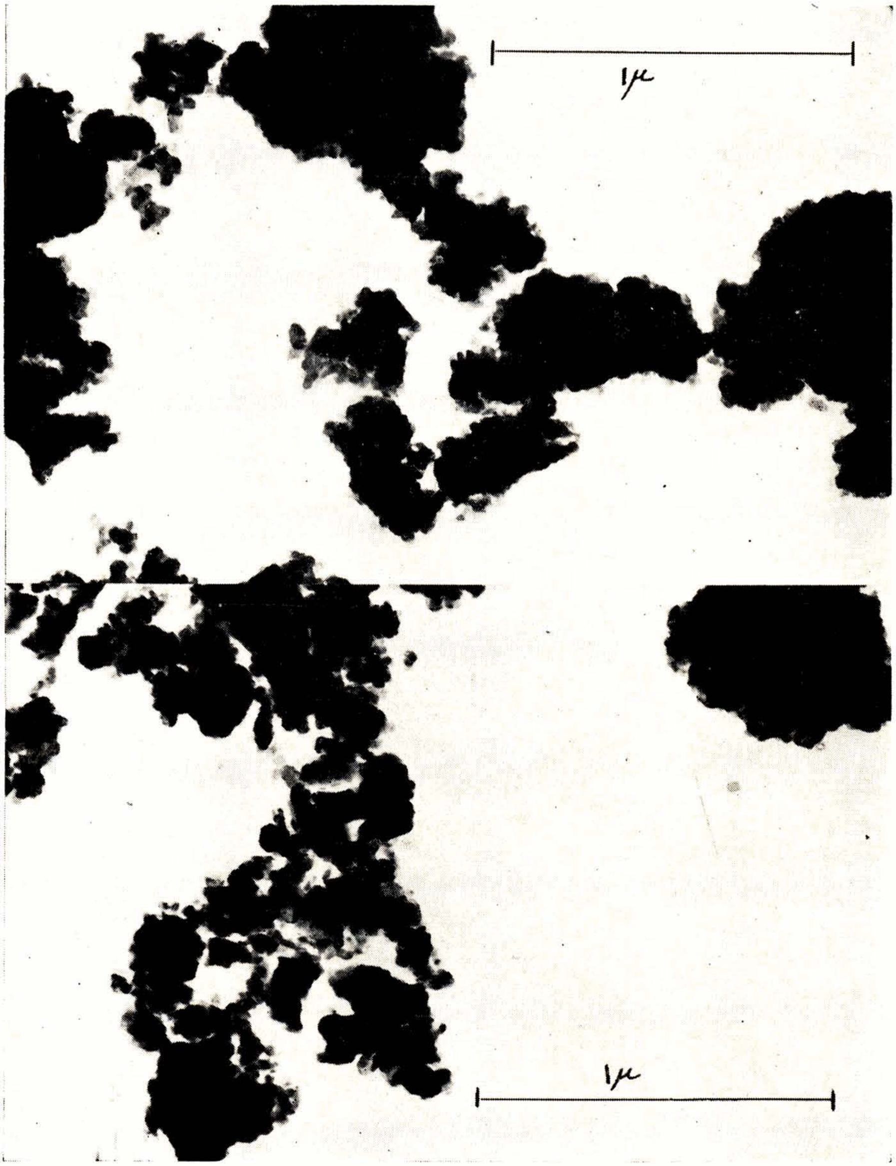


Fig. 3. α-form (top) β-form (bottom)

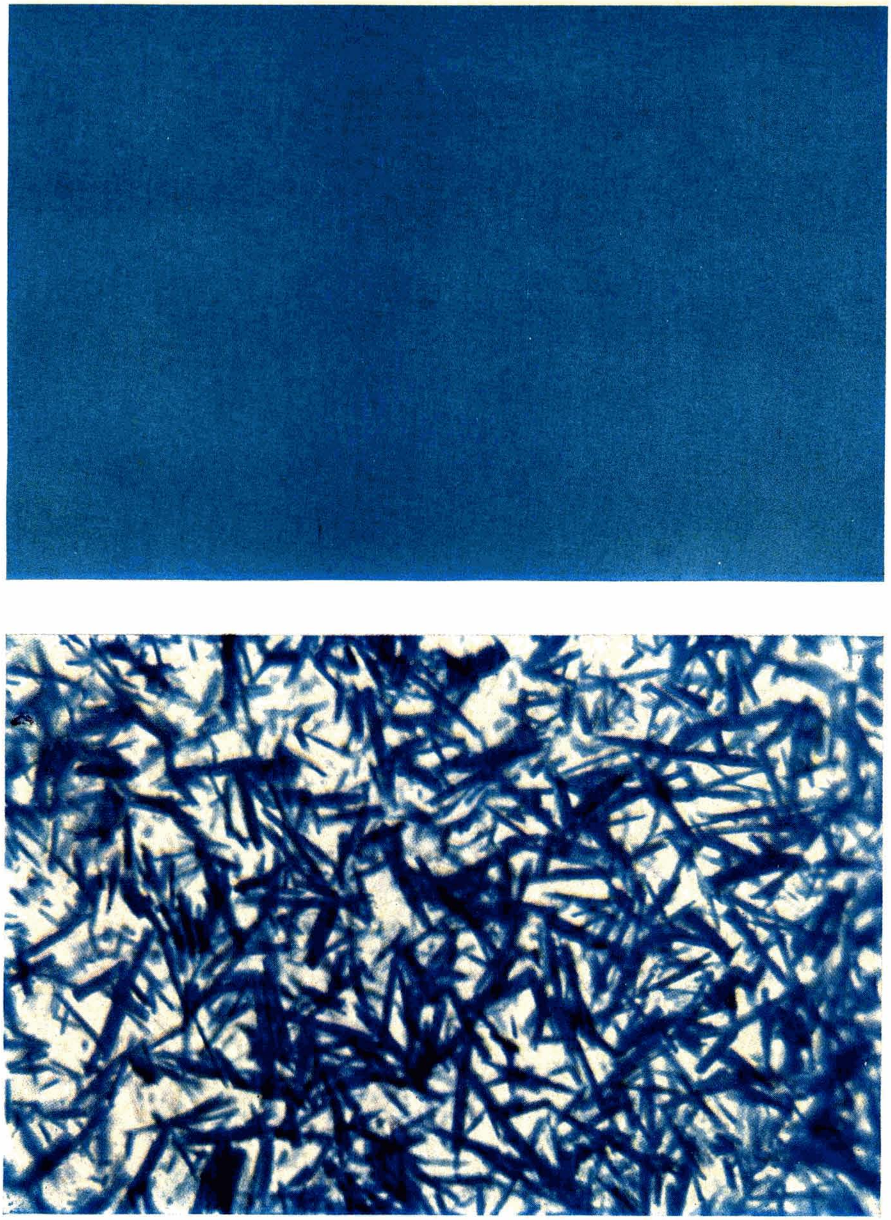


Fig. 4
Microphotographs of phthalocyanine blue, (top) alpha-form before storage, (bottom) recrystallised to beta-form after storage.

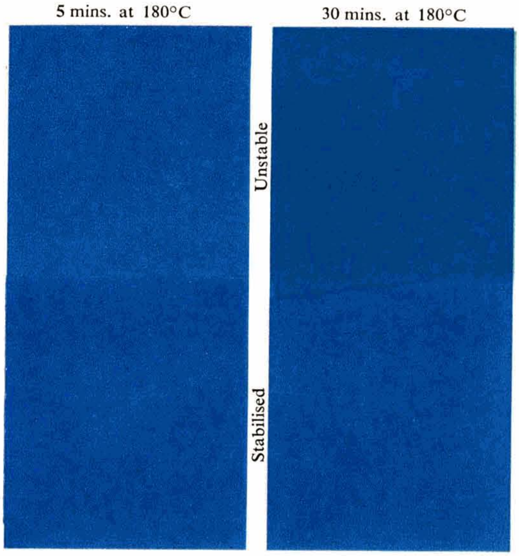


Fig. 5
Stability of alpha-form phthalocyanine blue in polyvinyl chloride.

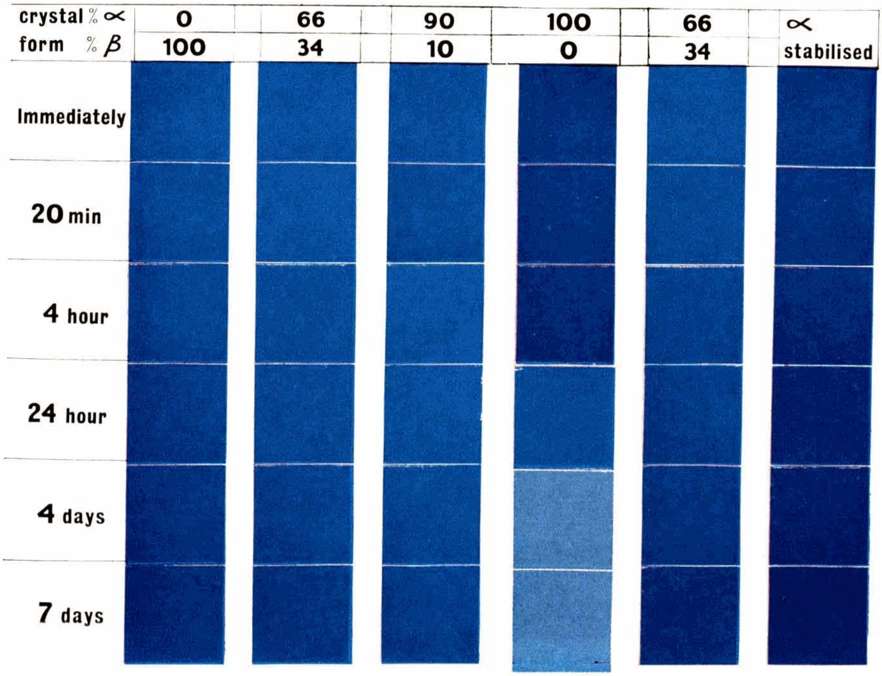


Fig. 6 Stability of alpha-form phthalocyanine blue in paint (the alpha-form used in the 2nd 66%/34% beta pigment was prepared by acid pasting).

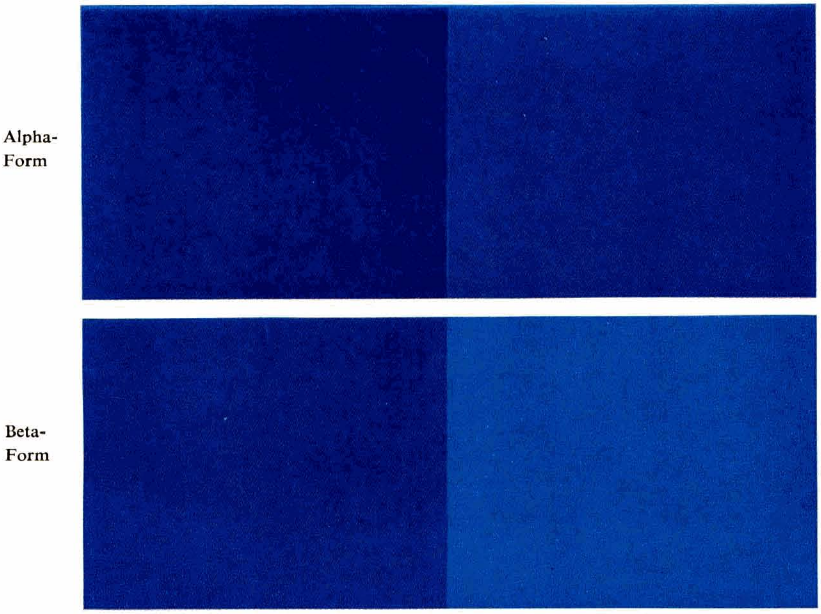


Fig. 7
Illustrations of the difference in hue between alpha and beta-forms of phthalocyanine blue in printing inks.

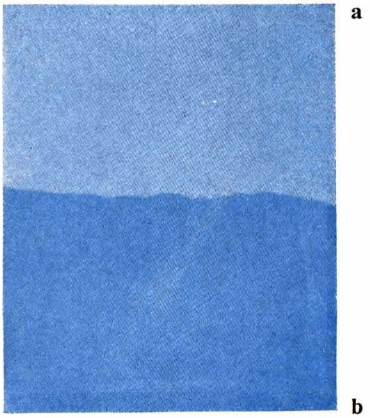


Fig. 8
Dispersibility in rubber of copper phthalocyanine blue (a) untreated, (b) treated.

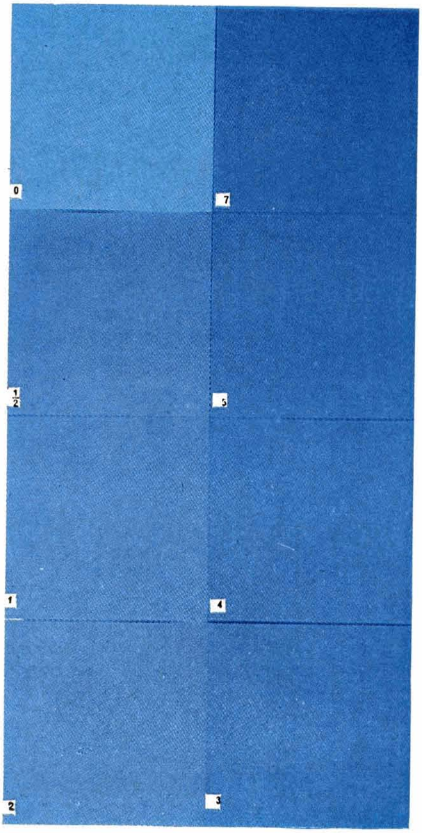


Fig. 9
Effect of salt grinding on the strength and hue development of copper phthalocyanine blue in relation to alpha-beta content. The figures on the panels indicate the times of salt grinding in hours.

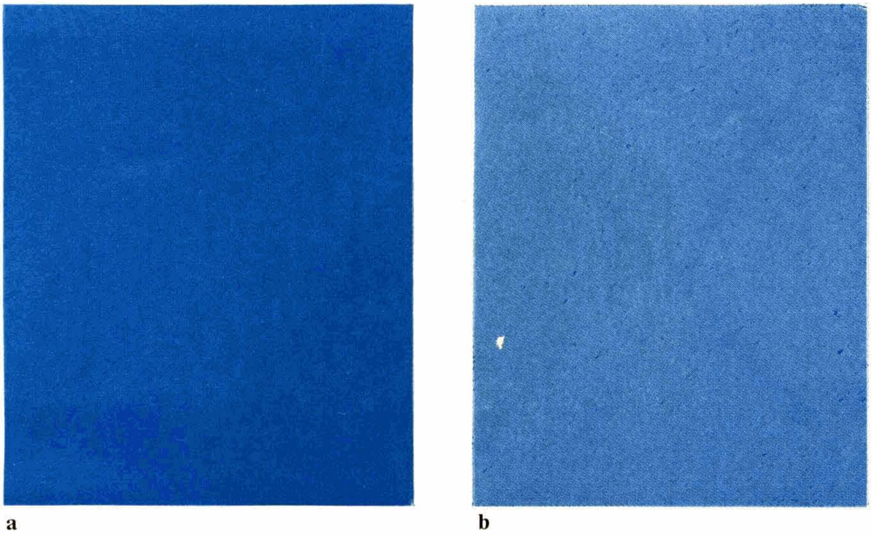


Fig. 10
Paint film flooding. Illustration of finger rubbing giving (a) paler and (b) deeper lines.

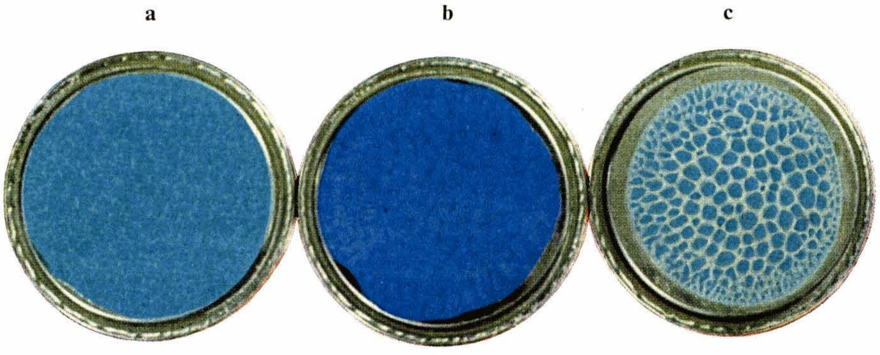


Fig. 11
Benard cell formation with (a), (c) pale edges, (b) deep edges.

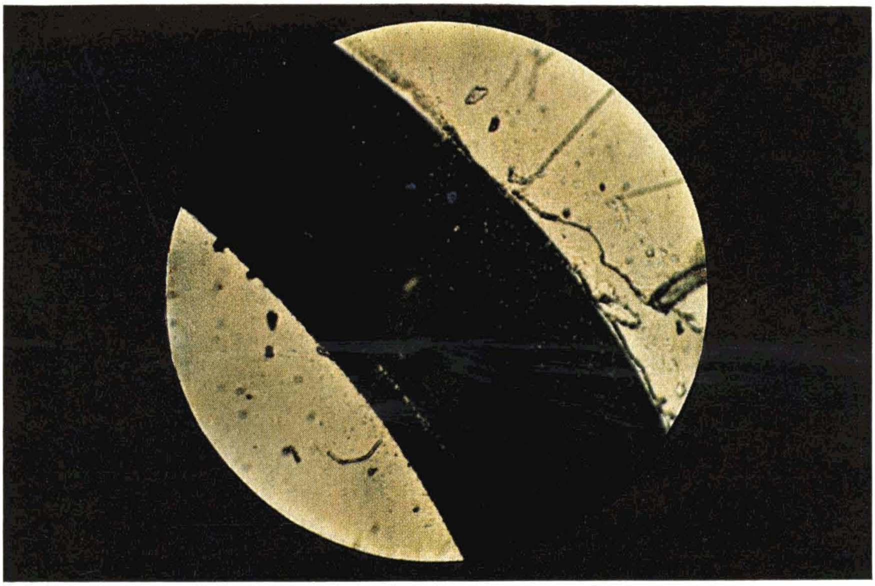


Fig. 12
Cross section of paint film illustrating flocculated phthalocyanine blue particles. Photograph by kind permission of the Paint Research Station.

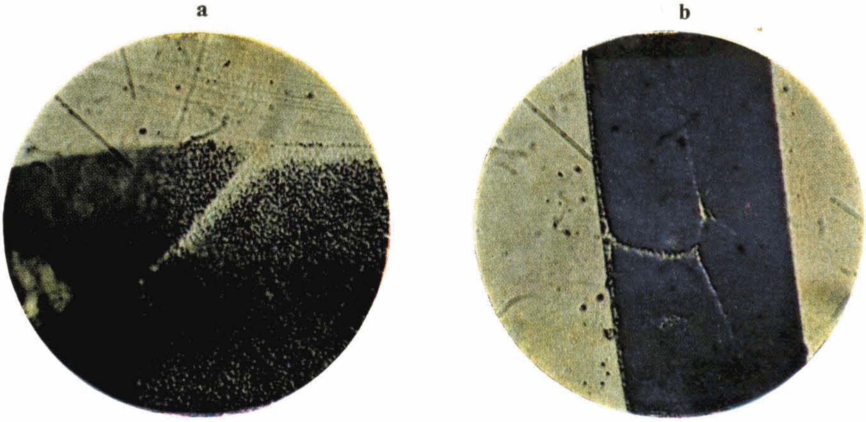


Fig. 13
Cross-sections of paint films showing (a) the effect at halo on a run down and (b) section devoid of pigment which looks blue to the eye.

Fig. 14
Paint film defects: (a) Satisfactory film, (b) showing a poor film and flocculated run down.

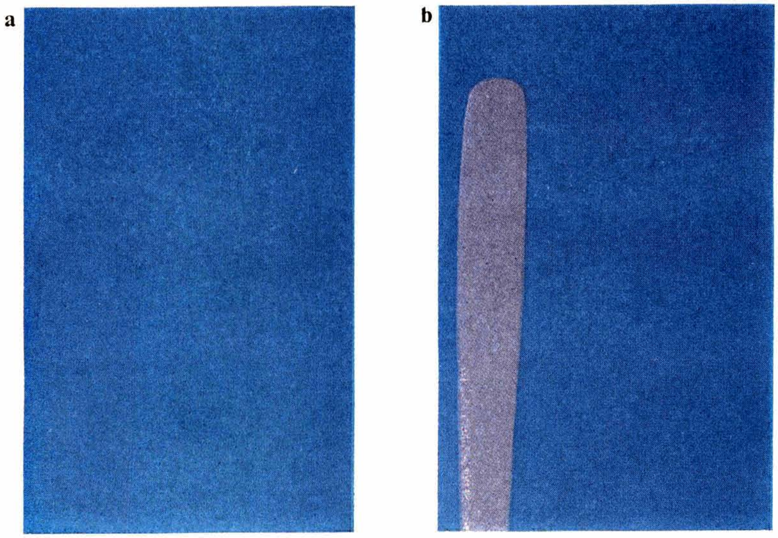
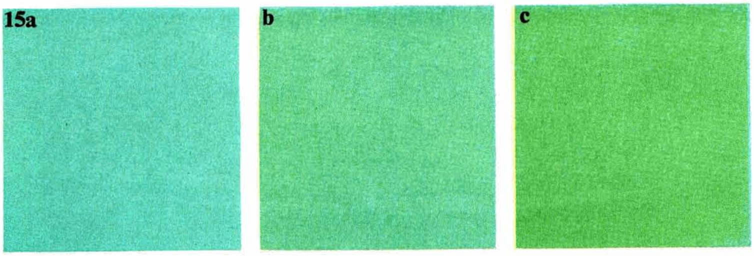


Fig. 15
Phthalocyanine greens showing the effect of chemical change. (a) copper polychlorophthalocyanine, (b) metal free polychlorophthalocyanine, (c) copper polychlorobromo phthalocyanine.



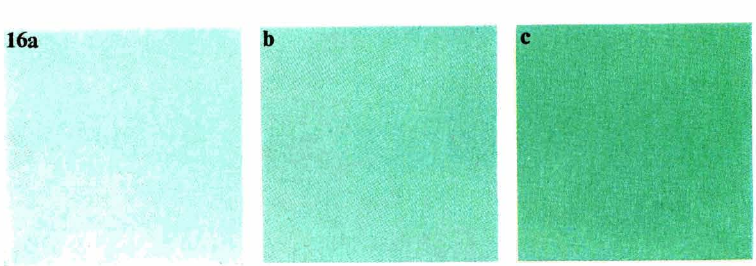
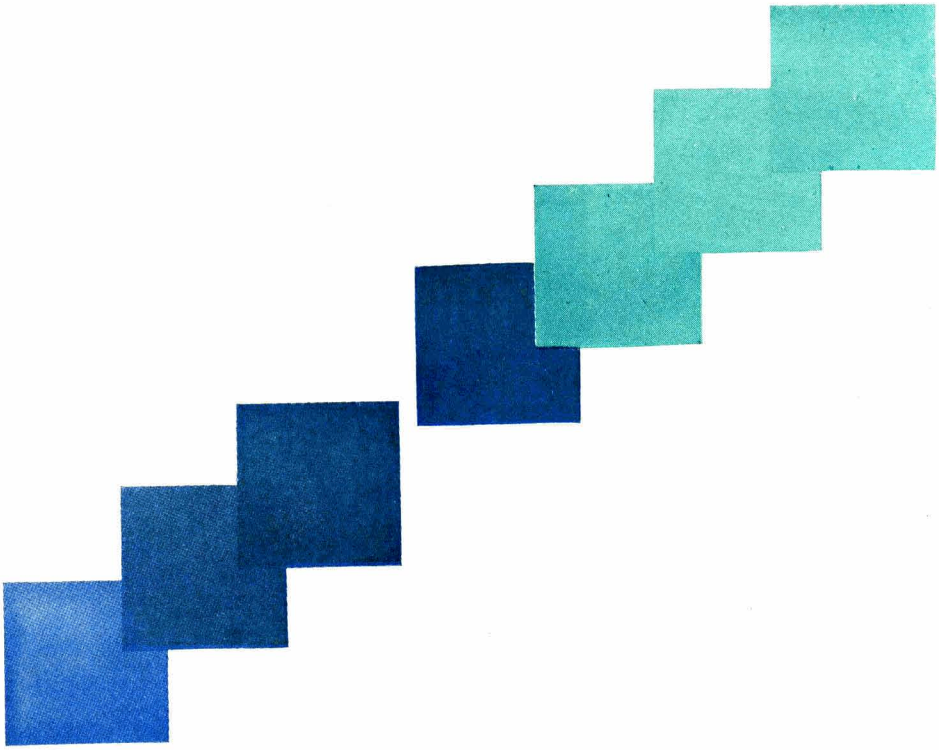


Fig. 16
Phthalocyanine greens showing the effect of physical change. (a) untreated pigments, (b) treated pigment at similar strength, (c) treated pigment at similar concentration.

Fig. 17
The phthalocyanine spectrum. Moving from left to right the pigments shown are, experimental blue, alpha-form copper phthalocyanine blue, beta-form copper phthalocyanine blue, metal free phthalocyanine blue, copper polychlorophthalocyanine, copper polychlorobromo phthalocyanine, experimental green.



molecules which are, of course, in the majority and the comparatively few dissolved ones, molecules continuously passing from the particle surfaces into solution and emerging from solution on to the particle surfaces. Molecules coming from solution would be expected to arrange themselves in the stable β-pattern. In addition there is the process of Ostwald ripening in which larger particles in suspension grow at the expense of the smaller and the smaller are generally likely to be those in the α-form. A closer examination of this mechanism has recently been made by Suito and Uyeda[2]. These workers studied suspensions of copper phthalocyanine in aromatic solvents and followed the changes by electron microscopy and X-ray diffractometry. They have revealed that some preliminary growth of metastable crystals precedes the actual transition into stable crystals and that two crystal habits are assumed by each crystal form. It seems likely that these discoveries will prove significant in phthalocyanine pigment technology although they do not alter the picture of pigmentary α-form reverting to larger crystals of β-form.

It is not always appreciated that there are other causes of crystallisation besides storage in aromatic solvents. It may occur in the absence of what are normally considered crystallising solvents and particularly at elevated temperatures. Such cases are found in printing ink phenolic resin systems with no solvent which are processed by high shear rates at elevated temperatures, and similarly in pvc where the plasticiser has a crystallising action at high temperatures during processing (Fig. 5). In these cases, once the material has been processed and has recooled, there is no further tendency to change. Such rapid changes associated with mechanical action are usually changes to a greener shade with little strength loss, since there is insufficient time for crystal growth. A similar quick phase conversion with little loss of colour strength can be made to occur in paints ; attention has been drawn to this by Anderson and Wallgren[3], but they do not discuss the mechanisms of crystallisation responsible. The present authors have studied this by examining the crystallisation of mixtures containing different proportions of α- and β-forms. The series of paint panels in Fig. 6 have all been made from the same paint system with a high aromatic solvent content and the same level of pigmentation using pigments with different proportions of α- and β-forms ; paints were applied after various periods of storage.

The sample containing 100 per cent β-form is substantially unaffected by time of storage. That containing 34 per cent β-form is almost at once completely converted to the β-form, but without great loss of strength. The pigment with only 10 per cent β-form is converted less quickly, but still gives the stable green shade. The sample containing practically no β-form shows a limited stability, holding its strength for some time, but in the end it deteriorates very badly. Such results are affected by the crystallising tendency of the medium and the amount of physical work applied, but this is representative of the two types of crystallisation. The position can be explained by postulating

(*i*) that a seeding action is given by any β-crystals present and

(*ii*) that crystal growth and hence strength loss depends on slow conversion of α to β-form.

For comparison the effect of such storage on a stabilised α-form blue whose strength and red shade are unaffected is shown.

Stable crystals of copper phthalocyanine

In the early days of phthalocyanine technology, the crystallisation defect was a serious restriction on the use of an otherwise excellent colour. Nowadays there are two ways of obtaining stable results : by using the β-form or the stabilised α-form.

The existence of the β-form was first recognised by von Susich in Germany in the mid-1930s, but it was not generally known till publication of the BIOS and FIAT reports after the war. A method was found to produce the β-crystal form in a suitable state for pigmentary use, but was not commercially available until about 1950 in the United States and not until 1957 in Europe, first in Switzerland and then in Britain. This β-form copper phthalocyanine pigment shows other advantages besides crystallisation resistance. It is closer than is the α-form to the ideal pure blue for three- and four-colour printing (Fig. 7), giving brighter combinations with other colours and inks of better flow than most α-forms. In paint systems it is less liable to flocculation defects than the α-form.

The other type of product which is resistant to crystallisation is the stabilised α-form usually obtained by introducing a substituent so that the arrangement of molecules to give the β-crystal is sterically difficult or impossible. By far the most important substituent used for this purpose is a single chlorine atom in the 4-position. Only the α-form of 4-monochloro-copper phthalocyanine exists so that crystallisation defects are impossible. It is interesting to note that 3-monochloro-copper phthalocyanine has both the α- and β-forms[4], presumably because its molecular shape is closer to that of the parent compound. It is not necessary to use a pure monochloro-copper phthalocyanine to obtain stability in practice ; a mixture of approximately equal parts with unchlorinated copper phthalocyanine is just as resistant, probably because the chlorinated copper phthalocyanine causes a discontinuity on the growing crystal faces. Such mixtures are made either from chlorinated intermediates or by chlorination of copper phthalocyanine itself and are of great technical importance. They tend to be slightly greener than the pure α-form and early versions were duller in shade.

Many other substituents prevent formation of the β-form, e.g. nitro, amino and phenylacetyl groups. Other metal phthalocyanines, such as those of tin, magnesium and aluminium, suppress crystallisation when present in quite small amounts, again presumably by upsetting the regular arrangement of the β-crystal lattice. Nevertheless, chlorination remains the most important basic method of stabilising the α-form.

The only reliable measure of crystallisation resistance is by tests of the type illustrated in Fig. 6, where a paint is compared at different stages of its storage life. Methods depending on boiling pigment with solvent are not reliable ; if the product is examined microscopically, significant changes may be below the limit of resolution, and if it is recovered for colour tests, changes in dispersibility may well mask the effect being looked for.

Methods for formation of pigments from crude

Crude copper phthalocyanine from the reaction vessel is normally in β-form with crystals much too large for effective pigment use and not reducible to pigment size by simple grinding. Methods of conversion to pigment provide a voluminous patent literature. There are two main methods ; these are treatment with strong acid and grinding with an inorganic salt.

Acid pasting

The acid treatment method, loosely called acid pasting, consists essentially of dissolving copper phthalocyanine blue in concentrated sulphuric acid followed by drowning the solution in excess water to reprecipitate the pigment as finely divided particles almost completely in the α-form. This is a purely physical action and any chemical purification of the starting material is incidental. Oleum, phosphoric acid, chlorosulphonic acid and organic sulphonic acids and mixtures of these are all possible solvents. Concentration of pigment in the acid can be varied and the precipitation temperature is a very important factor which may vary from below 0°C to 100°C or even higher if a closed system is used for the dilution. The product may be modified by addition of a surface active agent during precipitation or by an after-treatment, very often to improve texture. An example of this is in the production of a pigment for colouring rubber ; the two pieces shown in Fig. 8 are coloured with pigments prepared by similar processes except for a suitable after-treatment in one case. In passing it is worth noting that the copper in the complex is so firmly held that no breakdown of the rubber occurs so long as the pigment has been treated to remove traces of inorganic copper.

A more sophisticated method of acid pasting is known as permutoid swelling, where the pigment is treated with sulphuric acid of a lower concentration insufficient to dissolve it, but sufficient to form a slurry of the so-called sulphate which occurs as coarse green crystals and which decomposes to give the α-form on addition of water. One may use acid of concentrations about 60-80 per cent and work from either dry copper phthalocyanine or presscake. An important factor is adequate particle size reduction of the sulphate crystals by using a heavy duty machine of the dough-mixer type for example, before addition of water. The α-form is usually produced, although with concentrations below about 60 per cent sulphuric acid, some β-form may remain unchanged. Acid treatment processes can also be applied to substituted phthalocyanines or mixed phthalocyanines just as easily.

Salt grinding

The basic principle is that crude copper phthalocyanine is mixed with a grinding aid, usually an inorganic salt, and the mixture subjected to attrition grinding in a ball mill or similar plant. This causes a particle size reduction and at the same time a phase change, any β-form being converted to the α-form. The salt is finally removed by slurrying in water and the insoluble pigment recovered. A variation of this process is to grind wet with a non-crystallising solvent either at low viscosity in a ball mill or at high viscosity in a heavy duty mixer. In wet grinding it is not always necessary to use salt, and small shot, pebbles or sand have been reported as successful aids.

This is a much more time dependent process than acid pasting, and its course has been followed by examining samples removed from a test grind with sodium chloride in a ball mill (Fig. 9). It will be seen that the rate of

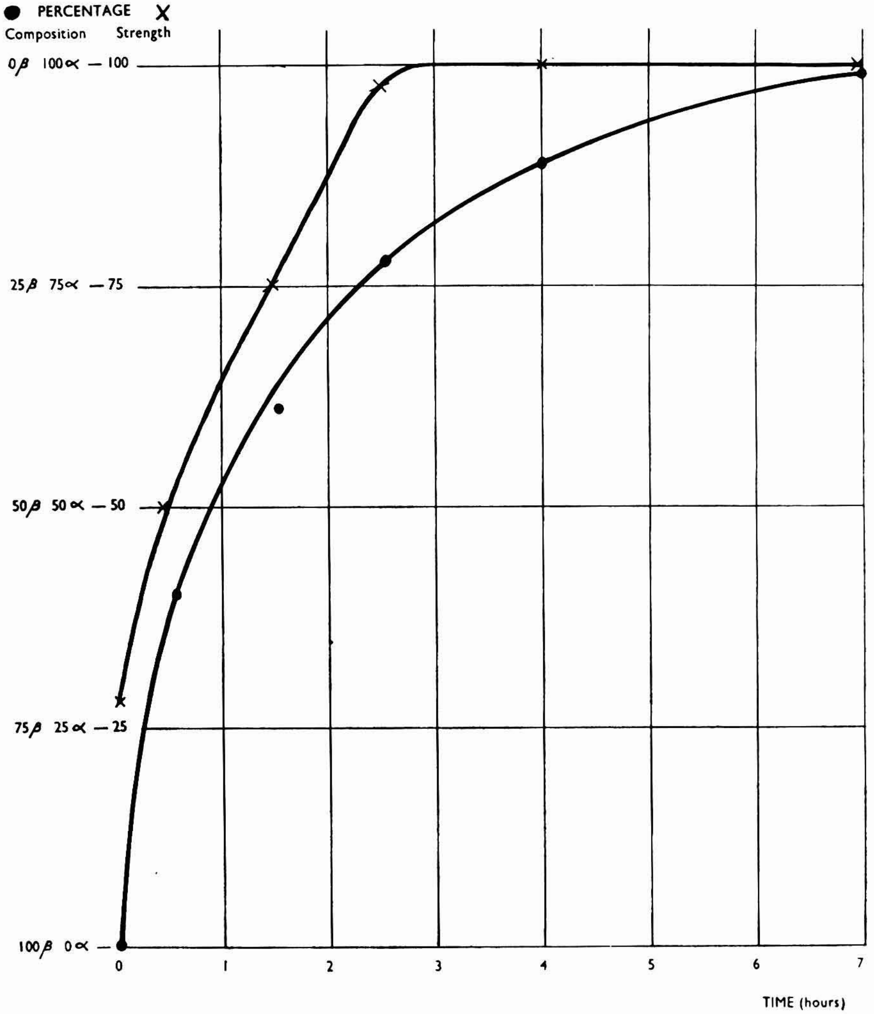


Fig. 9.

formation of α-form is rapid at first, then slows down, the same course being shown by the development of colour strength. It should be noted that maximum colour strength is developed earlier than the maximum α-content. There is also, of course, a reddening of shade, but this is masked by the comparatively large strength differences.

This is a remarkable process and its mechanism is not clearly understood. It seems likely that the salt particles act as small grinding elements on the pigment to reduce particle size, but the unexpected feature is the phase change. No reaction between colour and salt is involved ; this is clear from the wide range of materials which may be used as grinding aids, ranging from sugar or urea to inorganic salts or even small shot. One postulated mechanism is that grinding yields an amorphous product which only crystallises to the α-form when the mixture is slurried in water to remove the grinding aid. This is attractively analogous to the acid pasting process, but cannot be correct, since infrared examination of samples from a ground mixture of potassium bromide and copper phthalocyanine, without any aqueous treatment, reveals peaks due to newly-formed α- as well as residual β-crystals ; the α-peak increases with time of grind as might be expected. If, then, β-form is changed to α-form on grinding alone, one is forced to the conclusion that the arrangement of individual molecules in the crystal lattice is being fundamentally altered by the stresses set up in this comparatively crude process.

Production of β-form by salt grinding

Perhaps a more important application of the salt grind process is the production of a β-form pigment. One may start from either the α- or β-form, and treat with a crystallising solvent either during or after salt grinding. If no solvent is present in salt grinding, the product is the α-form, *irrespective* of the form of the starting material. When sufficient solvent is present during salt grinding, the product is the β-form, again irrespective of the starting material. Much apparent confusion in the literature arises from a failure to state this point explicitly.

The simplest method of producing a pigmentary β-form is to salt grind crude copper phthalocyanine in the presence of a crystallising solvent. The production of a satisfactory pigment depends on suitably balancing two mutually opposing forces : grinding, which increases pigment strength and tends to produce α-form, and crystallisation, which decreases pigment strength and produces β-form. Efficiency of the mill and increased grinding time give increased strength, but if they are overdone the product contains an unacceptable amount of α-form. Excess solvent and high temperatures of grinding help to preserve the β-form, but if they are not carefully controlled a weak product is obtained.

The other method, a salt grinding followed by solvent treatment, is a two-stage process, but is simpler to control. The mechanism is rather paradoxical. Salt grinding, as we have seen, produces the α-form, which when exposed to solvent generally gives large β-crystals of poor strength. How then does this process give rise to a pigmentary β-form? The answer is illustrated in the panels in Fig. 6, where certain proportions of α and β mixes show complete conversion to β-form without substantial loss of strength. Thus it is necessary to avoid overgrinding, thereby ensuring that sufficient β-crystals remain as seeds to permit rapid phase change without crystal growth. It is also essential to avoid undergrinding which would leave comparatively large β-crystals to pass through the solvent treatment unchanged and to reduce the final pigment strength. It is interesting to note that a mixture of, for example, 66 per cent α and 34 per cent β behaves similarly whether the α-form is prepared by salt grinding or acid pasting, as shown by the first and second pigments with this composition in Fig. 6 respectively. This confirms that phase change without strength loss is a function of α : β ratio rather than a direct result of the salt grinding technique as has sometimes been suggested.

Other crystal forms of copper phthalocyanine

The published literature on other forms of copper phthalocyanine is summarised in Table 1. Although the two delta-form reports and the R-form report give quite different preparative methods, the X-ray data tabulated suggest strongly that they are one and the same form, a suggestion supported by general agreement on the redness of the product. This redness could be a desirable commercial factor, but it remains to be seen whether it will be more than an academic curiosity, since the same result may be obtained by shading α-copper phthalocyanine with dioxazine violet which has similar stability. The answer to this question will probably depend on clarification of the resistance to crystallisation of the new form and to the economics of its production.

Table 1

Reported crystal forms of copper phthalocyanine blue lecture

Name	Reference	Preparation	Colour properties	X-ray diffraction spectrum lines between 4.5 and 6.0 Å
Gamma γ	USP 2,770,629 (Am.Cyan)	Swelling with H_2SO_4 of less than 60 per cent concentration	No information	5.57
Delta δ	BP 912,526 (ICI)	Exposure of α-form to benzene at ambient temperature	Very red shade	5.0-5.05
R	USP 3,051,721 (Am. Cyan)	Synthesis from phthalonitrile at high temperature and high shear rates (no chlorination occurs)	Redder than α-form Stable to crystallisation	5.07 (main line), 5.27
Delta δ	Fr.P. 1,301,080 (KVK)	Acid swelling on heavy duty mixer with grinding salt and urea	Redder than α-form Not stable to crystallisation	5.07 (main line), 5.18
Alpha α	—	—	—	5.49, 5.70
Beta β	—	—	—	4.79, 4.90, 5.74

Flocculation, flotation and flooding

There is another phenomenon, or rather range of phenomena, frequently met with in the use of phthalocyanine pigments in paint systems, where the common factor is a breakdown in homogeneity of a system containing blue and white pigments. The terms flooding, flotation and flocculation are in common use in this context and, as there is some variation in the precise meaning of these terms, definitions are necessary.

First, there may be a relatively greater concentration of one pigment, either blue or white, in the surface layer which is evenly coloured overall, as compared with the rest of the paint film. If the wet film is rubbed with the finger the surface effect is lost and a deeper or paler colour produced. Also, there is often a difference in shade between the colour of the dry film and that of the wet paint in the can. This is generally known as flooding (Fig. 10).

Alternatively a network of lines, either deeper or paler than the spaces between them, may be produced over the surface as the paint film dries. This is known to be associated with the formation of Bénard cells in the drying film and is generally distinguished as flotation (Fig. 11).

The third case is that described as flocculation, which is an inadequate description, since the word strictly refers to the cause rather than the effect. The term is commonly used in the United States, less so in the United Kingdom, to describe the case where shearing forces cause an increase in colour strength. It is generally considered that the particles in the original dispersion have congregated into flocs with larger effective particle size and hence lower colour value. These flocs are redispersed by shearing forces to give back the original strength. The effect must, of course, be clearly distinguished from poor initial dispersion. The fault of " flocculation " is often exemplified in practice by a comparison of sprayed and dipped panels. It will be noted that the practical distinction of this trouble from the white flooding described above is not easy, but it is seen to be a separate phenomenon. It is known that copper phthalocyanine does form such flocs, which are observed under the microscope, as shown by the film cross-section in Fig. 12, kindly supplied by the Paint Research Station. If a wet paint film gains strength on finger rubbing, but does not when the surface is broken gently with a hair, white flooding cannot be present.

At this point it should be noted that these unfortunate effects are not confined to phthalocyanine blue. Similar troubles are experienced with other high molecular weight organic colours such as dioxazines and have been traditionally associated with mixed colours such as Brunswick Green. These phenomena are much less evident when phthalocyanine blue is used with zinc oxide instead of the titanium dioxide. However, the increasing use of both phthalocyanine blue and titanium dioxide in the last 15 years has made the problem of such importance that no pigment manufacturer can afford to ignore it. Of recent years a number of products presenting a solution to the organic pigment aspect has appeared, but no complete solution is yet available and this is hardly surprising when the complexity of the factors and the variation in practical conditions are considered.

A number of excellent papers[5, 6, 7] discussing these effects is available and a study of the postulated mechanisms indicates that the whole system of solvent, binder, white and blue is involved. This has long been known to practical workers in the field.

Flocculation

Flocculation is affected by initial pigment particle size and shape and surface characteristics ; it is equally affected by the vehicle. Ease of wetting and adsorption of medium or solvent play their part and selective adsorption of certain fractions of an alkyd, for example, is a complicating factor. The pigment maker is thus asked to prepare a colour which will not flocculate significantly in any medium—this alone is a difficult task.

Flooding

Further factors arise with flooding. The tendency of a particle to move within a paint film is controlled by many factors including the degree of flocculation, and phthalocyanine blue and titanium dioxide may not react in the same way to given conditions. Perhaps more important is the tendency of either particle to remain in the surface layer after arriving there and this is controlled by the properties of the whole pigment/medium/air system with its three interfaces, pigment/medium, medium/air and air/pigment. This appears to require that the pigment worker matches the performance of the blue to that of the titanium dioxide in an unspecified system. However, if blue and white can be induced to associate, a considerable simplification is obtained and it is probable that in practice this is often achieved. In most pigment dispersions the colour is present in loose groups containing something of the order of ten particles and it is obviously advantageous if such groups contain both blue and white pigments.

Flotation

The occurrence of flotation presupposes the formation of Bénard cells and all the variables concerned in that are relevant—amount and evaporation rate of solvent, film thickness, atmospheric conditions, etc. There is more than simple blue and white separation involved here. The Paint Research Station has examined cross-sections of paint films showing flotation and have kindly made available the microphotographs shown in Fig. 13. The first shows a section through a line seen on the paint surface as a deep blue edge to a Bénard cell. It will be seen that the line does not contain an excess of blue pigment as might be expected, but is quite empty of pigment particles, its depth of colour being due to internal reflection. The second is a section through a blue halo such as is often seen at the edge of a run-down and again is quite devoid of pigment. This emphasises the fact that there is a great deal more to a paint film than is revealed by superficial examination and that it is vital to support speculation in this field by reliable experimental evidence.

To sum up, there are a number of factors quite outside the control of the organic pigment manufacturer, and these make it difficult to find a product which will be universally satisfactory. On the other hand, attention to the flocculating and associating tendency of the blue pigment has been rewarded by a number of products which are much more satisfactory than earlier pigments and work well in the majority of systems (Fig. 14).

Control of flocculation

It has been postulated that the tendency of phthalocyanine blue to flocculate is a result of its symmetrical structure which shows no dipole moment and thus lacks potential electrical charges whose mutual repulsion might oppose van der Waals' attraction forces. The most generally successful means of conferring resistance to flocculation is based on this hypothesis and consists of the introduction of various polar groups which will provide a charge on the particle surface. Such groups as sulphonic or carboxylic acids are the extreme cases of this technique, but less polar substituents such as CH_2OH or salts of *o*-carboxy benzamido groups have also been reported as effective.

The substitution must be carried out with some care since if it is overdone the solubility in alkali may reduce fastness of the pigment below an acceptable level. Fortunately it is found that as with stabilisation against crystallisation where a proportion of monochlorinated copper phthalocyanine confers resistance on a blend with unsubstituted material, only some of the copper phthalocyanine molecules need be substituted to give flocculation resistance. In fact, beyond a certain limit no further improvement is gained by increasing the content of polar molecules. One may cure white flooding in a paint by adding a certain proportion of substituted copper phthalocyanine and on going beyond this proportion, induce blue flooding. It is customary to apply this method to a blue already stabilised against crystallisation by, say, chlorination, and in addition to coat the particle with a suitable resin to overcome the poorer texture or dispersibility thought to result from the presence of the polar groups.

It should perhaps be pointed out that although this technique is most effective, it does not constitute a proof of the hypothesis on which it is based, and it seems at least as likely that the improvement found is due to an increased tendency of polar materials to associate with titanium dioxide.

There are other methods of overcoming flocculation difficulties, such as additions of aluminium benzoate or formation of copper phthalocyanine in the presence of titanium or zirconium oxides to give an intimate mixture. The latter obviously operates by affecting association with white, and gives a product of low strength. Additions of vanadyl or tin phthalocyanines reduce flocculation, possibly because the central metal atom has valencies not taken up by the phthalocyanine ring and which carry oxygen or chlorine atoms perpendicular to the plane of the molecule to upset its electronic symmetry. One very important point not always realised is that the β-form of ordinary unsubstituted copper phthalocyanine is much less liable to flocculation defects than the α- or non-crystallising α-forms, and that if the cleaner greener shade is acceptable, use of a β-form often provides a simple solution to the paint maker's difficulties.

Phthalocyanine green

The important phthalocyanine green pigment is copper polychlorophthalocyanine. Copper phthalocyanine chlorinates easily and as chlorine is introduced the shade moves towards green, rather slowly at first, and more rapidly after eight atoms have been introduced until at about 14 chlorine atoms the shade is the familiar one. The addition of the final two atoms to give complete substitution has little effect on shade.

Naturally there has always been a wish to extend the shade range further towards the yellow, and in the late 1950s this was achieved by two routes (Fig. 15). The first was to prepare the metal-free polychlorophthalocyanine, which is yellower than the copper compound in the same way as the metal-free unhalogenated phthalocyanine is greener than its copper derivative. The second route, which is probably more commercially attractive and certainly more widely used, depends on the long-known fact that replacement of some of the chlorine atoms by bromine gives a yellower shade. The technical problems in producing a bright and strong pigment from this compound were, however, considerable, and it was not until 1959 that yellow shade greens of this type became available commercially.

The finishing of phthalocyanine green pigments is a less complicated matter than with the blues, since dimorphism and hence crystallisation does not occur and it is generally considered that flocculation/flotation troubles are comparatively minor.

The green is generally isolated by precipitation from a reaction mixture and therefore first occurs in a fine particle size which can be used as a pigment straight from the chlorination process. Better results, more in accord with current demands on pigment properties, are obtained by introducing a finishing process. Acid pasting, as described earlier for blue, is common although the insolubility of the green demands more and stronger acid such as oleum or chlorosulphonic acid to obtain a suitable degree of swelling or solution. Salt grinding may be used, but since no phase change occurs, this technique lacks the flexibility and wide range of applications found with the blue. While there are not two crystal forms of phthalocyanine green, there is a distinction between more and less crystalline forms and crystallinity can be increased by appropriate solvent treatments although there is no substantial growth as with copper phthalocyanine itself. The green micro-crystals can be characterised by the sharper X-ray diffraction pattern and also by electron microscopy. This change has a notable effect on pigment properties—the more crystalline green is cleaner, stronger and disperses more easily than the starting material (Fig. 16).

The phthalocyanine gamut of colours

The colour range now known in phthalocyanine pigments is shown in Fig. 17. The commercially available pigments have been extended by the inclusion of a very red blue at one end and a very yellow green at the other. These are at present experimental products only and have been included to indicate the efforts always being made to extend the range further.

The preparation of a " phthalocyanine red " was for many years a goal eagerly sought by pigment chemists, but it now seems that this was a mirage, if a red chemically similar to copper phthalocyanine is meant. The phthalocyanine structure has been very thoroughly examined and has thrown up nothing of commercial value outside the blues and greens, while many other chemical classes, such as the quinacridones, give promise of a red with the properties of the phthalocyanines and this line now seems to be a more profitable one for the chemist to follow. Future developments in the phthalocyanine range are likely to be on the one hand extensions of the range towards redder blues and yellower greens, and on the other towards pigments tailored for special purposes, offering benefits in properties other than shade and strength. Such properties as flow, dispersibility, dispersion stability in different media are not yet well enough understood and it is in such fields as these that new advances in phthalocyanine technology may be chiefly expected.

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Discussion

MR. J. SMETHURST, from the chair, inquired whether in view of the interest of the printing ink industry in red-shade blues of superior fastness properties the lecturer could say whether the delta form phthalocyanine blues described were likely to become a commercial proposition in the near future.

DR. EASTON felt that this was a difficult question to answer with certainty. Literature reports differed on the stability in crystallising solvents, and this would have to be resolved. The economics of production would also be a vital consideration.

MR. H. F. CLAY asked if X-ray crystallography was used as a control test in the manufacturing process.

DR. EASTON answered that either this or infrared spectroscopy were possible control tools. His company generally used the latter as being simpler to operate than X-ray diffraction.

DR. D. A. PLANT referred to the cross-sections shown and asked whether the Paint Research Station had produced a cross-section through a Bénard cell showing white edges, and if so what it showed in comparison with the section shown through a cell with a blue edge.

DR. EASTON asked if Dr. Crowl would care to comment.

DR. V. T. CROWL said that it had not been possible to get a cross-section at the right point to cut through one of these edges of a white cell. This was something it was hoped to look into further, but as could be imagined getting the right one when cutting sections was rather a matter of luck.

MR. T. E. JOHNSON inquired whether it was likely that some of the paint manufacturers' problems would be solved by the newer types of titanium dioxide in association with phthalocyanine blues.

DR. EASTON replied that if these defects could be reduced by making changes in phthalocyanine blue it should be equally possible to do it by changing the titanium dioxide. The ideal would be suitable modifications in both cases.

DR. SMITH added that the answer was that it was likely—but how likely, of course, it was difficult to say. However, the changes in the white to which the questioner referred were not made with the intention of preventing flocculation of phthalocyanine blue ; by chance it could in fact work this way, but there were so many factors involved that it was at the moment only a matter of chance.

MR. I. S. MOLL commented that the " improvements " made in titanium dioxide quality over the past ten years had usually increased the severity of flocculation problems!

DR. W. CARR referred to the problem of the flocculation of phthalocyanine blue and the statement by the authors that the most popular explanation of this phenomena attributed it to the fact that the phthalocyanine blue molecule was a very symmetrical one and had a zero dipole moment. They had also said that attempts to overcome the flocculation were based on attempts to introduce polar groups into the molecule in order to increase its dipole moment. He asked if any dipole moments had actually been measured of the α forms, the β forms, and the non-flocculating forms that are on the market, and also phthalocyanine greens, dioxazine violet, and other pigments with flocculating tendencies, in order to establish whether there is a critical dipole moment value below which flocculation tendencies could be expected.

DR. EASTON replied that although he and Dr. Smith were willing to go along with this hypothesis so far, they did not consider it a full explanation of the facts. A thorough exposition of the polarity theory, with some quantitative data, had been given by Lacey, Roberts and Giambalvo[8].

MR. D. M. VARLEY asked whether a mixed α/β phthalocyanine gave commercial value in practice if the crystallising change occurred only slowly in less vigorous solvents, e.g. white spirit.

DR. EASTON said that mixed α/β phthalocyanines had commercial value when used under suitable conditions, either in non-crystallising media or in media whose crystallising power was adequate to induce rapid phase change without loss of strength. Those pigments with higher proportions of β-form, e.g. 34 per cent, gave rapid phase change in even comparatively mildly crystallising media.

MR. VARLEY asked if such a product had any value in the decorative market.

DR. EASTON replied that one would have to be careful in the decorative field, but even here such pigments are frequently very useful.

MR. VARLEY went on to ask whether a mixed pigment gave any advantage over straight β-form in strong crystallising solvents.

DR. EASTON said that there was not necessarily any advantage, but in particular cases benefits may be obtained from use of the mixed pigment.

MR. VARLEY finally asked if it was cheaper to produce.

DR. EASTON replied that it could be, but was not necessarily so.

MR. A. KING commented that in the production of rubber latex goods, " chlorine water dipping " after vulcanisation was used to reduce tack. In goods coloured with phthalocyanine blue this could lead to " greening." He asked if such relatively mild conditions could result in partial chlorination of the blue.

DR. EASTON said that although he was not aware of this phenomenon, he thought it might be due to either slight chlorination or oxidative degradation, but that investigation would be necessary in order to say which.

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Phenylmercury compounds as fungicides: Part IV

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Summary

Panels painted with different flat and gloss alkyd paints containing various phenylmercury fungicides were exposed outdoors in Lae, New Guinea. The rate of disappearance of mercury from the films was very high in all cases.

The attack of mould on the paint films was observed, and it was noted that, in general, the behaviour of paints containing fungicides was not appreciably different from that of other paints. A proposal is made for testing paints for mould resistance.

Composés de phénylmercure en tant que fongicides: 4ème Partie

Résumé

Panneaux-éprouvettes peintes avec une gamme de peintures aux alkydes, mates et brillantes, contenant plusieurs fongicides du type phénylmercure ont été exposés à l'extérieur à Lae, Nouvelle Guinée. En chaque cas, la vitesse de perte de mercure à partir des feuils était très élevée.

L'attaque sur les feuils de peinture provoquée par la moisissure a été appréciée. En général, on a noté que le comportement des peintures contenant fongicides n'était pas notamment différent de celui des peintures exemptes de fongicide. On propose une méthode pour évaluer la résistance à la moisissure de peintures.

Phenyl-Quecksilber Verbindungen als Pilzverhütungsmittel: Teil IV

Zusammenfassung

In Lea, Neu Guinea wurden Tafeln, die mit verschiedenen matten und glänzenden, eine Auswahl von auf Phenyl-Quecksilber basierenden Pilzverhütungsmittel enthaltenden, Alkydemaillen gestrichen worden waren, im Freien bewittert. Die Geschwindigkeit, mit der der Gehalt an Quecksilber aus den Filmen verschwand, war in allen Fällen sehr gross. Der Grad des Pilzangriffs auf die Anstrichfilme wurde beobachtet. Dabei wurde festgestellt, dass im allgemeinen das Verhalten von Pilzverhüter enthaltenden Farben sich nicht wesentlich von dem anderer Farben unterschied. Prüfmethoden für die Widerstandsfähigkeit von Anstrichmitteln gegen Pilzbewuchs werden vorgeschlagen.

Фенило–ртутные составы в качестве фунгисидов. 1V

Резюме

Панели окрашенные различными матовыми и блестящими алкидными красками содержащими разные фенило-ртутные фунгисиды были подвержены действию атмосферы в Лаео, Новая Гинея. Во всех случаях скорость исчезновения ртути из пленок была очень высокая.

Наблюдалось поражение красочных пленок плесенью, и замечено что, вообще, поведение красок содержащих фунгисиды не отличалось значительно от поведения других красок. Предложено способ для испытания устойчивости красок против плесени.

Introduction

In previous papers[1, 2] the fungicidal behaviour of di-(phenylmercury)-dodecenyl succinate (PMS) was reported. It was shown that PMS disappears fairly rapidly from all kinds of paint films, both indoors and outdoors. It was of interest to see how other phenylmercury compounds behaved. To study this, paint films containing phenylmercury acetate (PMA), phenylmercury oleate (PMO), phenylmercury naphthenate (PMN) and PMS were exposed in a humid tropical climate at Lae, New Guinea, and analysed for their mercury content at intervals. The fungus resistance of paints containing PMS and exposed under these conditions was compared with that of paints containing no fungicide.

Experimental details

Loss of different phenylmercury compounds from paint films

Flat and gloss alkyd enamel paints containing the various fungicides were brushed out on sheets of a polyethylene terephthalate polyester film, attached to both sides of masonite panels and mounted on racks at an angle of 45° and facing NNE at Lae. The mercury content of the paint film was measured at the start of the exposure, after four months, and after ten months. The results of these measurements are set out in Table 1.

Table 1

Mercury content of alkyd enamel paints exposed at Lae

Type of paint	Fungicide	Mercury in 100 mg paint film before exposure (μg)*	Mercury retained (%)			
			Upper side		Under side	
			4 months	10 months	4 months	10 months
Flat alkyd enamel	PMA	270	25	21	70	32
	PMN	256	34	9	55	36
	PMO	300	51	17	75	26
	PMS	312	43	16	76	22
Gloss alkyd enamel	PMA	249	44	28	66	42
	PMN	298	24	17	40	33
	PMO	350	35	18	59	39
	PMS	364	40	20	48	34

*100 μg in 100 mg paint films is equivalent to 0.1 per cent.

Fungus resistance of paint films containing PMS

Various paints containing PMS as fungicide and the same paints free of it were brushed on both sides of primed masonite panels (6 in by 12 in) exposed at Lae as described above. Inspection was carried out after six months and then at approximately three-monthly intervals. Each panel was assessed by noting the percentage area covered by mould and by the colour intensity according to the following scale :

9 black
8
7 dark grey
6
5 mid grey
4
3 light grey
2
1 faint grey
0 white

Paint films from the same paints were exposed on sheets of polyethylene terephthalate polyester film and these were analysed for their mercury content after 6 and 13 months. As it has been established by separate experiments that the loss of mercury from a paint film is independent of the substrate, it is safe to assume that the mercury content on the masonite panels is the same as on the plastic sheets.

Two commercially available types of paints were used. A flat paint with a pigment volume concentration (PVC) of 47 per cent is designated *F1*, the other gloss paint had a PVC of 17 per cent and is designated *G1*. The resin in *F1* was a 52 per cent length soya bean alkyd and the resin in *G1* was a 64 per cent length safflower oil alkyd. The paints were then modified by replacing part of the extenders or titanium dioxide pigment with the material stated in Table 2.

Zinc oxide : Zinc oxide with a PM compound is claimed to be specially effective as a fungicide[3].

Calcium carbonate : This is often added to paint formulations as an extender and many paint chemists believe that it has fungicidal properties.

Calcium silicate : This extender imparts to the aqueous extract from the paint film a pH on the alkaline side. The optimum pH range for mould growth appears to be mostly on the acid side, and calcium silicate may therefore be a useful ingredient for suppressing mould growth.

Barium metaborate : This compound has been recommended as a fungistatic extender in paint.

The results of these tests are summarised in Table 2 and the actual assessments are given in Tables 3 and 4.

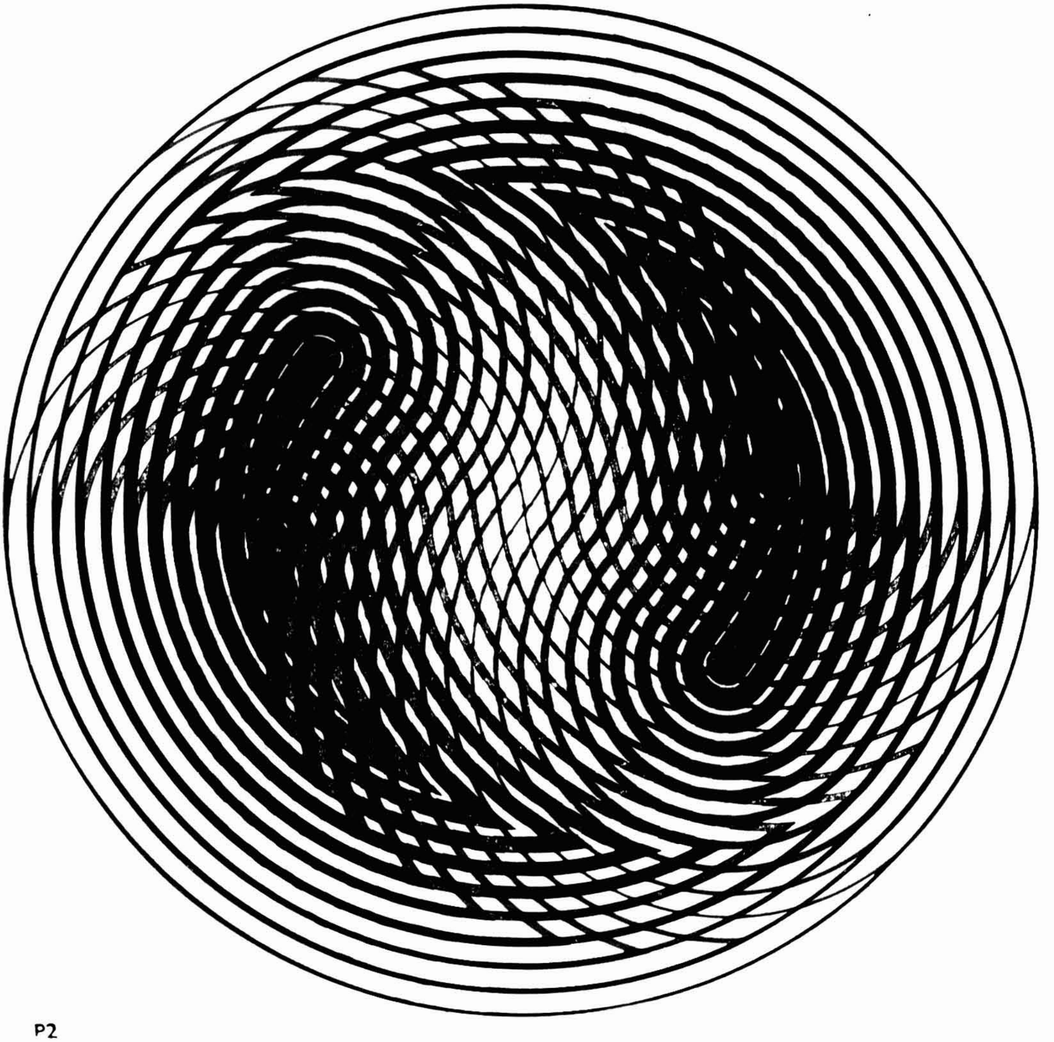
Table 2

Comparison of fungus resistance of paints containing PMS as fungicide with paints free of fungicide

Paint	Mercury in 100 mg paint before exposure (µg)*	Mercury retained (%)				Difference in fungus resistance
		6 months		13 months		
		Upper side	Under side	Upper side	Under side	
F1	0					Addition of fungicide does not make a difference
F1/A	290	24	31	12	14	
F2 .. .. (3 lb zinc oxide per gallon)	0					Addition of fungicide seems to make a difference especially on upper surface. Further work warranted.
F2/A	270	28	32	11	14	
F3 .. .. (3 lb calcium carbonate per gallon)	0					Addition of fungicide does make a small difference on underside
F3/A	273	23	27	15	13	
F4 .. .. (2 lb calcium silicate per gallon)	0					Addition of fungicide does not make a difference
F4/A	283	29	32	11	16	
F5 .. .. (3 lb calcium silicate per gallon)	0					
F5/A	267	27	30	13	19	Ditto
F6 .. .. (2 lb barium metaborate per gallon)	0					
F6/A	280	32	39	18	23	Ditto
G1	0					
G1/A	260	29	35	13	27	Ditto
G2 .. .. .. (2 lb zinc oxide per gallon)	0					
G2/A	282	29	30	18	17	Ditto
G3 .. .. (1.5 lb calcium carbonate per gallon)	0					
G3/A	250	28	42	22	22	Ditto
G4 .. .. (1.5 calcium silicate per gallon)	0					
G4/A	245	21	25	21	22	Ditto
G5 .. .. (2 lb barium metaborate per gallon)	0					
G5/A	290	29	34	16	23	Ditto

/A means addition of fungicides.

*100 µg mercury per 100 mg paint films is equivalent to 0.1 per cent.



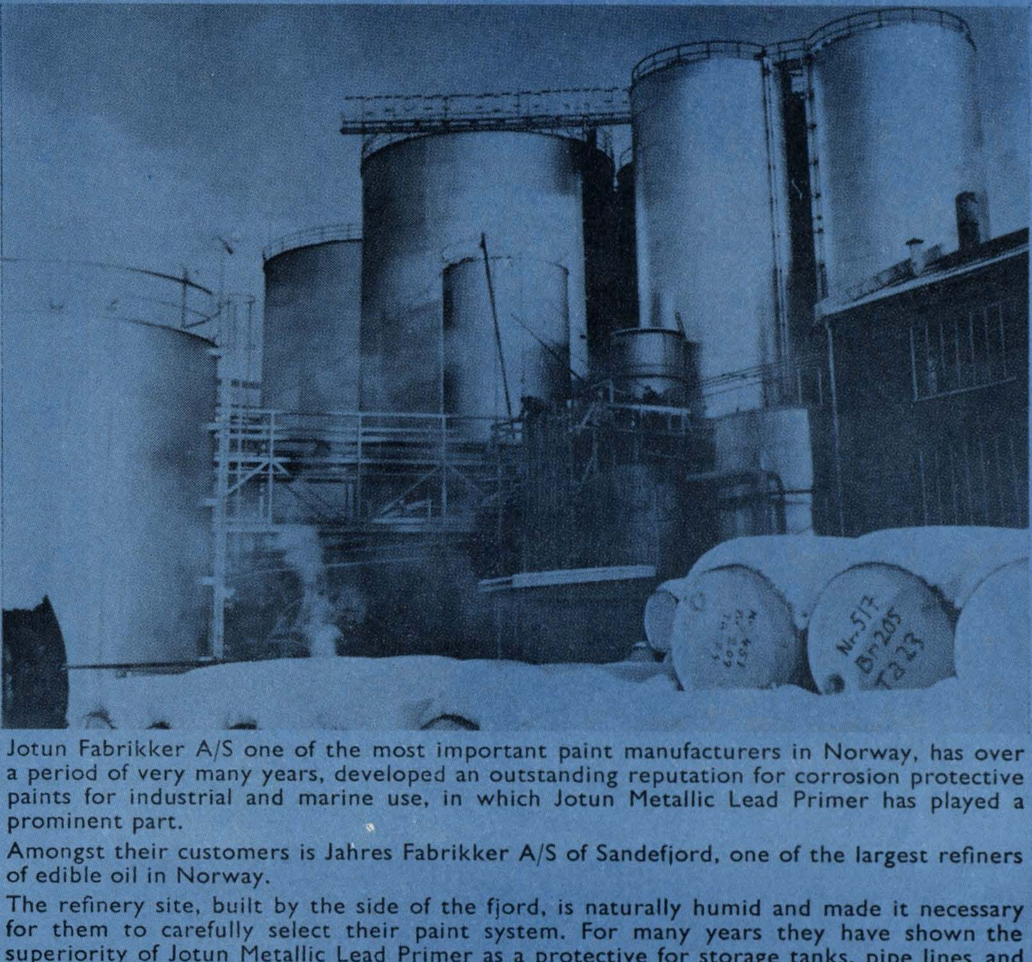

Nr. 517
Br 205
Ta 23

Table 3

Assessment of mould growth on flat enamel paints exposed at Lae, New Guinea

Paint	Upper side								Under side							
	6 months		9 months		13 months		16 months		6 months		9 months		13 months		16 months	
	I	C	I	C	I	C	I	C	I	C	I	C	I	C	I	C
1 a ..	3	90	0	0	9	5	9	5	5	90	6	100	6	100	7	100
b ..	3	90	0	0	9	5	9	5	6	90	6	100	6	100	7	100
1/A a ..	0	0	4	5	8	5	1-2	50	3	90	3-4	100	5	100	5	100
b ..	0	0	0	0	8	5	9	5	3	80	4-5	100	6-7	100	5-8	100
2 a ..	2	80	2	100	4	100	4	100	4	5	4	80	5	100	6	100
b ..	2	80	2	100	4	100	4	100	5	10	4-5	75	4-5	100	6-7	100
2/A a ..	0	0	0	0	8	5	3	5	3	20	2	20	2-3	80	3	100
b ..	0	0	0	0	8	5	2	100	0	0	2-3	10	3	60	2-3	100
3 a ..	2-3	40	2	55	2	50	3	100	5-6	10	4	80	4-5	100	6	100
b ..	3	30	2	50	2	50	2-3	100	4	10	3-4	80	5	100	6-7	100
3/A a ..	3	100	3	80	3	100	2	100	1-2	30	1	30	2-3	50	3	100
b ..	3	100	3	85	3	100	2	100	1-2	30	3	30	2-3	50	2-4	100
4 a ..	3-4	90	2-3	80	8	5	3	100	8-9	95	9	95	4-8	100	4-8	100
b ..	3-4	90	2-3	80	1-2	100	2-3	65	8-9	95	9	95	4-9	100	4-8	100
4/A a ..	2	100	2-3	70	3	100	1-2	100	6	95	6-7	100	4-9	60	5-9	100
b ..	3	100	3	70	3	100	1-2	100	6	95	5-6	100	8-9	100	5-8	100
5 a ..	3-4	90	2-3	80	8	5	5	30	8-9	95	9	95	4-8	100	7	100
b ..	3-4	90	2-3	80	8	5	6-8	50	8-9	95	9	95	4-9	100	4-8	100
5/A a ..	3	100	2-3	100	9	5	8	5	6	95	6-7	100	8-9	100	5-9	100
b ..	3	100	3	100	9	5	8-9	5	6	95	7-8	100	8-9	100	5-8	100
6 a ..	1	100	1	100	1	100	2	100	0	0	0	0	4	10	7	30
b ..	1	100	1	100	1	100	2	100	0	0	0	0	4	10	7	20
6/A a ..	3	100	1-2	100	1	5	1	15	0	0	0	0	1-2	85	3-4	100
b ..	3	100	1-2	100	1	5	2	30	0	0	2	10	4	30	5	100

I = Intensity ; C = Coverage, per cent ; /A means addition of fungicide.

Table 4

Assessment of mould growth on gloss enamel paints exposed at Lae, New Guinea

Paint	Upper side								Under side							
	6 months		9 months		13 months		16 months		6 months		9 months		13 months		16 months	
	I	C	I	C	I	C	I	C	I	C	I	C	I	C	I	C
1 a ..	3	100	2	100	3	100	2	100	3	10	3	100	4	100	3-4	100
b ..	3	100	2	100	3	100	2-3	80	4	10	3	100	5	100	4-5	100
1/A a ..	2	100	3	80	1	75	1-2	100	3	5	4	5	4	40	4	40
b ..	2	100	3	90	1	75	2	100	3	5	3	10	5	50	5	40
2 a ..	1	100	2	100	4	100	2	100	3	5	3	30	4	50	4-7	100
b ..	1	100	2	100	4	100	2	100	3	5	3	30	6	75	3-4	100
2/A a ..	2	100	1	100	1	100	1-2	100	3	5	4	10	4	40	4	40
b ..	2	100	1	100	1	100	2	100	3	5	4	15	4	50	5	40
3 a ..	3	100	2	100	3	100	2	100	3	30	3	60	4	100	4-5	100
b ..	3	100	2	100	3	100	2	100	3	10	3-4	80	5	100	4-5	100
3/A a ..	3	100	2	100	2	100	1-2	100	4	30	4	30	3	50	3-4	60
b ..	3	100	2	100	2	100	2	100	4	20	4	20	5	50	3	60
4 a ..	5	100	4	100	3	100	2	100	3-4	20	3	50	4	100	6	100
b ..	5	100	4	100	3	100	2	100	4	20	3-4	50	5	100	4-5	100
4/A a ..	3	100	3	100	3	100	3	100	6	20	3	30	4	50	4	60
b ..	3	100	3	100	3	100	2	100	6	20	4	30	5	50	5	55
5 a ..	1	100	0-1	100	1	100	1	100	4	5	3	15	4	100	5	100
b ..	1	100	0-1	100	1	100	2	100	3	5	3	15	4	100	4	100
5/A a ..	2	100	2	100	0-1	100	1-2	100	6	10	4	15	3-4	50	4	90
b ..	2	100	2	100	0-1	100	2	100	6	10	4	30	4	80	3	100

I = Intensity ; C = Coverage, per cent ; /A means addition of fungicide.

The conclusions drawn from the assessments depend, of course, on what is considered to be an important difference in the state of the panels. A difference in the intensity rating of two to three units in the middle of the scale and the extent of coverage from a certain degree onwards is really not of importance. It has been assumed that the percentage of coverage above which a difference becomes unimportant is 50 per cent. For instance, most people would judge the underside of the panels of paint 3 in Table 4 after 13 and 16 months equally objectionable, that is, a wall which shows this type of mould growth ought to be cleaned. On the other hand, formulations that show no mould growth or only very little (panel 2, paint 2, Table 3) seem to be of interest for further investigation. Again, formulations which show a 100 per cent coverage but only a very slight intensity (5 or 6 in Tables 3 and 4) would appear to be encouraging enough to justify further experiments.

Discussion and conclusions

The figures in Table 1 show that PMA, PMN, PMO and PMS disappear from the paint film very quickly, and it seems that the nature of the acid radical in the PM compound does not make any practical difference to this. In view of the results of O'Neill[4], who measured the rates of disappearance of cresylmercuric naphthenate, phenylmercuric octylphenate, and phenylmercuric *8* hydroxyquinoline from paint films and found that they all lost more than 50 per cent in three months, it seems unlikely that phenylmercuric compounds can be found which would be much more stable. The cause of the instability of this class of compound probably lies in the weakness of the carbon-metal bond, and this is unlikely to be influenced by the acid radical in the PM compound.

At the present time two methods are in use for evaluating fungicides in paint. The first is a laboratory technique carried out in a petri dish covered with agar containing a suitable nutrient. The paint to be tested is applied to a filter paper which is placed on the agar, and this is flooded with a suspension of spores and incubated. The formulation is accepted if very little or no mould grows on the filter paper. There are many variations of this test, mainly as to the substrate on which the paint is applied.

After many years of comparing the assessments of these laboratory tests with actual performance in the field most workers are probably convinced that no correlation exists. For instance, Reyne[5] has this to say about the method: " There is no correlation between results of these tests and the mildew resistance of these paints under conditions of use. We have records of paint containing 1½ oz of mildewcide which have failed the test, but did very well on a two-year exposure programme. Conversely, paints that passed the test and contained only ¼ oz of mildewcide per gallon of paint failed on exposure."

The failure of this test to predict actual field performance is not surprising if consideration is given to the vastly different conditions under which the testing is carried out compared with those prevailing in practice. For instance, the test does not take into account any loss of fungicide which may occur as a result of weathering. Some workers have tried to correct this by artificial weathering, but the correlation between the performance of paint films exposed to artificial and not real weathering is often poor, and artificial weathering studies are therefore rather unrewarding.

These laboratory tests should be used only to determine whether a compound has any fungicidal activity at all and whether or not further testing should be carried out.

The second method used is to expose test panels at sites where mould growth occurs. Recommendations for fungicides in paint are largely based on the outcome of these tests. However, there is no information on how far these exposure tests on panels agree with the actual behaviour of the mildewcides in the field. That such experiments have not been done is understandable, as they are very difficult to arrange in practice.

The only attempt known to the authors to correlate results on panels with a somewhat larger size experiment has been done by the Defence Standards Laboratories, Department of Supply, Melbourne[6]. A wall at Lae was painted with the various paints that had been tried out on the panels, but no correlation could be found. This attempt does not necessarily mean that nothing can be gained from such exposures, but it shows (*a*) the need to have some other indication to help to decide which of the paint formulations should be further tried, and (*b*) the desirability of trying those found promising on a more elaborate test fence trial.

(*a*) One of the authors has pointed out[1, 7] that for a fungicide to be effective a necessary but not a sufficient condition would appear to be a reasonable stability of fungicide in the paint film under similar conditions of exposure to those which the coating will eventually experience. Therefore it would seem that direct analysis of the fungicide in the exposed paint film could be helpful in making decisions as to the probable value of the fungicide in question. In the experiments reported here it can be seen from Table 2 that with two exceptions the addition of PMS did not make any great difference to the mould growth on the test panels. At the same time less than 22 per cent of the mercury was retained in the paint film on the upper side of the panels after exposure for 13 months, and less than 27 per cent was retained on the under side of the panels.

The great loss of fungicide in a comparatively short time and the little difference in the appearance of the panel makes it very probable that PMS at the level used will not be of great benefit in practice in any of the paints investigated, with the possible exception of the flat alkyd enamel paint containing 3 lb zinc oxide/gal. The behaviour of this paint is not due to a higher content of residual PMS, as can be seen from Table 2. The result could be chance, but the case requires further investigation.

The quantity of PMS added to the paints was considerably higher than that added by paint manufacturers. In view of the high losses of this fungicide in the paint film on exposure only a big increase in the amount added to the paint could possibly show any effect. However, economic considerations, the health danger of handling paints with a high concentration of mercury, and the possible incidence of staining[8] make such a course undesirable.

Table 1 shows that much different results from the other phenylmercury compounds investigated could not be expected, and taking into account the results obtained by O'Neill[4] it is reasonable to extrapolate to all phenylmercury compounds and state that the addition of this class of compound will not produce a worthwhile resistance to the growth of fungus on a paint film. Paint *F2* may possibly have to be excluded[9, 10] from this statement.

The conclusion that phenylmercury compounds are not very effective has also been reached by Whiteley[9, 10] and by workers at the Defence Standards Laboratories, Department of Supply[11], on the basis of panel exposures. The former estimates that the compound stays effective for 12 months and the latter found a loss of activity after 7 months.

An interesting result is shown for the paint formulations containing barium metaborate. Tables 3 (6) and 4 (5) show that these paints are somewhat less disfigured by mould than the other paints. They are not always free of mould, but the intensity on the upperside is rarely more than 1 and in the case of paint 6 (Table 3) there is (up to nine months) no mould growth at all on the underside, whereas paint 5 (Table 4) shows only little mould growth up to this time. It therefore would appear that barium metaborate should be further investigated.

(*b*) Once a paint formulation has been selected for further investigation on the basis of panel exposures the additional testing should be carried out before any recommendations are made.

The method envisaged uses a test fence approximately 3 ft from the ground and 7 ft high, built of a suitable material such as asbestos cement. The paint to be tested is applied to a strip 1 ft wide and the adjoining 1 ft strips are decorated with paint without active ingredients. This pattern takes into account the uneven and unpredictable distribution of mould growth over the height and width of a wall, and a fair judgment of the effectiveness of the additive under test may be made by comparison with the adjoining panels. If they have a heavier growth of mould it can be concluded that the formulation under test has some merit and can be recommended for large-scale use.

Acknowledgment

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New developments in the field of polyurethane lacquers*

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Summary

The features of light stable and weather resistant two-component biuret polyisocyanate lacquers based on hexamethylene di-isocyanate and polyesters are described. The influence of catalysts and solvents on the pot-life at different temperatures and binding media contents are discussed. The abrasion resistance of differently cross-linked lacquer films will be mentioned.

Développements récents dans la domaine des vernis polyuréthanes

Résumé

On décrit les propriétés particulaires des vernis Biuretpolyuréthanes, résistants aux intempéries et solides à la lumière, à base du di-isocyanate de hexaméthylène et des polyesters. L'influence exercée par catalyseurs et solvants sur la durée du pot aux températures diverses et aux teneurs différentes en liant est considérée. On mentionne la résistance à l'abrasion des feuils de vernis ayant des types variés de reticulation.

Neue Entwicklungen auf dem Gebiete der Polyurethanlacke

Zusammenfassung

Die Eigenschaften von Biuretpolyisocyanat-Zweikomponentenlacken auf Basis von Hexamethylendiisocyanat und Polyestern hinsichtlich Licht- und Wetterbeständigkeit werden beschrieben. Die von Katalysatoren und Lösungsmitteln bei verschiedenen Temperaturen und Bindemittelgehalten auf die Topfzeit ausgeübten Einflüsse werden besprochen. Die Abriebfestigkeit von auf verschiedene Weise vernetzten Lackfilmen wird ebenfalls erwähnt.

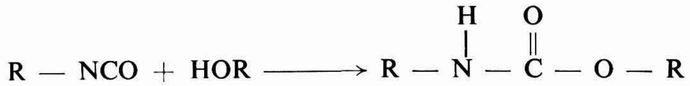
Новые развития в области полиуретановых лаков

Резюме

Описываются особенности установившихся легких, устойчивых против атмосферы, двухкомпонентных биуретполиизоцианатовых лаков основанных на гексаметилине ди-изоцианате и полиэфиров. Обсуждаются влияние катализаторов и растворителей на подогревание при разных температурах и на содержание связывающих средств. Упоминается сопротивление износу различных перекрестных лаковых пленок.

Introduction

As is known, the manufacture of polyurethanes is based on the reaction of isocyanate groups with alcoholic hydroxyl groups.



$$\mathrm{R - NCO + HOR \longrightarrow R - \overset{H}{\overset{|}{N}} - \overset{O}{\overset{\|}{C}} - O - R}$$

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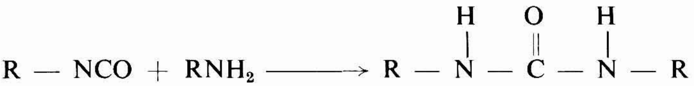
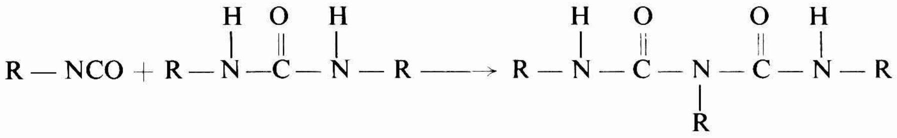
* Paper given to the Manchester Section on 12 March 1965.

In the presence of water or water vapour, the isocyanates form substituted urea compounds. Under certain conditions, these can be transformed to biuret compounds.

$$R - NCO + H_2O \longrightarrow RNH_2 + CO_2$$



$$R - NCO + RNH_2 \longrightarrow R - \overset{H}{\overset{|}{N}} - \overset{O}{\overset{\|}{C}} - \overset{H}{\overset{|}{N}} - R$$



$$R - NCO + R - \overset{H}{\overset{|}{N}} - \overset{O}{\overset{\|}{C}} - \overset{H}{\overset{|}{N}} - R \longrightarrow R - \overset{H}{\overset{|}{N}} - \overset{O}{\overset{\|}{C}} - \underset{R}{\underset{|}{N}} - \overset{O}{\overset{\|}{C}} - \overset{H}{\overset{|}{N}} - R$$

As starting materials for polyurethane products, the following di-isocyanates are, at the moment, of economic interest,

1. Toluylene di-isocyanate in its two isomeric forms ;
2. Diphenylmethane-4.4′ di-isocyanate.
3. Hexamethylene di-isocyanate.

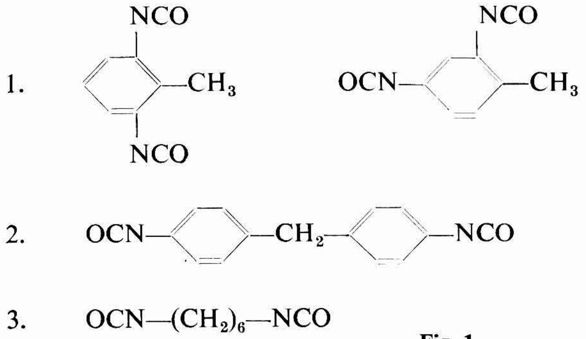


Fig. 1.

For coating purposes, toluylene di-isocyanate and hexamethylene di-isocyanate, as such, are unusable as they possess a high vapour pressure and are dangerous to health. Therefore, pre-adducts from these di-isocyanates are manufactured having a higher molecular weight which possess practically no vapour pressure. The amount of free di-isocyanate should be kept so low during processing that no physiological difficulties occur.

The following pre-reactions are usual at the moment :

1. Addition of a trivalent alcohol ;
2. Trimerisation to an isocyanurate ;
3. Reaction with water to a biuret compound.

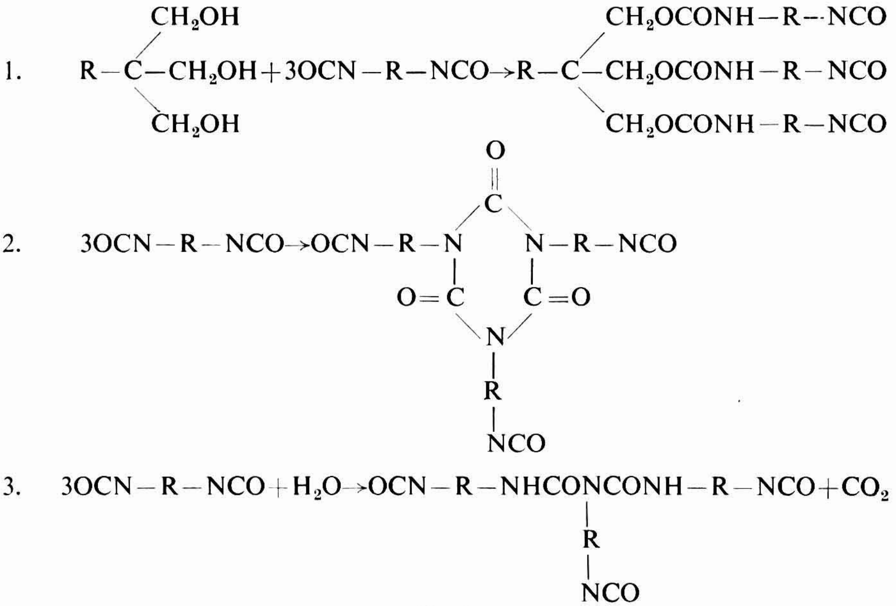
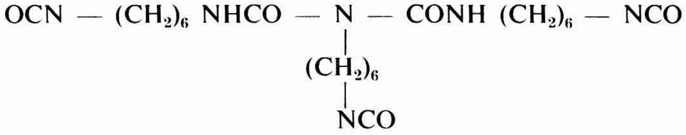


Fig. 2.

The vapour pressure of a diphenylmethane di-isocyanate is so low that it does not cause any health hazard.

Polyurethane coatings based on an aromatic polyisocyanate, show a marked tendency to yellowing and relatively early chalking as a pigmented system, when exposed to weathering. This is of secondary importance for a great number of applications especially where functional coatings are concerned. However, where high decorative demands are also made, aliphatic isocyanate compounds are to be preferred.

The oldest and most extensive experience as well as the best results so far, are with two-component coatings formulated with a polyisocyanate of biuret structure. The idealised structural formula of this compound built up from hexamethylene di-isocyanate is :



$$\mathrm{OCN - (CH_2)_6\ NHCO - \underset{\begin{array}{c}|\\(CH_2)_6\\|\\NCO\end{array}}{N} - CONH\ (CH_2)_6 - NCO}$$

The product in its 100 per cent form has approximately 22 per cent NCO groups and is still free flowing at ordinary temperature. The content of free di-isocyanate is below 0.75 per cent so that, given ordinary application conditions, there is no risk of hexamethylene di-isocyanate vapour escaping from the films.

The soft resin character of the aliphatic polyisocyanate and the lower rate of reaction as compared with aromatic isocyanates should be duly considered when selecting the hydroxylic co-reactant. To obtain a reasonable drying time, special resins should be used which, because of their drying characteristics, lead to the earliest possible dust-free time of the film. The OH groups should be located at a primary carbon atom since such hydroxyl groups are known to react with isocyanates much faster than those at secondary or tertiary carbon atoms.

Accelerators may be used to shorten the drying and curing times. The tertiary amines effective with aromatic isocyanates are unsuitable here, since their influence on the reactivity of the aliphatic NCO group is insufficient. Good results are obtained with certain metal compounds including particularly zinc octoate and naphthenate. Dibutyl tin dilaurate is one of the most effective activators. It will, however, be used only in exceptional cases as it drastically shortens the pot-life of the two-component mix. The usual zinc octoate level is of the order of 0.1 to 0.2 per cent on binding medium. With dibutyl tin dilaurate, however, a level as low as 0.01 to 0.02 per cent is sufficient. A standard combination of aliphatic polyisocyanate and a saturated polyester resin with approximately 8 per cent hydroxyl content is dust-free within three to four hours, and, in presence of 0.2 per cent zinc octoate, within 1½ to two hours. The ultimate hardness is reached after four to seven days in both cases. It is approximately 180 to 200 pendulum seconds as measured by the Albert Koenig method.

The change of pot-life in the presence of accelerators has great importance for the technician in the field. At a zinc octoate level of 0.2 per cent, a spraying formulation has a pot-life of approximately 12 to 20 hours, compared with 24 to 36 hours without accelerator.

The pot-life is, furthermore, influenced by temperature, concentration of the two-component coating and the nature of the solvent. Increased temperature results in a much faster drying of the coating so that satisfactory application conditions are obtained despite reducing the accelerator level. The difference in the times until the films are dust free, for example at 20°C and 30°C, lies between 1½ to 2 hours and 30 to 45 minutes.

Spraying formulations, as clear coatings, have an average solids content of 35 to 40 per cent. Brushing formulations, however, contain 50 to 60 per cent binder. This shortens the pot-life at equal accelerator level from approximately 12 to 20 hours to 3 to 6 hours (at room temperatures). With brushing formulations, therefore, the accelerator is normally omitted.

The influence of solvents on the pot-life is explained by the fact that polar, true solvents, such as lower ketones, esters and ether esters, for example, ethyl glycol acetate, have a better solvent power than, for example, the aromatics for the polyurethanes which are already forming in the coating composition during application. Such solvents, however, are popular as diluents for economical reasons. The higher their content in the solvent mixture, the shorter will be the pot-life.

The degree of normal potential cross-linking of the coating, has no bearing on the pot-life.

Tables 1-3 illustrate the dependence of the pot-life on the factors outlined in a coating comprising an aliphatic polyisocyanate (biuret structure)/phthalic polyester.

Table 1

Pot-life at different temperatures and varying zinc octoate contents : 40% binder

Zinc Octoate on binder	0°C	10-12°C	23°C	35°C	50°C
0	168 hrs.	107 hrs.	35 hrs.	24 hrs.	7 hrs.
0.1%	60 hrs.	23 hrs.	15 hrs.	6 hrs.	3 hrs.
0.2%	35 hrs.	16 hrs.	10 hrs.	5 hrs.	2½ hrs.
0.3%	17 hrs.	10 hrs.	8 hrs.	3 hrs.	2 hrs.
0.4%	8 hrs.	8 hrs.	2 hrs.	2 hrs.	1½ hrs.

Table 2

Pot-life at different temperatures and varying binder contents (0.2% zinc octoate as accelerator)

Binder content	0°C	10-12°C	23°C	35°C	50°C
20%	168 hrs.	135 hrs.	107 hrs.	40 hrs.	10 hrs.
30%	70 hrs.	33 hrs.	15 hrs.	10 hrs.	3½ hrs.
40%	40 hrs.	15 hrs.	8 hrs.	4 hrs.	2½ hrs.
50%	16 hrs.	6 hrs.	3 hrs.	2 hrs.	1 hr.

Table 3

Pot-life at different temperatures and varying solvents (40% binder, 0.2% zinc octoate)

Solvent	0°C	10-12°C	23°C	35°C	50°C
Methyl Ethyl Ketone	107 hrs.	28 hrs.	14 hrs.	5 hrs.	2 hrs.
Ethyl Glycol Acetate	132 hrs.	42 hrs.	16 hrs.	7 hrs.	3 hrs.
Ethyl Glycol Acetate/Xylene 7:3	38 hrs.	15 hrs.	4 hrs.	3 hrs.	2 hrs.
Cyclohexanone ..	107 hrs.	33 hrs.	12 hrs.	6 hrs.	3 hrs.

The importance of selecting the proper co-reactant for the aliphatic polyisocyanate has been discussed before. It is also of critical importance from the point of view of film properties.

Weather resistance, gloss retention and colour stability also are governed by the cross-linking polyol compounds. A yellowing resin is unlikely to form light-fast films even with aliphatic isocyanates. To achieve the desired optimum characteristics, it is necessary for the polyol compounds to possess similarly good properties. The best results have so far been obtained with phthalic polyesters. A resin with approximately 8 per cent OH groups and a softening point of 90 to 100°C may be considered to be the " standard " component. Polyether, and epoxy resins, are inadequate for chalking resistance. They are suitable hardeners, however, for finishes of maximum chemical resistance.

Outdoor exposure tests in a European climate on the North Sea and in the Alps have been under way now for over five years. After 4½ years, white pigmented films did not show any chalking. Finishes on weatherometer panels did not show any surface breakdown after 2,000 hours. They were tested in the Atlas Weatherometer type DLTS-X with one carbon arc lamp at continuous exposure to light and continuous spraying of the samples. The coated drum of a weatherometer was still satisfactory after 5,000 hours, after 6,000 to 7,000 hours the first chalking occurred, the white shade remained unchanged. Panels have been exposed to outdoor conditions for one to two years in Australia, South Africa, South America, Singapore and Florida. No chalking has so far been observed.

In the case of coloured coatings, it is essential to check with great care whether the pigments have the necessary degree of fastness. Only best rutile titanium dioxide should be used for white shades. With anatase types, the finishes exhibit chalking after outdoor exposure times as short as three to six months. A remarkable feature is the high pigment take-up in the standard formulation. With 150 per cent titanium dioxide, on binder, high gloss coatings are still obtainable. With organic pigments, the gloss is reduced at levels above 6 to 9 per cent.

When testing the colour stability of the aliphatic polyurethane coatings, it should be borne in mind that clear coatings on wood may often give the impression of a yellowing of the films when in reality the wood has stained, since UV radiation from the sun is but little absorbed by the film, and consequently its action on the wood is unrestrained. In practical applications it will be necessary to include UV absorbers in such cases. Some UV lamps, used for short-term light fastness tests, may also result in incorrect findings on the tendency of the coating to yellow. It is necessary to know the UV spectrum of the instrument. In daylight, the range is from 300 to 400 millimicrons, but shorter wave radiation may affect the materials which have very good stability on exposure to sunlight.

The high abrasion resistance of the aliphatic polyurethane films is another remarkable property. The weight losses with the Taber Abraser apparatus after 1,000 turns of the CS 10 roll under 1 kilo load are listed in mg in Table 4.

Table 4

Length of exposure	Degree of cross-linking (Cross-linking agent as % theoretical)		
	70%	100%	130%
After 14 days	32 mg.	23 mg.	20 mg.
After 30 days	15 mg.	11 mg.	8 mg.
After 60 days	20 mg.	11 mg.	9 mg.

These are the results obtained with a standard formulation which gives very tough films. Optimum resistance is reached only after two to four weeks. In all probability, it is only after this time that the solvents have completely left the film. The abrasion resistance increases with cross-linking. In the extreme case, the polyisocyanate without polyester addition would react with the atmospheric moisture to form polyurea. The abrasion data then are summarised in Table 5 (1,000 turns, CS 10, 1 kg).

Table 5

Aliphatic polyisocyanate reacted with atmospheric moisture to give polyurea :

Length of exposure	(a) without accelerator	(b) with accelerator
After 3 days	9 mg.	5 mg.
After 14 days	3 mg.	3 mg.
After 1 month	4 mg.	4 mg.
After 2 months	4 mg.	4 mg.

Another valuable feature which should not be overlooked, is the good solvent resistance of the polyisocyanate/polyester film. It is believed to head the list of all films drying at room temperature. While alcohols and alcohol/water mixtures soften the film after approximately one week, it regains its initial hardness after drying in the air.

Moisture cured aliphatic one-component coatings are, in theory, obtainable. At present, the available experience is insufficient to allow detailed information on this subject. Suitable drying times of the polyurea films are obtainable by including catalysts which do not influence the shelf life of the polyisocyanate coatings during times of, so far, seven months.

The production of urethane oils with hexamethylene di-isocyanate instead of toluylene di-isocyanate have so far not given any special advantage. The oil or alkyd character is so predominant that the relatively small portion of the aliphatic isocyanate is unable to bring about a fundamental change of the property pattern.

Blocked aliphatic polyisocyanates could be valuable for stoving finishes. The phenol customarily used with aromatic polyisocyanates, for this purpose, however, would be unsuitable since it leaves a slight yellowing after stoving. Alcohols such as butanol, on the other hand, are linked so firmly to the aliphatic isocyanates that the stoving temperature would have to be very high and the times would have to be so long as to affect the colour stability of the film. (Stoving at 200°C for half-an-hour to one hour would be required.)

[*Received 12 March 1966*

Discussion

Mr. Woolrich asked, as the vast majority of examples given in the paper were of application on metal surfaces, if the same weathering properties applied to clear lacquers on bare wood. In his experience, poor adhesion resulted from the application of polyurethane lacquers to wood.

Dr. Mennicken replied that the intrinsic durability of the light-fast pigmented polyurethane lacquers had been established for over five years on laboratory panels. Weather durability of clear varnishes on wood depended on several conditions, including the type and quality of the substrate, number of coats, pretreatment of the wood, and care of application. Clear varnishes on test panels carefully brushed showed good results for about three years in a normal European climate. In practice, the difficulty with wood was the inability to seal completely the wood to prevent the ingress of moisture. If the wood was not completely sealed, eventually the pressure of the water from the wood would cause loss of adhesion between the film and the wood although the film itself remained intact. In fact it had been found that loss of adhesion was in many cases due to a breakdown in the surface of the wood, that is, the cohesion of the film itself is better than the adhesion between the cells and layers of the wood.

MR. A. C. JOLLY asked if the non-yellowing polyurethane systems were sensitive to any film laid down on a substrate prior to application of the polyurethane.

DR. MENNICKEN said that, for economic reasons, light fast polyurethane lacquers were generally only used for the top coats. The primer could be formulated with any other conventional materials, which were not badly affected by the solvents of the polyurethane lacquers.

MR. JOLLY enquired whether in this case any differences would be shown up in lack of intercoat adhesion at a later stage.

DR. MENNICKEN replied by saying that providing the primer was resistant to the solvent used in the polyurethane material the results would be quite satisfactory. If the solvent resistance of the coating which was being overcoated by the polyurethane lacquers, was sufficient to resist the polyurethane lacquer during application and drying, intercoat adhesion was generally found to be satisfactory.

MR. JOLLY also asked whether there was any difference found by substituting T.M.E. for T.M.P. in either or both the polyol-isocyanate adducts and polyesters.

DR. MENNICKEN replied that, although theoretically there would not be a difference, this substitution had not been investigated.

MR. J. J. KAVANAGH enquired if zinc octoate was preferred to stannous octoate on technical or commercial grounds as a catalyst for lacquer systems of this type.

DR. MENNICKEN said that in his experience stannous octoate was not as effective as zinc octoate which was more familiar and available. He had not, however, carried out a complete review of all the possible catalysts.

MR. C. BARKER asked if the yellowing and chalking which occurred with aromatic isocyanates was due to the same mechanism of degradation and what was the mechanism of yellowing with aromatic isocyanates which differed from that of aliphatic isocyanates which the lecturer stated did not yellow?

Dr. MENNICKEN said that it was difficult to give a completely satisfactory explanation to this question of yellowing. Ideally the aromatic isocyanates were completely cross-linked with no remaining reactive groups. It was possible, however, that there could be some saponification which could give rise to aromatic amines which would produce coloured compounds, and this was a possible explanation of yellowing. This was probably due to unreacted NCO groups which could react with moisture giving the aromatic amine and liberating carbon dioxide. In this way it was possible for the films both to yellow and to break down resulting in chalking.

Mr. W. F. MCDONNELL asked Dr. Mennicken to explain why the urethane oils had better water resistance and alkali resistance than conventional oil modified alkyds.

DR. MENNICKEN replied that this was due to the greater alkali resistance of the urethane linkage compared with the ester linkage. The ester group was easier to saponify.

MR. T. E. JOHNSON referred to the use of moisture-cured polyurethanes on wood. There could be some reaction between the coating and the moisture contained in the wood. He asked if there were problems involved in obtaining adhesion with these materials when recoating after a period of, say, two years' exterior exposure.

DR. MENNICKEN replied that the adhesion when overcoating with moisture cured systems was bad, especially after a longer period, say more than one week after the application of the first coat. Improved results could be obtained by sanding the surface.

MR. BATTERSBY asked what was the toxic effect of isocyanate when it did occur?

DR. MENNICKEN replied that the toxic symptoms were generally an inflammation o the mucous membrane. This was due to the vapour of certain isocyanates or to the inhaling of aerosol droplets when polyurethanes were sprayed. In this case ventilation would be necessary, not only for the aerosol droplets, but also for the exhaustion of the solvents.

MR. BATTERSBY also asked if there was a difference between moisture-cured and two-pack systems in their toxic symptoms as he personally had experienced differences between the two. He had found that the one-pack moisture-cured materials had a considerable effect whereas the two-pack materials appeared to have little or no effect.

DR. MENNICKEN replied that the toxicity depended on the vapour pressure of the isocyanate material used and the three types mentioned in his paper were all non-toxic in paints and varnishes applied under normal conditions. The content of volatile isocyanates could be held so low that no hazard was to be expected. He considered it was the responsibility of paint manufacturers to ensure the correct material was used, otherwise it was possible that polyurethane products as a whole could acquire a bad reputation.

MR. R. S. MONK asked if moisture scavengers could be used to produce a stable pigmented one-pack system.

DR. MENNICKEN replied that this was theoretically possible ; the problem was to obtain the pigment in a completely dry form. This could be achieved in several ways, for instance by heating or by dispersion in solvents with moisture absorbing materials such as molecular sieves or by the use of other moisture scavengers. Although these methods were feasible theoretically, in practice none of them had so far proved entirely satisfactory. Another possibility was to react the pigment initially with an isocyanate to ensure that all free moisture was removed before pigmenting the base material.

MR. A. C. JOLLY asked if Dr. Mennicken would confirm that the moisture-cured isocyanates had a lower toxic hazard than the two-pack isocyanate material because of the higher concentration of isocyanate in the two-pack systems.

DR. MENNICKEN said that this was not necessarily the case. It was dependent on the isocyanate materials used.

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Surface properties of titanium dioxide pigments*

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Summary

The adsorption on to titanium dioxide pigments of pure compounds representing the functional groups of an alkyd resin was followed by construction of the isotherms and by infra-red spectroscopic examination of the pigment adsorbate complex. It was shown that the nature of the carboxyl/pigment interaction was related to the presence or absence of inorganic surface treatment on the pigment. The hydroxyl/pigment interaction appeared to be unrelated in type to the presence of surface treatment.

Propriétés superficielles des pigments du dioxyde de titane

Résumé

On a étudié l'adsorption par pigments du dioxyde de titane des composés purs—représentant les groupements fonctionnels d'une résine glycérophtalique—au moyen de dressage des isothermes et l'analyse spectroscopique dans l'infra-rouge du complexe du pigments et de l'adsorbat. On démontre que la nature de l'interaction du pigment et le groupement carboxylique dépend de la présence ou de l'absence de traitement superficiel inorganique du pigment. L'interaction du pigment et le groupement hydroxylique se comporte indépendament de la présence de traitement superficiel.

Eigenschaften der Oberflächen von Titandioxidpigmenten

Zusammenfassung

Nach Adsorption chemisch reiner Verbindungen, welche die funktionellen Gruppen eines Alkydharzes darstellen, auf Titandioxidpigmenten folgte der Aufbau der Isothermen und die infrarot—spektroskopische Untersuchung des Pigmentadsorbat-Komplexes. Es wurde gezeigt, dass die Art der Reaktion zwischen Karboxylgruppe und Pigment davon abhing, ob die anorganische Pigmentoberfläche behandelt worden war. Scheinbar steht die Art der Reaktion zwischen Hydroxylgruppe und Pigment in keinem Zusammenhang mit stattgefundener Oberflächenbehandlung.

Поверхностные свойства грунтовых красок на базе двуокиси титана

Резюме

В статье описывается адсорбция на грунтовых красках, на базе двуокиси титана, чистых соединений функциональных групп алкидной смолы, и затем следуют конструкция изотермов и инфракрасный спектроскопический анализ комплекса адсорбционной грунтовой краски. Показано что характеристика взаимодействия карбоксил/грунтовая краска связана с присутствием или отсутствием неорганической поверхностной обработки грунтовой краски. Взаимодействие гидроскил/грунтовая краска повидимому не имеет типового отношения к присутствию поверхностной обработки.

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*Presented to a joint meeting of London Section OCCA and the Colloid and Surface Chemistry Group, SCI on 10 January, 1966.

Introduction

Before pigment/medium interactions and their possible effects upon dispersion phenomena may be explored it is essential to obtain an understanding of the nature of the pigment surface and the possible reactions into which this surface may enter with the components of the medium. In the present paper some of the studies carried out as part of a general research programme at the Paint Research Station are described.

When a pigment is introduced into the medium a series of inter-related interfacial reactions will be initiated. These will include competing adsorption/desorption phenomena, some of which may be specific for certain medium components. A process of this type is believed to be responsible for an occasional paint instability, the medium being depleted by adsorption on to the pigment of material, which when present assisted in keeping the high molecular weight fraction in solution.

Interfacial processes are frequently extremely sensitive to the presence of impurities in the system. The list of possible impurities in a paint system is extremely large, but one substance, water, may be considered as a universal contaminant. Practically all pigments and solvents and frequently also the medium[1] contain water to an extent which must be considered significant bearing in mind its polarity and low molecular weight. This water might compete with the medium components for active sites on the pigment surface in the extreme case blocking the surface to the medium, or it may combine with the pigment resulting in a surface differing from that expected from the stated composition. An example of the possible water content of a pigment may be drawn from the present study where it was found that a widely used grade of titanium dioxide contained, over several batches, easily removable water equivalent to more than three monolayers surrounding the pigment particle. On this basis a gallon of gloss paint could contain about 60 g of water from the pigment alone.

The classification of alkyd covers a wide range of related complex surface coating resins consisting essentially of the reaction products of drying oil fatty acids, a dibasic acid and a polyhydric alcohol ; frequently, but not exclusively, the dibasic acid is *o*-phthalic and the polyhydric alcohol glycerol or pentaerythritol. The proportions of the components and the extent to which reaction is taken may be varied over wide limits according to the intended end use of the resin. The complexity of these resins made a direct examination of pigment/medium interaction exceedingly difficult. To overcome this, simple materials were used as type compounds representing various functions present in the resin such as carboxyl, hydroxyl, ester and chain unsaturation in order to obtain a suitable background of information for use in later studies involving materials more closely resembling actual resins.

For pigmentary purposes both the anatase and rutile crystal forms of titanium dioxide are employed with a TiO_2 content varying from about 93 to 99 per cent, the remainder mainly consisting of deliberate additions made to control various properties of the pigments[2, 3]. These additions are mainly present as surface treatments, hence a relatively small concentration based upon the total pigment may in fact represent a high concentration on the pigment surface. Rutile pigments frequently contain a zinc modifier (present to about 1 per cent as

ZnO) added to the slurry before calcination. The more usual surface treatment materials, added after calcination, are alumina and silica, though hydrous titania is not uncommon[2]. Recently organic surface treatment materials have been incorporated in certain grades of pigment ; these materials were not included in the present study.

A technique for the infra-red spectroscopic examination of adsorbed material on a pigment surface was developed at the Research Station by I. T. Smith[4]. This technique has now been extended and combined with adsorption methods in the examination of the interactions between the type compounds and the titanium dioxide pigments.

Experimental

Materials

The adsorbates were generally of Eastman White Label, BDH Specially Pure or Fluka Puriss Grades and where necessary were purified further by recrystallisation and chromatographic techniques. Purities were checked by GLC and in all cases were appreciably better than 99 per cent. Carbon 14 labelled stearic acid (stearic acid—1-C14) was supplied by the Radio-chemical Centre in benzene solution and was used without further purification.

Benzene, of a laboratory pure grade, which had been treated with calcium hydride and fractionated was used as the main solvent. No water could be detected by the Karl Fischer method. This solvent was not ideal, but the choice was dictated by future sections of the programme where low solubility of alkyd resin components in an aliphatic solvent would be an embarrassment.

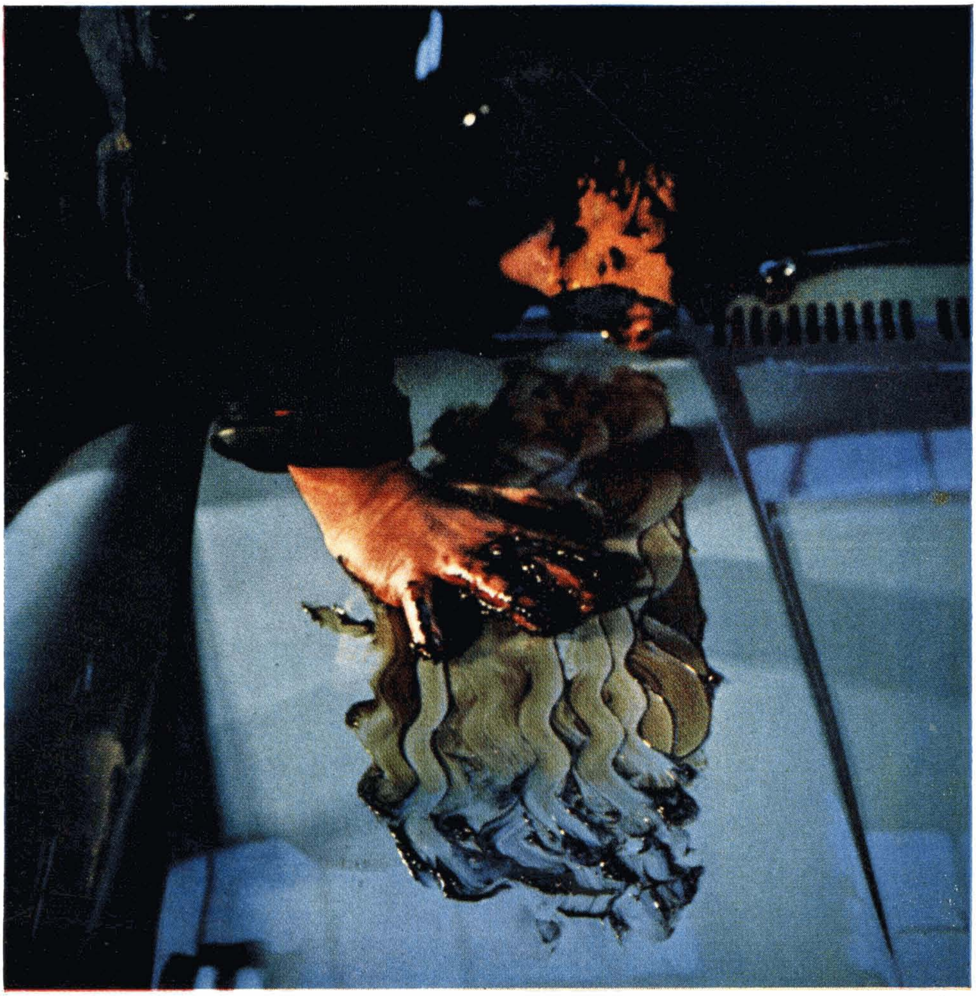
In this investigation all of the pigments were sulphate process production materials and were chosen to cover a wide range of types. The modifier and surface treatment indicated in Table 1 were as given by the manufacturers. The surface areas were BET nitrogen adsorption figures.

Table 1

Pigment Types

Pigment	Type	Modifier and surface treatment			TiO_2 content (%)	Surface area m^2/g
5*	Rutile	Zn	Al	Si	93	11.4
6a	,,	Zn	Al	Si	94	9.5
7a	,,	Zn	—	—	97	5.9
20	,,	—	Al	Si	95	10.0
7c*	,,	—	—	—	99	7.2
8	Anatase	—	Al	Si	95	13.2
9	,,	—	Al	—	95	9.5
10b*	,,	—	—	—	98	9.5
10x	,,	—	—	—	99	12.0
21	,,	—	Al	Si	97	13.0

The main pigment types investigated are marked with an asterisk in the table. The remainder were included to elucidate various points which arose.



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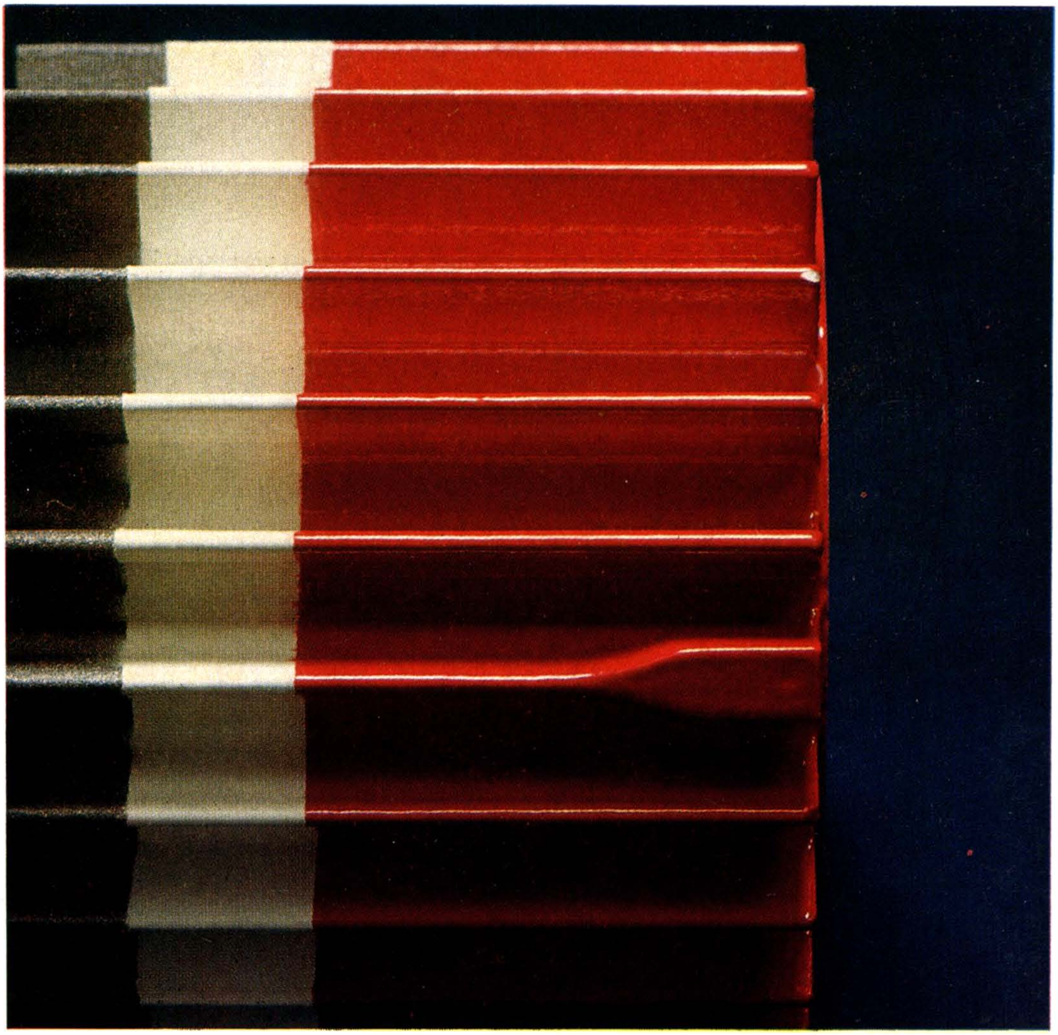


Badische Anilin- & Soda-Fabrik AG Ludwigshafen am Rhein

As received the pigments contained water, a greater proportion being present in the complex types than in the simple ones. Over several batches the water content of the various grades was found to be remarkably constant.

It was assumed that a pigment in equilibrium with an atmosphere of 60 per cent relative humidity at 25°C could be considered as an average normal material. This was justified by a comparison of the water content of the pigments as received and after the equilibration. Pigments so equilibrated are designated " normal " in this paper.

To follow the effects of water upon adsorption processes two further conditioning treatments for the pigments were chosen :

To obtain a reference condition where all easily removable water was absent, the pigments were heated at 150°C to constant weight—these were designated " dried " materials. The temperature of 150°C was chosen as being unlikely to result in permanent changes in the pigments particularly the complex types. The " dried " pigments were hygroscopic and rapidly readsorbed moisture from the atmosphere returning in all cases to materials indistinguishable from the original pigments. These dried pigments were not anhydrous in the academic sense ; heating at temperatures appreciably above 150°C resulted in further weight loss, but the process was no longer reversible for the complex pigment types.

The final conditioning method was chosen to demonstrate the effects of gross adsorbed water upon the pigment properties. It consisted of exposing the pigments in a closed vessel over an open dish of water at 25°C. These pigments, though designated " wet," were not visibly wet, but remained free flowing powders.

The water contents of some of the pigments as supplied and after conditioning are shown in Table 2.

Table 2

Water contents of pigments
(Weight loss at 150°C)

Pigment	As supplied	" Normal " treatment	" Wet " treatment
5	11 mg/g	11 mg/g	44 mg/g
7a	4 ,,	4 ,,	120 ,,
7c	2 ,,	2 ,,	61 ,,
10b	3 ,,	3 ,,	36 ,,
10x	3 ,,	3 ,,	69 ,,

Procedure

The experimental procedure essentially consisted of a series of adsorption experiments followed by recovery of the adsorbent and infra-red spectroscopic examination of the adsorbent/adsorbate complex.

The adsorption experiments were carried out using a constant ratio of conditioned pigment to adsorbate solution with in all cases an excess of adsorbate. Except where gross adsorbed water was present, seven days was normally allowed for equilibration ; under the " wet " condition up to 30 days was required. The equilibrium solutions were assayed by titration in the case of acids and gravimetrically for the alcohol and ester. Radioactive stearic acid was assayed, adsorbed on a tissue disc, by means of a " windowless " scintillation counter.

For the infra-red examination, the pigments were separated from the equilibrium solution by rapid filtration followed by washing a portion of the recovered pigment with n-hexane to remove loosely held material. Both the washed and unwashed portions of the pigment were examined.

The spectra were all obtained using a Unicam S P 100 spectrophotometer. The self-supporting disc technique used for presenting the samples to the instrument in the earlier work was largely replaced by mulls in Fluorolube and Nujol and a potassium bromide reinforced disc technique.

The latter method, which consisted of placing a layer of potassium bromide in the die and sprinkling an even layer of the sample on top and then pressing, differed from the usual potassium bromide disc technique in that no mixing or grinding together of the pigment and potassium bromide occurred. This method was adopted as it was found that grinding with potassium bromide could cause changes in the sample which were dependent upon the time of grinding.

The frequency ranges covered by these methods were :

Nujol mull—
 375-700 cm^{-1} and 1500-1800 cm^{-1}.

Fluorolube mull—
 1300-1800 cm^{-1} and 2700-4000 cm^{-1}.

Reinforced disc—
 375-4000 cm^{-1}.

Results

Spectroscopic examination of the pigments

The spectra of the pigments alone showed a sharp band in the region of 3700 cm^{-1} due to surface hydroxyl which was most distinct for the simple pigments (Fig. 1). After the adsorption of stearic acid no free hydroxyl groups could be detected. Free water when present could be observed at 1620-1640 cm^{-1} and near 3400 cm^{-1}. The band at 1620-1640 cm^{-1}, due to the presence of liquid water, was not found in the spectra of the pigments dried at 150°C with the exception of pigment 5. This pigment as supplied by the manufacturers contained more water than the other pigments, presumably due to the extensive surface treatment.

It was possible to distinguish between the anatase and rutile pigments by the variations in bands in the far infra-red region. The rutile pigments showed three distinct bands in the regions of 430, 535 and 660 cm^{-1} whilst the anatase pigments showed only two bands at 475 and 690 cm^{-1} (Fig. 2). A further band in the region of 350 cm^{-1} was known to be present in both spectra[5], but this was beyond the range of the S P 100 instrument.

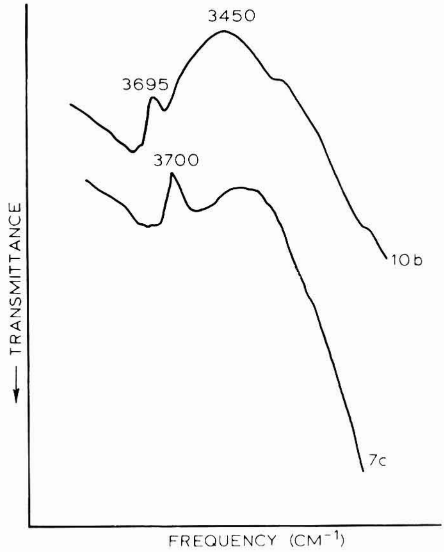


Fig. 1. Surface hydroxyl on pigments 7c and 10b

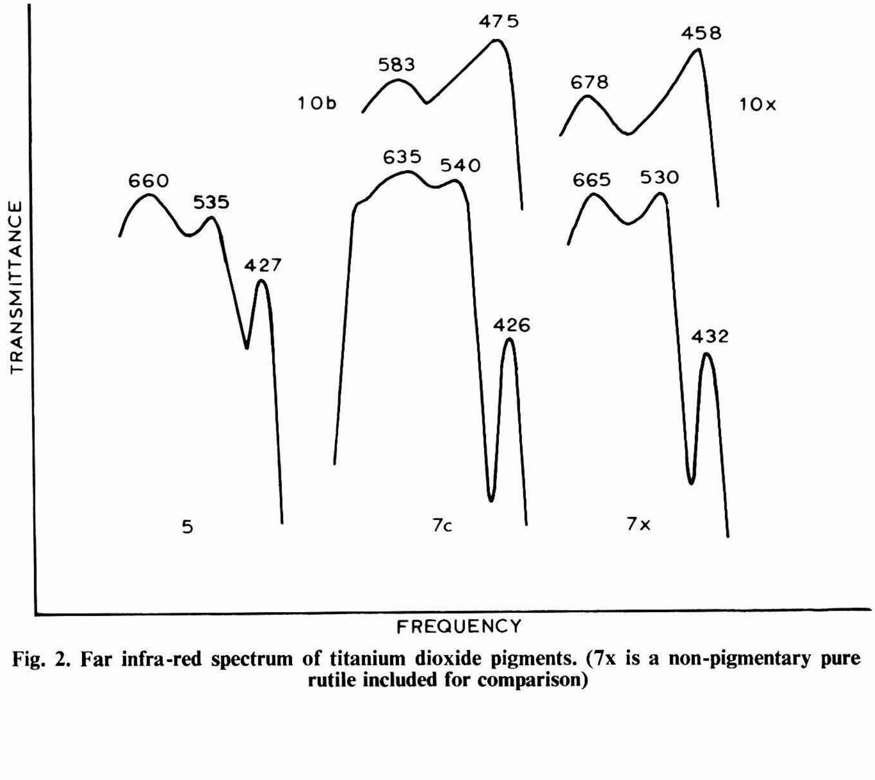


Fig. 2. Far infra-red spectrum of titanium dioxide pigments. (7x is a non-pigmentary pure rutile included for comparison)

Adsorption of stearic acid reduced the intensity of the highest frequency band for both pigment types. Removal of the adsorbed acid resulted in its reappearance.

This could be interpreted as a decrease in the polarity of metal-oxygen bonds associated with the surface of the pigment by the presence of the adsorbed molecule. No similar change in band intensity was produced by the adsorption of water on to the pigment surfaces.

Adsorption of stearic acid

Stearic acid was chosen to represent the carboxyl group with, it was hoped, a minimum of influence by the remainder of the molecule. One aspect in which this ideal was not realised was in the effect of the hydrocarbon chain on solubility. This was low—2 per cent solutions being essentially saturated.

Isotherms for the adsorption of stearic acid on to four representative pigment types are shown in Figs. 3-6.

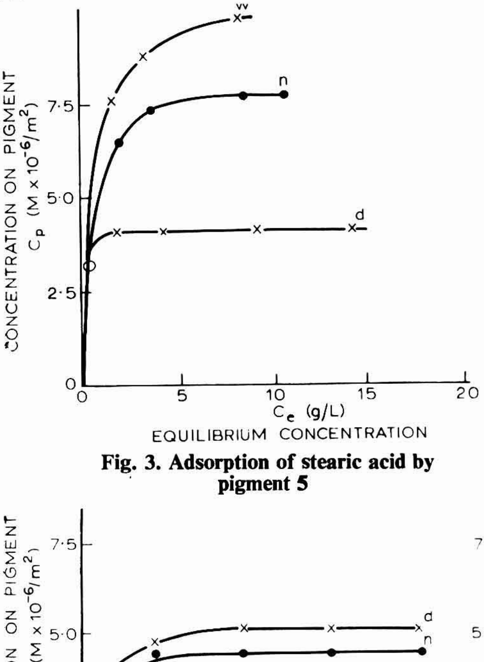


Fig. 3. Adsorption of stearic acid by pigment 5

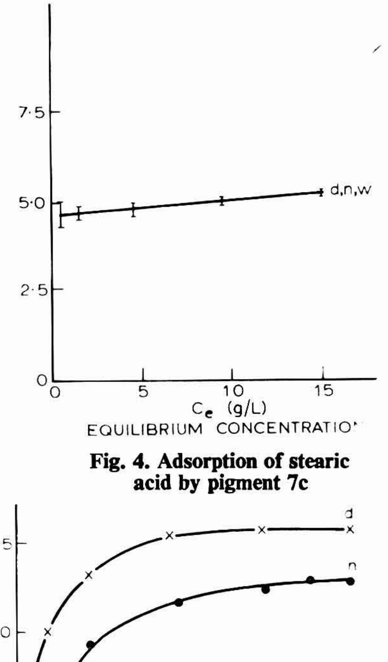


Fig. 4. Adsorption of stearic acid by pigment 7c

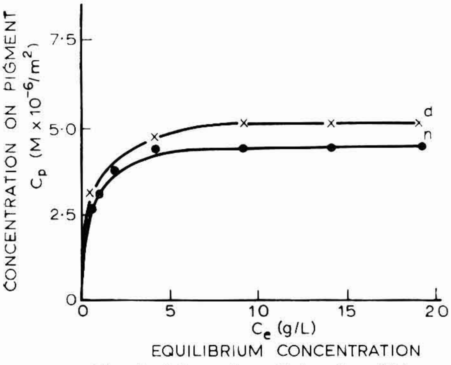


Fig. 5. Adsorption of stearic acid by pigment 8

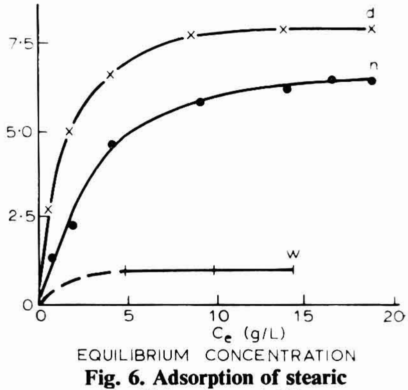


Fig. 6. Adsorption of stearic acid by pigment 10b

The steep initial slope of the isotherm for adsorption by the complex rutile pigment indicated a high affinity of the surface for the acid. This was confirmed by the use of alcohol as solvent instead of benzene in an adsorption experiment. It was considered that the high concentration of hydroxyl from the solvent would swamp adsorption sites on the pigment surface and largely prevent adsorption of the acid unless a specific affinity existed for the acid. Under this condition the complex rutile pigments adsorbed an appreciable quantity of stearic acid, but for the simple pigment types no adsorption could be detected.

In all cases the isotherms for the adsorption of stearic acid from benzene solution had a long flat region indicating that there was little tendency for multiple layer formation.

Increasing the concentration of water on the complex rutile pigment not only increased the quantity of acid adsorbed but also increased the time required for equilibration (Fig. 7). The effect upon the equilibration rate of the simple anatase pigment though was only marginal.

Exchange reaction between carbon-14 labelled stearic acid adsorbed on to the pigment surface and unlabelled stearic acid in solution were used to demonstrate differences in the attachment of the acid to the pigment surfaces. These experiments, which were carried out under conditions such that a labelled molecule leaving the pigment surface was unlikely to become reattached, showed a large difference between the simple anatase and complex rutile pigments. For the simple anatase the process was rapid, being essentially completed in one hour, whereas for the complex rutile the exchange was slow and of an exponential form, about 85 per cent reaction being attained only after 200 hours (Fig. 8).

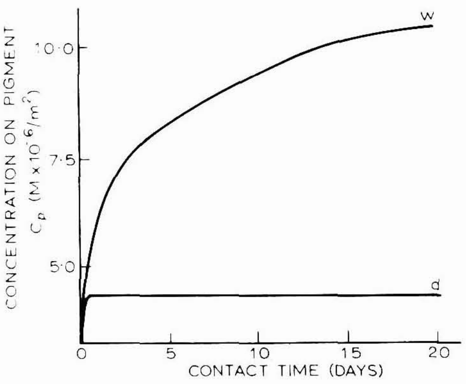


Fig. 7. Effect of water on adsorption of stearic acid by pigment 5

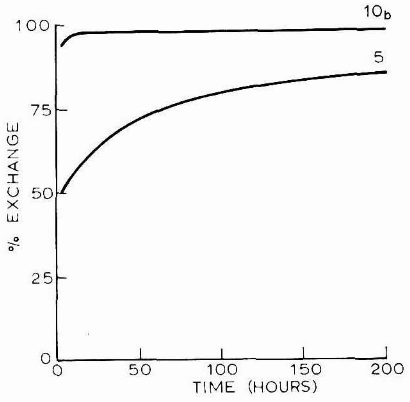


Fig. 8. Exchange of adsorbed stearic acid

Some indication that the strength of the acid/pigment linkage might be related to the strength of the adsorbed acid was given by exchange experiments involving a phthalic acid half ester, a strong acid of a type present in an alkyd resin. It had already been demonstrated[4] that the fundamental acid/pigment interaction of this type of acid was essentially similar to stearic acid, but in exchange processes it was found to displace stearic acid rapidly from both types of pigment surface with little evidence that it, in turn, could be displaced by stearic acid.

In the initial infra-red spectroscopic examination of adsorbed materials on pigment surfaces it was shown[4] that stearic acid could be adsorbed onto titanium dioxide pigments by two mechanisms—the formation of a carboxylate ion (i.e., ionic adsorption) or adsorption of the free acid by hydrogen bonding (acidic adsorption). These two processes are readily differentiated in the infra-red spectrum ; the carboxyl band at 1702 cm^{-1} for solid stearic acid being replaced by two carboxylate ion bands at lower frequencies in which the asymmetric stretching band is stronger than the symmetric. The exact frequencies of these bands are dependent upon the metal with which the ion is associated.

The spectra of the carboxyl region for adsorbed stearic acid on six of the pigment types showed that both the ionic and acidic processes were involved (Fig. 9). Both of the zinc modified rutile pigments (5 and 7a) showed a strong carboxylate ion band at 1540 cm^{-1}. Some of acidic form, as shown by the band at 1704 cm^{-1} was also present in the simpler pigment, 7a. The two surface treated anatase pigments showed the presence of both ionic ($\approx$1590 cm^{-1}) and the acidic (1706 cm^{-1}) forms with the proportion of the acidic form greater on the pigment carrying the simplest surface treatment.

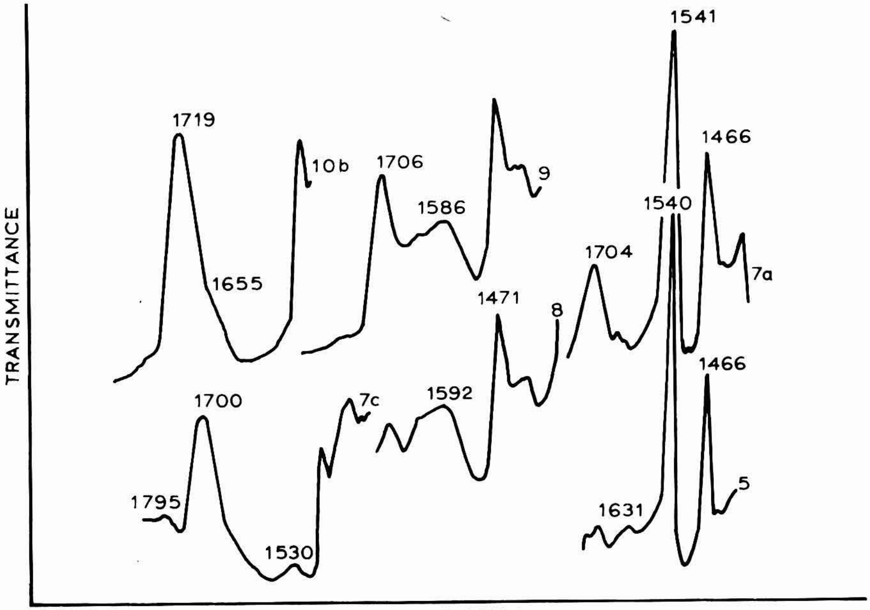


Fig. 9. Spectra of pigments with adsorbed stearic acid

Acidic adsorption predominated on the two simple pigments though a small band due to ionic material was observed at 1530 cm^{-1} for the rutile pigment 7c.

After washing the pigment/adsorbate complex with n-hexane only the more strongly held material remained on the pigment (Table 3).

Table 3

Form of stearic acid remaining on " normal " pigments after washing with n-hexane

Pigment	Material remaining after washing
5	ionic
7a	ionic with trace of acidic
7c	acidic with trace of ionic
20	ionic
8	ionic
9	ionic with trace of acidic
10b	acidic with trace of ionic
10x	trace acidic and trace ionic

Increasing the water content resulted in an increase in the proportion of the ionic adsorption on all of the pigments. Adsorption of stearic acid by " dried " pigment 5 resulted in a spectrum which was distorted in the 1500-1700 cm^{-1} region and showed two small peaks at 1544 and 1570 cm^{-1} (Fig. 10). The increased water content of the " normal " pigment resulted in the appearance of the strong ionic band at 1540 cm^{-1}. A further increase in water content further enhanced this band and also a shoulder appeared on the band at 1590 cm^{-1}.

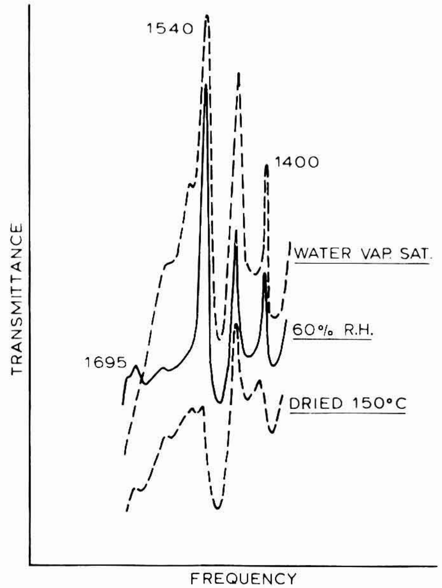


Fig. 10. Effect of water on spectrum of adsorbed stearic acid on pigment 5

The main carboxyl band frequencies found for stearic acid adsorbed onto several of the pigment types varied with the pigment composition (Table 4).

Table 4

Band frequencies of the carboxyl region for adsorbed stearic acid

Pigment	Modifier and surface treatment			Acidic material	Ionic material
5 R	Zn	Al	Si	1715-1695 W	1540 S
7a R	Zn	—	—	1704	1541
20 R	—	Al	Si	—	1585
7c R	—	—	—	1713-1700 S	1530 W
8 A	—	Al	Si	1706	1592
9 A	—	Al	—	1706	1586
10b A	—	—	—	1723 S	1555 VW
10x A	—	—	—	1718	1530 VW

R—Rutile A—Anatase

When zinc was present in the pigment the frequency of the carboxylate ion band was 1540 cm^{-1} even when, as in the case of pigment 5, a considerable amount of other surface treatment material such as alumina was present. This band frequency of 1540 cm^{-1} was also found both for stearic acid adsorbed onto zinc oxide and for zinc stearate. In the absence of zinc, the surface treated pigment showed a stearate ion band in the region of 1585 cm^{-1} which corresponded to that found for aluminium stearate. The similarity between the ion band frequency found for the zinc free surface treated rutile pigment 20 and the surface treated anatase pigments 8 and 9 renders unlikely the possibility of the crystal form of the core TiO_2 having appreciable effect on the nature of the surface ion. The frequencies shown in Table 3 for the weak stearate ion bands found for the simple pigments (7c, 10b, 10x) corresponded to those obtained for a material purporting to be hydrated titanium stearate. The shoulder which occurred at 1590 cm^{-1} in the spectrum of stearic acid adsorbed on to "wet" pigment 5 (Fig. 10) indicated the possibility of reaction with the aluminium of the surface coating under this condition.

Throughout this examination the spectra were obtained, as previously described, from samples of the pigment which had been isolated from the pigment/adsorbate system and dried. This procedure introduced a possibility that the observed phenomena were a product of the isolation and drying stages and did not occur in the actual dispersion. To examine this possibility dispersions were prepared of the pigments in solutions of stearic acid in n-undecane, Nujol and acid-free linseed oil. After equilibration, the dispersions were carefully centrifuged to concentrate the pigment into a loose layer and the excess solution decanted. The pigment paste was examined spread between rock-salt plates.

In all cases the results were essentially identical to those obtained for the dried samples, justifying the continued use of the technique of isolation and drying before spectroscopic examination.

Adsorption of stearyl alcohol

Stearyl alcohol appeared to be the logical choice of material to represent the hydroxyl function as it retained the hydrocarbon chain of stearic acid. Large differences in behaviour between acid and alcohol were observed, both in the effect of water and in the shape of the isotherms (Figs. 11, 12 and 13). Water impeded the adsorption of the alcohol by all of the pigments, under " wet " conditions no adsorption being observed. In all cases going from the " dried " to the " normal " conditions resulted in a shift of the isotherm towards a higher concentration region. Except for the " dried " complex rutile pigment 5 all of the systems gave isotherms of the S form. This type generally occurs when the grouping on the adsorbate active in the adsorption process is localised and a moderate degree of intermolecular attraction exists resulting in vertical orientation of the adsorbate molecules on the substrate. The adsorbate must also meet competition for the substrate sites either from the solvent or from another substance present in the system[6].

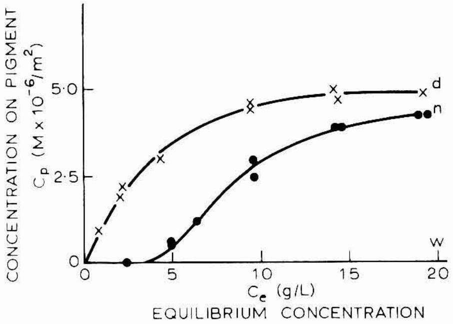


Fig. 11. Adsorption of stearyl alcohol by pigment 5

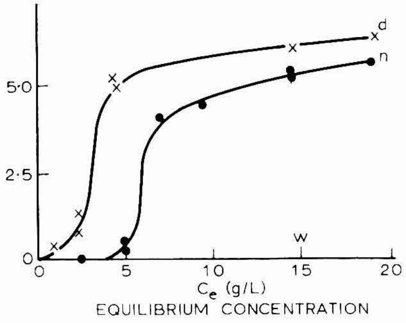


Fig. 12. Adsorption of stearyl alcohol by pigment 7c

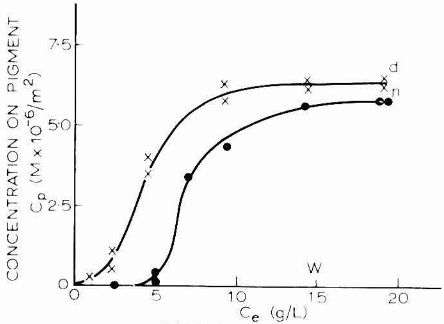


Fig. 13. Adsorption of stearyl alcohol by pigment 10b

In the present system it was probable that the solvent was the competing substance ; Doorgeest[7] obtained a similar S isotherm for the adsorption of stearyl alcohol from benzene on to titanium dioxide and a normal L type from n-heptane solution. However the changes brought about in the present system by water must be considered significant.

The spectrum of the adsorbed alcohol on all the pigments showed a broad band in the region of 3400 cm^{-1} consistent with a hydrogen bonding mechanism.

The pigment surface hydroxyl band in the 3700 cm^{-1} region could not be observed indicating that these surface hydroxyls formed at least part of the adsorption sites.

A major difference, as compared with stearic acid was in evidence for an interaction involving the hydrocarbon chain. On all of the pigments the $-CH_2-$ stretching frequency of the adsorbed alcohol was shifted to a value lower than that found for the alcohol either in solution or as a solid (Table 5).

Table 5

$-CH_2-$Stretching frequencies of stearyl alcohol

Mode	Solid	Solution	Adsorbed
Asymmetric	2926 cm^{-1}	2930 cm^{-1}	2917 cm^{-1}
Symmetric	2860	2859	2849

A frequency shift in this direction implied that lower energy was required for the chain stretching vibrations and was tentatively assigned to a close packed oriented system moving in unison.

(A similar frequency shift has been recorded for a polymeric hydrogen bonded structure.)

Adsorption from a mixed acid/alcohol solution

In a practical alkyd medium both acidic and hydroxylic groups would normally be present and competition between these for the available adsorption sites would be expected. As a first approximation to this condition the adsorption from mixed equimolecular solutions of the acid and alcohol was followed. The results for both the " dried " complex rutile and simple anatase materials were similar. In both cases the total adsorption was the same as when the acid was present alone. As the concentration increased the adsorption the alcohol passed through a maximum whereas that of the acid continued to rise as is shown for the rutile pigment in Fig. 14.

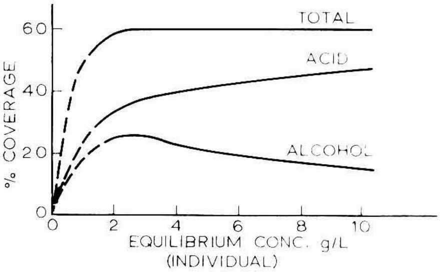


Fig. 14. Adsorption by pigment 5 from a solution containing stearic acid and stearyl alcohol

This preference for acid adsorption by both pigments at higher concentration was of interest considering the differing mechanisms of acid adsorption by the two pigment types.

Adsorption of an unsaturated acid

The combination of carboxyl group and chain unsaturation was represented by oleic acid. With this acid system care was necessary to prevent oxidation during the adsorption stage. It was found that no detectable oxidation occured if :

1. The acid was of a very high purity.
2. Light was excluded from the adsorption vessels.
3. The adsorption time was kept to the minimum required for equilibration.

In an early experiment using a so called 97 per cent pure oleic acid sample (actual purity nearer to 87 per cent) and without exclusion of light, rapid oxidation occurred. This was more severe with the simple anatase pigment than with the complex rutile.

In all instances appreciably less oleic acid was adsorbed by the pigments than was observed for stearic acid (Figs. 15, 16 and 17). No constant ratio could be found between the quantities of the two acids adsorbed under comparable conditions. Adsorption of the oleic acid by the complex rutile pigment 5 was not enhanced by the increase in water content on going from the " dried " to the " normal " condition while for the simple rutile pigment a decrease in the adsorption of oleic acid occurred with increasing water content of the pigment. Both pigments 5 and 10b showed an appreciable slope in the oleic acid adsorption isotherm indicating a possibility of multilayer formation.

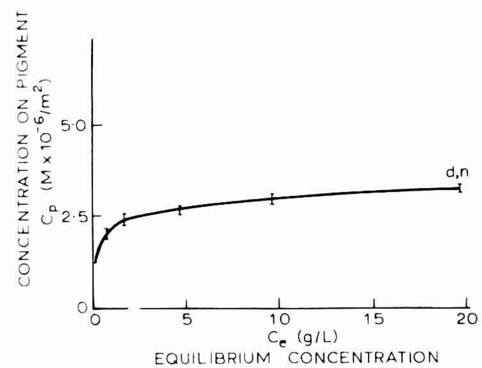


Fig. 15. Adsorption of oleic acid by pigment 5

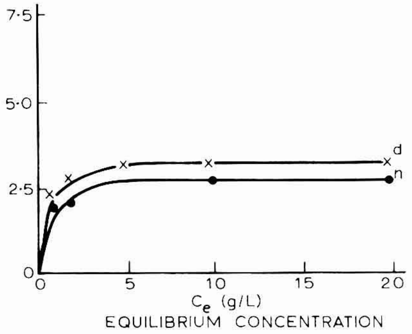


Fig. 16. Adsorption of oleic acid by pigment 7c

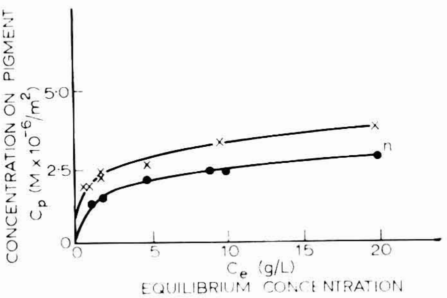
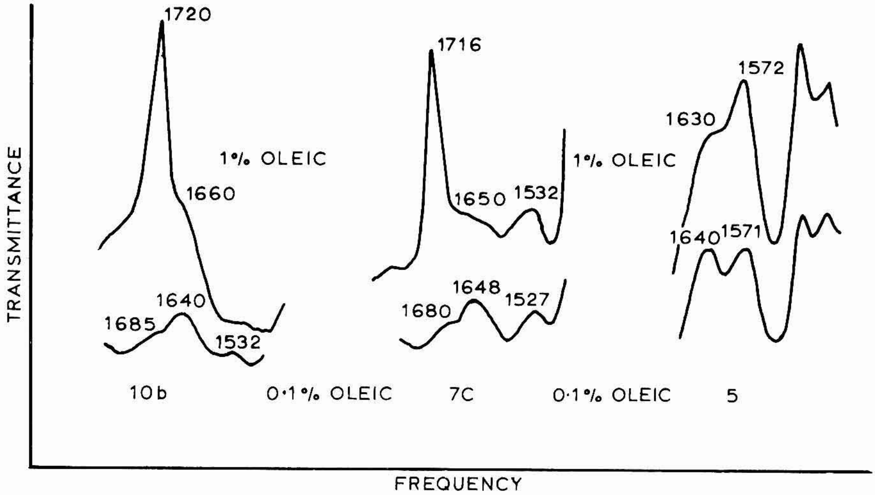


Fig. 17. Adsorption of oleic acid by pigment 10b

Similarities in the carboxyl region of the adsorbed oleic acid spectra (Fig. 18) to that of stearic acid were evident. The carboxylate ion band frequency observed on the complex rutile pigment 5 (1572 cm^{-1}) was identical with that found for oleic acid adsorbed onto zinc oxide. The two simple pigments, 7c and 10b, showed essentially acidic adsorption but with a trace of ionic material on the rutile pigment 7c as indicated by the band at 1532 cm^{-1}. From a low concentration solution ionic adsorption occurred preferentially on both simple pigments. A band was observed in the 1640 cm^{-1} region of the spectra of both stearic and oleic acids adsorbed onto the " normal " pigments. This was believed to be related to the presence of water but no definite assignment has been made.



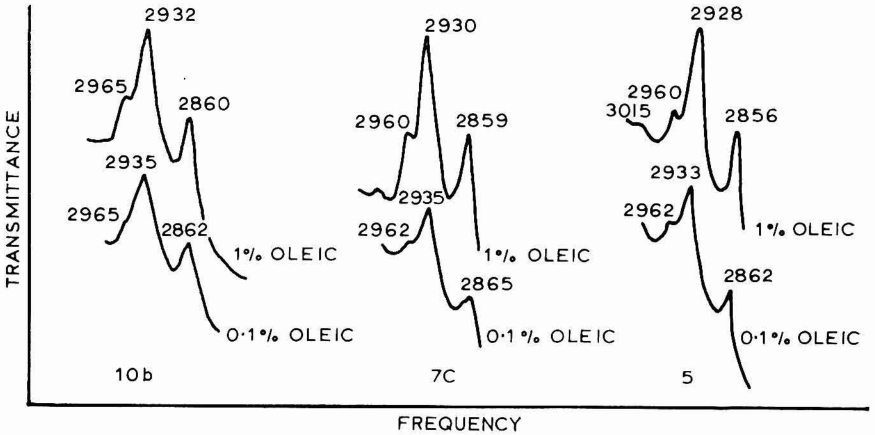


Fig. 18. (top) Spectra of adsorbed oleic acid in the 1550-1750 cm^{-1} region (carboxyl)

Fig. 19. (bottom) Spectra of adsorbed oleic acid in the 2850-3020 cm^{-1} region (chain and unsaturation)

The presence of appreciable acidic material was detected from the spectrum of pigment 5 after adsorption of oleic acid from a high concentration solution (20 g/l). The presence of this material, which could be readily removed by washing with n-hexane, provided further evidence for multi-layer formation.

The $-CH_2-$ and $-CH{=}CH-$ regions of the spectrum of adsorbed oleic acid were perturbed indicating that the hydrocarbon chain was involved in an interaction (Fig. 19). The $-CH_2-$ stretching frequencies were in all cases shifted to a higher frequency than the values of 2929 and 2857 cm^{-1} found both for liquid and dissolved oleic acid. This frequency shift was in the opposite sense to that found for adsorbed stearyl alcohol. No similar shift was found for adsorbed stearic acid. The band for $-CH{=}CH-$, normally found at 3007 cm^{-1} for oleic acid, could only be observed on pigments 5 and 7c after adsorption from higher concentration solutions. Even here the band was of low intensity and the frequency shifted to a higher value.

Adsorption of Esters

The last system so far examined, ester group, employed methyl stearate as the type compound. No evidence for adsorption of this material by the complex rutile, simple rutile or simple anatase pigment was observed. This result could be of great importance considering the preponderance of ester groupings present in a practical resin.

Discussion

The stearic acid adsorption and exchange experiments demonstrated that differences existed between the simple anatase and the complex rutile pigment in the mode of attachment of the acid. The high affinity of the complex rutile pigment for the acid, indicated by the shape of the adsorption isotherm, was confirmed by the appreciable adsorption by this pigment of acid from a solution in alcohol ; a condition under which no adsorption by the simple pigments was observed. The effect of water also indicated a difference in mechanism between the pigment types, enhancing the adsorption of stearic acid by the complex rutile pigments and considerably suppressing the process for the simple anatase materials. The exchange experiments gave results similar in form to those obtained by Shepard and Ryan[8] for the exchange of adsorbed acids from what they termed " reactive " (e.g. aluminium) and " non-reactive " (platinum) surfaces. The exchange from the " reactive " surface was of an exponential form as obtained in the present work for the complex rutile pigment and that for the " non-reactive " surface resembled the simple anatase material. These results pointed to a chemical adsorption process stimulated by the presence of water for the complex rutile and a physical process for the simple anatase.

This was in full agreement with the spectra obtained for the various samples of pigment/adsorbate complex. In the presence of water stearic acid was present on the complex rutile pigment almost entirely in the form of a carboxylate ion. When the water content was restricted, as in the dried sample, the spectrum was distorted in the carboxylate ion region but did not show a band in the region of 1700 cm^{-1} for the carboxyl group of acidic form. On the simple anatase pigments, the strong absorption band in the region of 1720 cm^{-1} indicated that the stearic acid was present essentially in an acidic form. It was possible to qualitatively relate the complexity of the pigment surface treatment and modification with the effect of water on the adsorption process and the proportion of ionic to acidic forms of the adsorbed acid (Table 6).

Table 6

Relationship between pigment complexity effect of water and ion/acid ratio

Pigment		Effect of water	ion/acid ratio
5	most complex	increased adsorption	almost all ion
8		small decrease	fairly equal
9	↓	larger decrease	more acid than ion
10b	simplest	very large decrease	almost all acid

The simple rutile pigment, 7c, fell out of this sequence in that, even though the adsorbed stearic acid was predominantly acidic in form, water had little apparent effect on the adsorption process. No straightforward explanation for this was found.

Zinc, when present in the pigment, dominated the adsorption process even in the presence of larger amounts of other surface additions, such as alumina, which would have been expected to be active. The only evidence found for the possible interaction of stearic acid with the aluminium of the complex rutile pigment was in the presence of a large amount of water when a shoulder appeared at 1590 cm^{-1} on the side of the main carboxylate ion band.

In the spectra of adsorbed stearic acid on the simple pigments very weak absorption bands were observed which corresponded in frequency to those obtained for a material purporting to be a hydrated titanium stearate.

The exchange experiments involving the phthalic acid half ester gave indication that the strength of the acid/pigment band could be related to the strength of the acid. Even though the fundamental pigment/acid interaction had been shown to be essentially as for stearic acid[4], that is determined by the pigment surface, there was evidence that it was more firmly held on the surface of both the complex rutile and simple anatase pigments than was stearic acid. To some extent this was supported by other work at the Research Station where it was found that a low molecular weight highly polar fraction of an alkyd was selectively and tenaciously adsorbed by titanium dioxide pigments.

A similar conclusion can be drawn from the results of a study into the gloss of alkyd/UF stoving finishes pigmented with titanium dioxide pigments reported by Alpert[8]. In this paper the gloss of the finish was related to the interaction of the resin with such pigment surface coating materials as hydrous alumina and it was concluded that ionic reaction products were retained on the pigment surface associated with adsorbed water and affected the degree of flocculation of the pigment and hence the gloss of the paint film.

Alpert also showed that if the pigment was pretreated with a weak acid temporary loss of gloss of the finish resulted, but if a strong acid, such as a phthalic acid half ester was used in the pretreatment the loss of gloss was permanent. This indicated that the resin acids could displace the weak acid but not the stronger one.

The carboxyl group pigment surface interactions of oleic acid were essentially similar to those of stearic acid. Zinc again dominated the adsorption of the acid by the complex rutile pigment. A major difference, though, was in the effect of water in the adsorption of acid by the complex rutile. For both acids an increase in the water content favoured the formation of the carboxylate ion but for stearic acid only was there also a large increase in the quantity of acid adsorbed. A possibility was that the water caused a swelling of the pigment surface treatment resulting in the formation of fissures into which stearic acid could penetrate. Oleic acid was prevented from entry either by the configuration of the hydrocarbon chain resulting from the unsaturation or by a secondary attraction between the acid and substrate due to the unsaturation tending to orient the acid molecules in a near parallel plane to the pigment surface. The infra-red spectra clearly demonstrated that both the methylene groups and the unsaturation were involved in some interaction for which no analogous behaviour was found for stearic acid. The band due to unsaturation, normally found at 3007 cm^{-1} for oleic acid could not be observed in the lower concentration experiment, and, where it appeared in the higher concentration experiments was weak and of higher frequency. This pointed towards an interaction between the unsaturation and the pigment surface but the evidence was not unequivocal.

The form of the stearic acid adsorption isotherms indicated only single layer formation, but for oleic acid the isotherms showed the possibility of a multilayer system. This was further supported by finding a significant quantity of non-ionically bound, easily removed, oleic acid on the complex rutile pigment recovered from higher concentration experiments. The frequency found for the carboxyl band of this acid was similar to that found for hydrogen bonded acid on the simple pigments. This indicated that the ionic process could only occur for the first layer of adsorbed acid.

The failure to observe an interaction between an ester group and the pigments was of great interest considering that this grouping predominates in a resin system. It is still necessary to determine whether any secondary effects exist which would influence the adsorption of other active groups in the presence of an ester.

The interaction between hydroxyl groups and the pigments was similar for all the pigment types and involved a hydrogen bonded linkage with the pigment surface. The process was sensitive to the presence of water in the pigment, no adsorption of alcohol being observed under the " wet " condition. The solvent, benzene, complicated the examination of the adsorption of the alcohol due to known interactions with hydroxylic materials. In the present system possibilities existed for solvent/solute and solvent/substrate interaction as well as the solute/substrate process, these further being complicated by the presence of water. The S isotherms probably resulted from this complex series of interaction.

The interaction involving the hydrocarbon chain of the alcohol, revealed by spectroscopy, was in itself interesting but was probably irrelevant when considering an alkyd resin as a simple long straight chain alcohol of this type would not be present. The residual hydroxyl groups in the resin would consist of odd unesterified groups on the polyhydric alcohol residues which formed part of a complex molecular structure.

The mixed acid/alcohol system indicated that the acid would normally dominate adsorption from a practical system. This effect was also demonstrated at the Research Station by Baxter[10] using a different solvent system in which an alcohol/solvent interaction of the type which can occur with benzene did not exist and therefore it can be concluded that a similar process may occur in the practical case.

So far only the adsorbate part of the adsorption complex has been considered. Whilst this was the major object of the exercise, some evidence was obtained of the nature of the surface sites of the pigment which participated in the adsorption process. The decrease in intensity of the far infra-red band in the 660-690 cm^{-1} region for both simple anatase and complex rutile pigments, on the adsorption of stearic acid indicated that a metal-oxygen band near to or in the pigment surface was influenced by the adsorption process.

Adsorption of both stearic acid and stearyl alcohol by the pigments resulted in the disappearance of the 3700 cm^{-1} band due to surface hydroxyl groups. In the case of the alcohol a band at 3400 cm^{-1} appeared which was consistent with a hydrogen bonding adsorption mechanism. These results indicated that the pigment surface hydroxyl groups formed at least a portion of the sites active in the process.

Conclusions

A picture is now emerging of the manner in which the active polar groupings of an alkyd resin may become attached to the various grades of titanium dioxide pigments. The primary point of interaction almost certainly involves a residual carboxylic acid group of the alkyd resin, the nature of the interaction being decided by the nature of the pigment surface.

On simple pigment types a hydrogen bonded mechanism was found to predominate, a process sensitive to the presence of water.

On the complex treated pigment types an ionic mechanism was operative increasing in extent with increasing complexity of the pigment treatment. This process was dominated by zinc, when present in the pigment, and was enhanced by the presence of water. It would be of considerable interest to ascertain if the increased ionic process brought about by the increased water content could be related to other changes in property of a paint such as reported by Alpert[9].

The presence of unsaturation in the adsorbate molecule did not affect the basic carboxyl group/substrate interaction but did influence the shape of the adsorption isotherms. The spectroscopic evidence indicated that the unsaturated hydrocarbon chain was entering into an interaction, probably with the substrate and resulting in an orientation of the molecule on the substrate.

Acknowledgments

The authors wish to thank their colleagues for assistance in these studies, particularly Mr. B. Kelman for carrying out many of the infra-red examinations, and also the Council and Director of the Research Association of British Paint, Colour and Varnish Manufacturers for permission to publish this paper.

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Discussion at London Section

A SPEAKER asked for information on the nature and purpose of the surface treatment of the pigments.

MR. SHERWOOD said that there was a lack of published information on the details of the surface treatment process. It was known that zinc was generally added to rutile pigments before calcination, to assist in the formation of the rutile crystal form and also to control chalking, that is a degradation of the medium and release of the pigment from a paint film on exposure. Alumina and silica were normally added after calcination, and it is believed that the pigment is added to a solution of aluminium sulphate and sodium silicate, with adjustment of pH to cause precipitation. Rechmann has shown that the ratio of alumina to silica is critical and affects ease of dispersion, oil adsorption and tinting strength.

MR. G. C. HURST asked what would be the effect of the commonly used techniques of dispersion on the delay in achieving equilibrium between the pigment and the adsorbed material.

MR. SHERWOOD said that the impact energy in a ball mill was large and might be expected to have some effect, but he had not yet done any work on the effect of shear on adsorption processes.

DR. J. A. KITCHENER said that in the flotation of oxide minerals by fatty acids such as oleic it was generally found that part of the adsorbed " collector " was extremely difficult to remove with solvents. Was this so in the systems studied ?

MR. SHERWOOD said that the ionically-bound material was strongly held on the surface, and only material adsorbed as the free acid could be easily removed with solvents.

SIR ERIC RIDEAL said that the alumina-silica treatments should affect the proton donation properties of the surface ; this was the case with oil cracking catalysts. Was the effect of the treatment also dependent on the Al/Si ratio ? The surface acidity of cracking catalysts could be titrated.

MR. SHERWOOD said that it had been shown that the alumina/silica ratio could affect the properties of the pigment. The treated pigment surfaces acted as bases, as was shown in the formation of the ionically bound acids. Alpert, Jacobsen and Mitton had shown that the treated surfaces could be titrated against an alkyd resin solution and the process followed electrometically.

MR. C. I. SNOW asked whether the pigment surfaces were uniformly coated ? Rutile titania was a highly anisotropic crystal, and some surfaces might be more easily coated than others.

MRS. S. M. RYBICKA agreed that this was a possibility.

DR. S. H. BELL said that on this question of the distribution of the coating agents on the individual particles of titanium dioxide, the balance of evidence, e.g. from electron microscopy, seemed strongly on the side of a fairly even coating over the surface.

Reverting to an earlier question about pigment/medium interactions in dispersion, it had to be remembered that at the outset of any paint-making process the medium had to be introduced into a mass of dry pigment. There was a wetting penetration and particle separation stage ; also full dispersion in the sense of forming a stable system of completely separated particles might not be achieved.

When talking about percentage coverage of particles by adsorbed molecules, Mr. Sherwood had been very careful to say that the calculations were based on a BET gas adsorption surface area, and dimensions for space occupied by, say, a fatty acid based on ideally oriented molecules. In practice, all the surface available to a gas might not be available to different types of molecules.

MR. SHERWOOD said that the BET surface areas used were obtained after drying the pigments under vacuum at 130°, and the available surface area of a pigment which had been exposed to water vapour could be appreciably different.

MR. K. W. GREEN asked how the " wet " pigments were prepared. Also, what were the units used to express the water content ?

MR. SHERWOOD said that the " wet " pigments were exposed as a thin layer in a dish over water at 25°C in a closed vessel. The units were mg water per g pigment.

DR. KITCHENER said that many oxides exhibited slow surface changes when exposed for long periods to water vapour. Was there any evidence for this with the " wet " titania ?

MR. SHERWOOD said that normally the pigments were left over water for 21 days. Longer periods had been examined, with no noticeable difference.

MRS. RYBICKA said that the experiments on the adsorption of stearic acid and the effects of water gave information which could be of value in the identification of different types of pigment.

MR. M. C. GRINT asked whether further work would be done using organically-treated titanium dioxide pigments.

MR. SHERWOOD said that this would depend to some extent on the published information available on the nature of the organic surface treatment. There was no point in examining a pigment carrying a completely unknown surface treatment, particularly when such treatments were liable to be changed.

MR. K. GOLDSBROUGH asked whether Mr. Sherwood would enlarge on the effect of the adsorption processes on the behaviour of the pigments in paint media. There had been a reference to an improvement in gloss with higher COO—/COOH adsorption.

MR. SHERWOOD said that he had referred to the observation that the highly polar low molecular weight portions of alkyds appeared to be associated with poor dispersion. It was thought that a bridging effect between adjacent pigment particles occurred. In column experiments an alkyd resin had been passed over titanium dioxide pigment, which had adsorbed mainly the low molecular polar function. This pigment recovered from the column had poor dispersion properties, while fresh pigment used with the alkyd which had been through the column (i.e. the unadsorbed fraction) showed good dispersion. Further work was in hand to extend the investigations into the more practical systems.

MR. SNOW said that if there was only a low molecular weight alkyd molecule on the surface of the pigment particles, it was possible for them to approach quite closely under London forces. It was the same phenomenon, but a different explanation.

MR. SHERWOOD agreed with this possibility.

Dr. Bell expressed surprise that no one in the discussion had commented (from the viewpoints of their various technical paint interests) on the effects of water on pigment behaviour dealt with by Mr. Sherwood. Water had worried the paint industry for years, whether in materials painted or in paint itself—meaning water in non-aqueous paints. It now seemed clear that moisture in the system could increase or decrease the adsorption of various organic substances from media, according to the type of pigment surface and the nature of the substance adsorbed. It now looked as though it might in due course be possible to deal with the effects of water in a quantitative way. It might prove that subtle variations in moisture content provided the clue to variations in products from apparently similar paint-making operations. Control at the right level for each type of formulation might become a significant feature of paint making.

Mr. R. C. Tarring expressed the appreciation of the Colloid and Surface Chemistry Group of the SCI at this opportunity for collaboration with the London Section, and thanked the authors for presenting a very interesting lecture.

Corrigenda

"*An Investigation into the effects of painting over millscale*," June 1966.

p. 466. Under Test Procedure, first line should read "Steel specimens 12 in × 10 in × $\frac{3}{8}$ in"

p. 469. Figures 2 and 3 should be transposed.

Guest Editorial

8th FATIPEC Congress Scheveningen 1966

The Fatipec Congress is held biennially in the years between OCCA Conferences. The 8th Congress held at Scheveningen, the seaside suburb of the Hague, was attended by over 1,100, of whom about 800 were participating members. At least 20 countries were represented, easily the largest contingent being from Western Germany ; there were nearly 100 British, including wives, about 75 per cent being from materials suppliers, 10 per cent from research associations and government laboratories and 10 per cent from paint manufacturers. The Hague, although the administrative capital of Holland, has been described as the largest village in Europe, a tribute to its easy, self-contained society and its comparative freedom from the pressure of industrial life.

Holland is physically one of our nearest neighbours ; our histories have touched at many points, and our temperaments are so similar that friendships develop quickly, but many of us from the United Kingdom know much less than we should about the Dutch and only gradually on this visit did we become aware of the greatness of their history and their achievements. Some hint of this was given in an absorbing opening lecture on Dutch life, art and philosophy by Dr. Sjollema. History surrounded us at the municipal reception, an informal social evening in the Ridderzaal, the royal throne room in the Binnenhof. Engineering genius became apparent when we visited the main sluices (now nearing completion) of the Deltawerk project, an enormous effort in flood control and desalinisation and, incidentally, an exacting corrosion test for paint under both fresh water and half-tide salt water conditions. The work we saw is only part of a countrywide programme of sea control, land reclamation, port extension and industrial development. The high level of the arts in Holland became evident in a special concert given by the Hague's own orchestra, the Residentie-Orkest, which was of high international standard and was received with rapt attention and enthusiastic applause. Finally, as evidence that, like some other people, one Dutchman could be artful as well as an artist, we were given an enthralling account by Dr. de Wild, one of the original investigators, of the case of the van Meegeren forgeries of "Vermeers" and "de Hoochs."

However this was not purely a pleasure trip ; technical sessions ran from 9 a.m. to 6 p.m. on most days, often with three sections meeting concurrently, a tempo more satisfactory to the conscience than OCCA's much sparser schedule but, in the event, too much for the technical digestion. In conversation there was much favourable comment on OCCA conferences, particularly with regard to the quality of papers and the discussions.

The theme of the Congress, " Scientific Research on the Protection of Substrates by Paint," was wide enough to cover most aspects of paint technology and had attracted a large number of papers ; nine were presented in plenary sessions and 62 in the three sections, which were concerned with composition and film formation, interaction with the substrate, and ageing and deterioration. The scope ranged from an academic and excellently presented Belgian plenary paper on the effect on properties of controlled variation in tacticity of methyl methacrylate polymers, through a number of papers on electrocoating, preparation of metal substrates, paints for specific conditions (including sub-Alpine pipelines), a useful group of papers on adhesion, and some first-class studies of electron microscopy in weathering and pigment research.

Two papers came from the USSR, two from Israel and one from South Africa, besides others from USA, Hungary, Poland and most of the countries of Western Europe. Such a wide geographical distribution not only introduced ideas (such as those in a Russian paper on the effect of plasticiser concentration on film properties)

ESTER SOLVENTS

C.S.R. CHEMICALS

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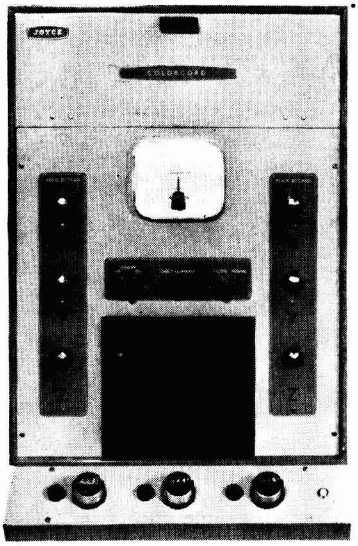

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which were new to our own technology but also, in the section on weathering, highlighted some interesting contrasts in paint requirements and performance in different countries. OCCA sponsored a plenary paper, in which Mr. F. G. Dunkley reviewed the efforts now being made to improve the quality of protective painting of iron and steel so as to achieve something much nearer the inherently possible performance of paint systems. There were eight other UK papers, including two from the Paint Research Station, on the ionic properties of paint media and adhesion to metal substrates, two from BISRA, on the painting of iron and steel and the painting of sprayed zinc and aluminium, and others dealing with the protection of cadmium, blistering of zinc rich primers under marine immersion, organic chromates in anticorrosive paints, and thick film chlorinated rubber paints.

Even with three sections running concurrently and almost four full days of meetings, the time available for each paper was very limited. Papers were grouped according to subject so that the value and interest of each individual paper was enhanced, but there was little opportunity for the keen discussion which one felt would have developed at an OCCA Conference ; indeed the few questions that time allowed rarely probed beyond the immediate scope of the written lectures.

Many papers were very good, and some were very clearly presented ; a few were mere " pot-boilers " and a few others were almost lost in presentation, either through language difficulties (even with the aid of simultaneous translation into three languages which was generally very well done), or careless preparation. It is a paradox that, whilst at a conference the presentation of papers, or many of them, may add little to the understanding of the book of preprints, the latter can only be brought into being in association with the conference. Certainly it would be difficult for many to justify attendance at the FATIPEC Congress for the lectures alone, but the opportunity to talk with most of the leading paint technologists of Europe away from the factory, the laboratory and the telephone is of inestimable value. Clearly the UK material suppliers realise this ; the paint manufacturers, judging from their small representation at the Hague, are rather more insular.

T. R. BULLETT

Reviews

STANDARD METHODS FOR THE ANALYSIS OF OILS, FATS AND SOAPS

By the OILS AND FATS DIVISION OF THE INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY, 5th Edition, incorporating the First Supplement (1966), Butterworths, London, approximately 130 sheets. Price 50s.

The present issue consists of a loose-leaf folder enclosing the fifth edition (dated 1964) comprising about 40 standard tests, together with a first supplement of methods added up to the end of 1965. They are for the determination of monoglycerides, small amounts of water, benzidene values and phosphorus contents of oils and fats, and of glycerol in soaps.

This is a book to be used rather than read, but the purchaser is advised to give some attention to the introductory historical note by J. Vizern, which underlines the difficulties encountered by a distinguished body of men over the years, and may suppress his impatience at so much remaining to be done. One detects almost a note of triumph in the observation that, after five years of heated argument, a spectrometric technique was adopted in preference to subjective methods for colour determination, and wonders how long it will be before topics like thiocyanogen and polybromide values, which have been obsolete for nearly a quarter of a century, are discarded in favour of more modern procedures.

The slowness of achieving international agreement must inevitably cause a book like this to have an old-fashioned look, but it could have been reduced—and its value greatly enhanced—if the results obtained in the numerous co-operative investigations undertaken by the committee had been treated statistically and some indication given of the precision of each test. At present, we have only bald descriptions without even a hint of the number of significant figures to which the results should be expressed. This could lead to as much trouble in practice as the interpretation of languages, which has received attention. The text is presented both in French and in English, but it is made clear that in cases of dispute the French version is official. This curious pronouncement suggests that it is beyond the wit of man to give an accurate description of an exact analytical procedure to a trained chemist in more than one language at a time. If so, the use of an official language must present a considerable disadvantage to anyone foreign to it. In actual fact, a random comparison of the texts did not reveal any significant points of difference which could conceivably lead to trouble. It would, however, be more convenient if the French and English versions were face-to-face instead of back-to-back.

We are indebted to the Analytical Sub-Committee of the Oils and Fats Division of IUPAC for their painstaking work over the years and for the publication of their findings. Internationally agreed standard methods are of inestimable value and it is to be hoped that, one day, these will reach the stage where universal adoption becomes possible.

M. R. MILLS.

DICTIONARY OF ORGANIC COMPOUNDS : SECOND SUPPLEMENT 1966

Editor R. STEVENS. Eyre & Spottiswoode (Publishers) Ltd., London, 1966. Pp. 221. Price £10.

Heilbron's dictionary of organic compounds first appeared in 1934 and soon became established as the foremost reference book of its kind. It went through three editions before Sir Ian died in 1959 while the fourth edition was being revised. The fourth edition, in five volumes together with the first supplement, was published in 1965.

The intention is to publish annual supplements, every fifth supplement being a cumulative edition containing both new material collected during that year and that brought forward from the previous four supplements. This Second Supplement incorporates new material published in and before 1965, together with corrections to the earlier volumes.

Compounds are arranged in alphabetical order. Certain minor changes in nomenclature and order have been introduced to bring the work into line with current practice of the Chemical Society and the International Union of Pure and Allied Chemistry. Entries which are supplementary to the main work are indicated by an asterisk. Errata to the main work and first supplement are included in the appropriate alphabetical place giving the page number of the original entry.

At £10 (nearly 1s. per page) this is expensive by any standards ; the complete work to date, five volumes and two supplements, costs £120. However, when one considers the amount of work involved in producing a work of this kind such a price can be more easily understood.

G. L. HOLBROW.

SURFACE CHEMISTRY

Proceedings of the Second Scandinavian Symposium on Surface Activity, Stockholm, 18-19 November 1964. Sponsored by The Royal Swedish Academy of Engineering Sciences. EDITORS : PER EKWALL, KJELL GROTH AND VERA RUNNSTROM-REIO. Published by Munksgaard, Copenhagen, 1965.

The use of surface active agents in aqueous solutions has been studied extensively and has resulted in a considerable literature. The present volume contains the papers read at Stockholm in 1964 and makes a further useful contribution to the knowledge of the practical applications of these materials.

The symposium was concerned with the uses of, in the main, water-soluble surfactants in a number of fields ranging from mineral flotation to problems in biology. In all, some 25 papers were read and these present a great amount of interesting information not only on the uses of surfactants but also on the properties of their solutions and the energy changes which take place at interfaces during the wetting process. Contact angle is discussed and an interesting method described for its measurement.

Four papers of direct interest to paint technologists are those dealing with the choice of surfactants for different purposes (mainly for emulsification using HLB values) ; the use of the cloud point to identify or characterise non-ionics of the polyethyleneglycol types ; the properties and uses of aluminium soaps (most of this is well known) and surface chemistry in lubrication. The latter paper discusses the use of surfactants in relation to adsorption on the metal surface and the prevention of corrosion. This paper contains the germs of ideas which could possibly have useful application in the paint field. Interesting topics in related fields are the uses of selective adsorption in mineral processing to concentrate low quality ores (a process of increasing importance) and the relative efficiencies of wet and dry grinding processes for solids.

Although a large part of the book deals with applications of surfactants outside the surface coatings field, a perusal of its contents is a rewarding experience for anyone interested in surface activity. The individual papers are referenced but the usefulness of the book would be increased by an index and possibly by classification of the topics. It is largely free from printing errors and would be a useful addition to the reference library.

W. M. MORGANS.

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.)*

A new petroleum naphtha solvent, specially formulated to meet the requirements of the paint industry, is announced by CARLESS, CAPEL & LEONARD LTD. This product is an addition to the range of petroleum naphtha solvents introduced by the company at the OCCA Exhibition in March this year.

Carless, Capel & Leonard Ltd. has also announced that it is stepping up production of its hexane paraffinic hydrocarbons to meet the increasing industrial demand for hydrocarbons distilling in the 58-72° C range.

The Tung Board, Blantyre, Malawi, has appointed the CHATFIELD APPLIED RESEARCH LABORATORIES LTD. to deal with technical enquiries from prospective users in Great Britain and Europe.

It has been announced that on 1 July 1966, THE AEROGRAPH-DEVILBISS COMPANY became The DeVilbiss Company Ltd. It claims to be the only company in the finishing field able to offer comprehensive manufacturing, engineering, sales and service, almost anywhere in the world, for complete finishing systems or individual items of finishing equipment.

Production of a new colourless polyester lacquer for the furniture industry has started at the paint and lacquer factory in Debica, POLAND, and the factory in Debica will produce 500 tons of this colourless polyester lacquer in 1966.

KESTNER EVAPORATOR AND ENGINEERING COMPANY LIMITED, of Greenhithe, Kent, has stated that it has developed a pneumatically operated reciprocating fluid mixer which provides a controlled degree of agitation whilst maintaining a consistently optimum flow pattern.

THE MASTERMIX ENGINEERING CO. LTD. has introduced a new bench model 2 h.p. Dissolver which it says is designed for the rapid dispersion, dissolving, emulsification, mixing, etc. of pigments, resins, paints and inks besides many other liquids and pastes.

Publications Received

Publications received by the Association include " Isobutyl vinyl ester, its properties and uses," published by ICI Limited, " World Oils and Fats Statistics 1962/1965 " prepared by the Economics and Statistics Department, Unilever Ltd., " Copolymer emulsion paints " published by Vinyl Products Limited, and " Glycerine and its derivatives " published by the United Kingdom Glycerine Producers Association.

Annual Report of the Council, 1965

ADOPTED AT THE FOURTH ANNUAL GENERAL MEETING OF THE INCORPORATED ASSOCIATION HELD AT THE PAINTER-STAINERS HALL, LITTLE TRINITY LANE, LONDON, E.C.4, ON 30 JUNE 1966

General

During the year under review there have been many successful innovations in the Association's activities.

The Seventeenth Technical Exhibition (22-26 March) was the first to be held at Alexandra Palace, London, N.22 and a report appears later under the Exhibition Committee's Report. Detailed reviews of the Exhibition appeared in the May and June issues of the *Journal.*

The Association's Biennial Technical Conference was held at Torquay (25-29 May) with headquarters at the Palace Hotel and this was the first occasion when Workshop Sessions were arranged. The main theme of the Conference Technical Sessions, when a total of nine papers was presented, was " Recent Progress in Coatings Technology " and there was a good attendance on each of the three mornings. All the available places at the Workshop Sessions on the Friday afternoon were filled ; the subjects covered by these sessions were " The Influence of Moisture on Surface Coatings," " Critical Design of Experiment " and " International Harmonisation of Testing Methods." As a result of the success of the Workshop Sessions, it is Council's intention to develop the practice of holding them at future conferences.

In addition to the usual social functions and activities this was the first occasion during the course of a Conference, when the President of the Association (Dr. J. E. Arnold) gave an Organ Recital, which was well attended and very much enjoyed. The Conference Diary appeared in the August issue of the *Journal.*

At the February meeting of Council a suggestion was received that Council might present to each Section a Chairman's badge of office. It was decided that the opinion of Section Committees should be sought and subsequently at the meeting of Council in June it was agreed that arrangements be made for the production of Chairmen's Badges to be presented to each Section. The design was based on the Presidential insignia and was carried out in gilt and blue. These badges were despatched to Section Chairmen, both in the United Kingdom and overseas in November and many appreciative comments have been received. At a later date a similar badge was despatched for the Chairman of the Australian Federal Committee ; this took the form of a green and gilt badge on green ribbon.

At the February meeting of Council also a proposal to issue a Joint Programme Card for all the United Kingdom Sections, Branches and Student Groups in place of the former individual Section and Branch programme cards was submitted. This suggestion was referred to the Section Committees concerned and as a result of further discussion in Council in June, it was decided that for the first time a Joint Programme Card should be published for the 1965/66 session. Following the despatch of these Cards, Council was glad to learn at its meeting in October that they had been well received and a number of favourable comments had been made thereon.

In January the London Section organised a one-day Symposium on " Paint versus Corrosion " which was well attended and the Transactions and Communications section of the April issue of the *Journal* was devoted to the papers presented at the Symposium, together with the discussions.

At the November meeting, Council was pleased to accord Section status to the Irish Branch of the Bristol Section. The Branch had operated for four years and their elevation to Section status became effective on 1 January 1966.

On 9 March members of the Council were pleased to entertain members of the Board of Directors of the Federation of Societies for Paint Technology who were on a visit to Europe. An informal meeting was held between the President, the Honorary Officers and the General Secretary of the Association and six members of the Federation's Board in the afternoon at which matters of mutual interest were discussed. The Dinner in the evening was attended by

12 members of the Federation's party and the Association was represented by the President, the Honorary Officers, the Chairmen of five United Kingdom Sections and Representatives on Council of the Australian and South African Sections, Dr. H. W. Keenan, a Past President and a Member of the Liaison Committee, Mr. G. N. Hill, who has the distinction of being the only Honorary Member of both organisations, and the General Secretary. A report of this Commemorative Dinner, together with photographs, appeared in the May issue of the *Journal.*

In addition to the reception at Wax Chandlers' Hall on 9 March, the Executive Secretary and members of the Board of Directors of the Federation of Societies for Paint Technology were also welcomed on a visit to the Technical Exhibition at Alexandra Palace and during a private visit to this country in July, Mr. R. W. Matlack (Executive Secretary) called again at the Association's offices.

As a result of the discussions which took place with Members of the Board, arrangements were made for a copy of the film entitled " Operation Success " prepared by the Federation of Societies for Paint Technology on careers in the paint industry to be loaned to the Association and this was shown to Members of Council at the conclusion of the meeting held in June. Because of the differences between the educational systems in the United States and the United Kingdom it was felt that the film was not wholly relevant to the situation in the United Kingdom, but with regard to the use of the film abroad, Mr. R. W. Matlack, Executive Secretary of the Federation of Societies for Paint Technology, indicated that he would be prepared to lend the film, if a copy were available, to the overseas Sections of the Association.

Dr. L. Valentine attended the 43rd Annual Meeting of the Federation of Societies for Paint Technology in Atlantic City, New Jersey, in October and conveyed greetings on behalf of the Council. A report of Dr. Valentine's visit to the United States will appear in the February 1966 issue of the *Journal.*

On 28 May at the Palace Hotel, Torquay, the Third Annual General Meeting of the Incorporated Association took place. Dr. S. H. Bell was elected President and the following Vice-Presidents were elected :—

Mr. A. Aitkenhead
Mr. J. C. Anner
Mr. F. Evans
Mr. A. S. Fraser
Mr. J. Smethurst
Dr. H. W. Talen
Mr. N. A. Bennett

The Honorary Officers were elected as follows :—

Honorary Secretary	Mr. I. C. R. Bews
Honorary Treasurer	Mr. F. Sowerbutts
Honorary Editor	Mr. D. S. Newton
Honorary Research and Development Officer	Mr. A. T. S. Rudram

The report of the Auditors on their scrutiny of the postal votes was received and it was announced that the following members had been elected to the Council for the years 1965-67 :—

Mr. T. E. Johnson
Mr. N. H. Seymour
Mr. H. C. Worsdall

Votes of thanks to the retiring President, retiring Council Members and the Honorary Officers of the Association were carried with acclamation.

The Seventh Australian Convention took place (17-20 June) in Terrigal, a popular seaside resort approximately 50 miles from Sydney. A record number of 227 delegates from five States and overseas attended and were welcomed by the Chairman of the New South Wales Section (Mr. M. J. Leahey), the Convention Secretary (Mr. D. M. D. Stewart) and Organisers of the Convention, which was held under the auspices of the Australian Federal Committee. The technical sessions were held each day (Friday and Saturday) and a total of nine papers was presented. The Australian Convention Diary appeared in the September issue of the *Journal* and some technical papers from the Convention will be published in the *Journal* and the *Australian O.C.C.A. Proceedings and News* in due course.

At the official opening of the Convention, the speaker (Mr. R. Simmat) was presented, by the newly-elected Chairman of the Australian Federal Committee (Mr. E. T. Backous), with the first copy of the second edition of the *Australian Raw Materials Index*, which was prepared by a special sub-committee from the South Australian Section. Members of Council have had the opportunity of seeing a copy of this new publication at the meeting in October and were unanimous in extending congratulations both to the Australian Federal Committee and to the Raw Materials Index Sub-Committee on the attractive production.

The paper " Phthalocyanine Pigments and their Application " by R. F. Hill, which was awarded first prize in the Scottish Section's Essay Competition, was published in the July issue of the *Journal.*

The Third Annual Convention of the two Sections in New Zealand took place at Wairakei (13-15 August). The Convention was organised by the Auckland Section and was again held at the Wairakei Hotel, which is a suitable venue for members travelling from both Auckland and Wellington. Seventy-two members and their wives from all parts of New Zealand, together with six members from Australia, attended the Convention and at the official opening they were welcomed by the Chairman of the Auckland Section (Mr. P. B. Hunt). Six papers were presented at the technical sessions and following the official closing of the Convention a joint committee meeting of members of both the Sections' Committees was held to discuss proposals for the next convention and other matters of mutual interest. A report of the New Zealand Convention appeared in the October issue of the *Journal.*

In September the Manchester Section organised a Conversazione and small exhibition for Junior Members jointly with the North East Liverpool Technical College under the title " Modern Instrumentation in the Surface Coating Industry."

On the evening of 13 October, Members of Council had the pleasure of receiving 12 Past Presidents (two of whom are Honorary Members), eight Past Honorary Officers and one Founder Member at the Reunion Dinner at Wax Chandlers' Hall. After Dinner the President gave his customary review of the Association's activities during the year and then made a presentation to Mr. D. S. Newton who, due to pressure of business commitments, had been compelled to resign in September as Honorary Editor of the Association, an appointment he had held since 1962. As his successor, the Council had been pleased to appoint, at its meeting earlier in the day, Mr. A. R. H. Tawn. The President then entertained the company with an interesting, illustrated talk entitled *A Chevron Argent Between Three Mortcours.* A full report and photographs appeared in the December issue of the *Journal.*

In the Report of the Council for 1964 reference was made to the completion of the History of the Association, up to the date of Incorporation, undertaken by Mr. G. Copping (an Honorary Member and Past Honorary Editor), with Dr. S. H. Bell (President) and Dr. H. A. Hampton (Past President and the South African Section's Representative on Council). As a result of the replies received to the notice included in the November 1964 issue of the *Journal*, Council decided at its meeting in February, on the recommendation of the Publications Committee and supported by the Finance Committee, that the History should be brought up-to-date for the Fiftieth Anniversary of the Association in May 1968 and at that time issued free to all members. A notice to this effect appeared in the April issue of the *Journal* and in view of this postponement, Mr. Copping wrote a short article entitled *The Story of O.C.C.A.*, which appeared in the August issue of the *Journal.*

The work on the Association's Research Project at Nottingham University continued, but it was with regret that Council learned at its meeting in November of the resignation of the Research Fellow (Dr. K. E. Lewis) with effect from 31 October. A report of the Technical Committee's meetings in Nottingham and information so far available on progress made in the Project are included later under the Report of that Committee.

In 1965, members of the Association were accorded honours by other organisations ; Mr. J. Wright, attached to the London Section, was awarded the City and Guilds of London Institute Insignia Award and Mr. P. Walker, Honorary Treasurer of the Thames Valley Branch of the London Section, was awarded second prize in the Roon Foundation Awards Competition. Mr. Walker presented his paper entitled *The Effect of Water on the Adhesion of Surface Coatings* at the Annual Meeting of the Federation of Societies for Paint Technology in Atlantic City (previously referred to in this Report).

During the year work on two co-operative research projects has commenced in two Sections, the subjects being " Colour Matching " and " The Reliability of Subjective Assessment " in the Hull and Manchester Sections respectively.

The South African Section, in co-operation with the Natal and Witwatersrand Technical Colleges, organised a nationally recognised course in Paint Technology at a level slightly higher than the City and Guilds Advanced Examination.

During the year the liaison established by the London Section with Dutch and Danish societies continued. In January Mr. M. R. Mills presented a paper in Copenhagen to the Danish Varnish and Paint Chemists' Association entitled " The Chromatography of Paint Oils ". In March Dr. Van Loon of Nederlandse Vereniging voor Verftechniek presented a paper to the London Section, the title of which was "The Interaction Between Paint or Varnish and the Substrate ". In October Mr. P. Fink-Jensen from the Danish Varnish and Paint Chemists' Association presented a paper to the London Section entitled " Rheological Properties of Paint and two new instruments for measuring them ".

During the year, members attached to overseas Sections have visited the Association's offices and the secretariat have been pleased to help them in many ways. Mr. A. R. Penfold (Honorary Member attached to the New South Wales Section) who arrived during the latter part of 1964, was a frequent visitor during the early part of the year, particularly for meetings of the Council's Steering Committee on Australian Affairs, and Council were pleased that both he and Mrs. Penfold were able to attend the Torquay Conference in May.

In March and April the Honorary Publications Secretary of the Victorian Section, Mr. G. Hartshorn, was in this country and had the opportunity of attending the Seventeenth Technical Exhibition and of having discussions with the Honorary Officers of the Association.

The Council was saddened during the year to hear of the deaths of Professor T. P. Hilditch, an Honorary Member of the Association since 1948 ; Mr. M. E. Dougherty, who was a Vice-President in 1934-36 and who during the period 1924-38 was continuously engaged as Honorary Auditor, Honorary Treasurer or on the Finance Committee ; Mr. K. L. Jones, who was Chairman of the South Australian Section from 1960-62 and who at the time of his sudden death was serving as the Section's Honorary Publications Secretary. Obituary notices appeared in the October, November and September issues of the *Journal* respectively. At the meeting in November, Council learned of the death of Mr. A. Pass who was an Elective Member of Council 1957-59 and who served on the London Section Committee and represented the Association on Technical Committees of the British Standards Institution for many years. Mr. Pass was also well known as a lecturer on several occasions, the last being at the Torquay Conference in May 1965. An obituary notice will appear in the January 1966 issue of the *Journal.*

The Council wishes to place on record its sincere appreciation of the outstanding service rendered to the Association by the General Secretary, Mr. R. H. Hamblin, and the members of his staff.

Membership of the Association

The number of members elected during the year was 422 compared with 394 in 1964. The net increase in total membership was 170 compared with 153 in 1964.

Council notes with pleasure the considerable increase in Junior Membership and thanks all members who have been instrumental in encouraging applications and in fostering Student Group activities.

Council is gratified to note that for the first time in the Annual Report the total number of members is shown in excess of 4,000.

The list below shows the total strength of the Association as at 31 December 1965 :—

Section	*Ordinary*	*Associate*	*Junior*	*Honorary*	*Total*
Bristol (incl. *Irish Branch) ..	128	30	4	—	162
Hull	71	13	5	—	89
London (incl. Southern and Thames Valley Branches)	864	162	31	3	1,060
Manchester	351	72	29	1	453
Midlands (incl. Trent Valley Branch) ..	191	40	15	2	248
Newcastle	156	25	60	—	241
Scottish (incl. Eastern Branch) ..	113	44	43	—	200
West Riding	61	17	5	—	83
General Overseas ..	319	30	1	1	351
Auckland	39	26	4	—	69
New South Wales ..	256	139	6	1	402
Queensland	32	33	2	—	67
South African ..	162	48	5	—	215
South Australian ..	48	25	2	—	75
Victorian	206	93	22	—	321
Wellington	48	15	—	—	63
West Australian ..	23	23	4	—	50
Total 1965	3,068	835	238	8	4,149
Total 1964	2,967	812	191	9	3,979
Net increase/decrease during 1965	+101	+23	+47	−1	+170

*Since 1 January 1966 Irish Section.

The Council

During the calendar year the Council has met five times, the average attendance being 19. All meetings were held in London.

Committees of Council

The Committees of Council met as set forth below :—

Exhibition Committee	2
Finance Committee	3
Light Fastness Committee	1
Publications Committee	2

Finance Committee

Chairman—The Honorary Treasurer, Mr. F. Sowerbutts

The market value of all investments, including the Ethel Behrens Fund, at the end of the year was £72 lower than the purchase price compared with £1,274 lower than the purchase price at the end of 1964.

As stated in the Annual Report for 1964, the Finance Committee was then much disturbed by the deficit on that year's working and when it became apparent that, despite an increase in income of £3,400, a much larger deficit would be incurred in 1965, the Finance Committee held a special meeting in the Autumn to draw up estimates for the long-term financial requirements of the Association and to make recommendations to Council. The main recommendation was that a higher proportion of the Association's income should be derived from membership subscriptions and this was considered for the first time at the November Council meeting.

Light Fastness Committee

Chairman—Mr. G. A. Campbell

The Committee met in May and had a full discussion on the present position of instrumentation in the measurement of colour differences, and its possible application to the assessment of light fastness. It was decided that with BS.1006 in common use now in the pigment using industries, the Committee should devote its full efforts to the application of instrumental methods, regarding BS.1006 as an interim method being the best hitherto recommended. The Committee agreed, therefore, to invite the interest of those members of the Oil and Colour Chemists' Association whose daily work included experience of colour measurement apparatus and the assessment of colour differences, and a letter has been agreed for publication in the February 1966 issue of the *Journal.*

Publications Committee

Chairman—The Honorary Editor, Mr. A. R. H. Tawn

The Committee met twice during 1965 under the Chairmanship of Mr. D. S. Newton who felt obliged to resign in September owing to increasing business commitments. His successor is deeply conscious that the satisfactory record for the year is a tribute to Mr. Newton's endeavours rather than his own.

The *Journal* for 1965 showed a modest increase in size over the previous year, containing a total of 1,172 pages of text, 773 (66%) of which were devoted to 44 technical papers. The corresponding figures for 1964 were 1,042, 621 (60%) and 45. The Committee welcomes this increase in technical content which, it considers, must be fostered if the *Journal* is to maintain its place in the technical literature of the world and to provide the service which members and subscribers alike have rightly come to expect.

Nevertheless, after making due allowance for a number of lengthy Conference papers, the indications are that many authors are still reluctant to publish the results of major investigations in serial form or to submit research notes and short communications on restricted topics. The protracted accumulation of material for comprehensive reports necessarily deprives both authors and readers of the advantages of early publication ; the *Journal* loses an element of vitality and the authors risk loss of priority with scant compensation beyond the satisfaction of producing a *magnum opus* once in a while.

Attention may again be drawn to the Correspondence columns which afford opportunities for the presentation of brief technical communications as well as for comment and controversy. In 1965, ten letters to the Editor occupied some 15 pages. This represents a small increase on the 1964 figures but the Honorary Editor feels that much more use could be made of this feature.

Survey of published papers :

	1963	1964	1965
(*a*) *Sections*			
Australia and New Zealand	2	3	4
Bristol	1	—	2
Hull	—	1	—
London	3	5	8
Manchester	3	10	2
Midlands	1	1	—
Newcastle	1	1	—
Scottish	1	—	1
West Riding	—	—	—
South Africa	—	—	—
(*b*) *Direct*			
U.K.	12	14	12
Overseas		10	6
(*c*) *Conference*	9	—	9
(*d*) *Memorial Lecture*	1	—	—
	34	45	44

The publication of the Paint Technology Manuals has continued ; Volumes I-V are now available and Volume VI is expected to appear in time for the 1966 Exhibition. *An Introduction to Paint Technology* is to be reprinted with the addition of a chapter on recent developments and work is in hand on the preparation of the Decennial Index to the *Journal* covering the years 1956-65.

Both the present Honorary Editor and his predecessor wish to record their sincere appreciation of the help and support of the Publications Committee, referees, reviewers and the Staff at Wax Chandlers' Hall which has been so necessarily sought and so freely given during the year.

Technical Committee

Chairman—The Honorary Research and Development Officer, Mr. A. T. S. Rudram

Dr. K. E. Lewis, the OCCA Research Fellow, resigned at the end of October 1965. The Technical Committee met at Nottingham University on 19 October to receive a report from Dr. Lewis and to discuss future research activities with Dr. G. D. Parfitt. A short report of the work undertaken by Dr. Lewis, under the guidance of Dr. Parfitt, has been accepted by the Honorary Editor for publication in the *Journal*, and a detailed paper on one aspect of the research work is to be submitted for consideration for publication at a later date.

The Technical Committee considered that there had been good progress, with a significant contribution to knowledge about rutile surfaces and a new concept for the stability of dispersions in non-aqueous, non-polar solvents.

Technical Education Committee

Chairman—Dr. H. W. Keenan

The hope was expressed in the Annual Report for 1964 that further Paint Technology Manuals would be published in 1965 and the Committee is pleased to report that considerable progress has been made.

Early in the year the Association's publishers (Chapman and Hall Limited) expressed concern regarding the size of Volume IV (Application and Testing of Surface Coatings) and, following negotiations, arrangements were made for this particular Volume to be divided into two parts, e.g. Volume IV *The Application of Surface Coatings* and Volume V *The Testing of Paints.* Volume IV became available in August and Volume V in November and it was possible to maintain the price of these Volumes at 35s. each.

By the end of the year the printing of Volume VI *Pigments, Dyestuffs and Lakes*, had been almost completed and it is expected that this Volume will be published in time for the 1966 Exhibition.

The Committee feels that the publication of these six Volumes will lead to increased sales and, in the meantime, every endeavour is being made to complete the work on Volume VII *Works Practice* which it is hoped to publish before the end of 1966.

Exhibition Committee

Chairman—The Honorary Treasurer, Mr. F. Sowerbutts

As already stated, reviews of the Exhibition appeared in the May and June issues of the *Journal*, collated by Mr. R. A. Brett and Dr. V. T. Crowl (Honorary Publications Secretary of the London Section) from reports by 39 members, but the Committee wishes to emphasise that this was the first Exhibition to be held at Alexandra Palace and the first occasion when the Exhibition had been open on five days. Despite fears expressed to the contrary, the attendance at the new venue was in no way impaired and many exhibitors confirmed from the enquiries received that there were many more visitors than at any previous exhibition. The attendance was estimated at 11,000 and representatives from 27 countries are known to have visited the Exhibition. Visitors were gratified to find that the journey to Alexandra Palace from central London was very much easier than expected. Those travelling by Underground train to Wood Green Station were conveyed quickly by the free bus shuttle service operated by the Association, and those travelling by car found there was ample free car parking space available.

For the first time since 1960 the Exhibition was held under one roof, there was a record number of exhibitors (107 stands) and a record stand floor area of over 20,000 square feet. More space was available for seating areas, which was greatly appreciated by exhibitors and visitors alike.

The Guest of Honour at the Exhibition Luncheon held at the Savoy Hotel, London, W.C.2, on Monday 22 March was the Rt. Hon. Lord Hill of Luton, P.C., Chairman of the Independent Television Authority and Chairman of Laporte Industries Limited. In the presence of 350 members, exhibitors and guests, Lord Hill replied to the Address of Welcome by the President of the Association, Dr. J. E. Arnold who, at the express wish of Lord Hill performed the opening ceremony by cutting a tape in front of the Association's Information Bureau at Alexandra Palace. Later in the afternoon Lord Hill visited the Exhibition and made a tour of the stands accompanied by the President, the Chairman of the Exhibition Committee and the General Secretary.

For the seventh year in succession parties of sixth form science students were invited to the Exhibition and 420 students from 20 schools attended over three days, during which short talks were given prior to the students seeing the Exhibition, the speakers on this occasion being Dr. S. H. Bell, Mr. G. L. Holbrow and Mr. A. T. S. Rudram. The theme of the Technical Education Stand was *Chemistry and Physics in the Study of Pigment Behaviour* and by way of illustration a series of 12 electron microscope photographs of pigments and extenders was supplied by the Paint Research Station. The stand was again staffed by representatives from Technical Colleges and the paint and printing ink industries.

Liaison Committee

Chairman—The President

During the year the most important activities in the field of liaison have been the visit by members of the Board of Directors of the Federation of Societies for Paint Technology in March and visits by Dr. W. J. Nijveld (President of the Federation d'Associations de Techniciens des Industries des Peintures, Vernis, Emaux et Encres d'Imprimerie de l'Europe Continentale) and Mr. H. Meyer (President of the Federation of Scandinavian Paint and Varnish Technicians) to the Conference at Torquay. Mr. W. Hoback also represented the Federation of Societies for Paint Technology at the Torquay Conference.

On both these occasions the Honorary Officers and General Secretary took the opportunity of discussing matters of common interest with the representatives from the other organisations.

The Association was represented on other organisations as follows :

TECHNICAL TRAINING BOARD FOR THE PRINTING INK AND ROLLER MAKING INDUSTRY : Mr. K. Pond, Dr. G. L. Fuchs and Mr. R. R. Coupe.

PAINT APPRENTICESHIP COUNCIL : Dr. H. W. Keenan and Mr. G. Copping.

PAINT TRADE BENEVOLENT FUND : Mr. G. Copping and Mr. D. E. Roe.

THE PARLIAMENTARY AND SCIENTIFIC COMMITTEE : The President and the Honorary Secretary.

THE BRITISH NATIONAL COMMITTEE FOR CHEMISTRY : The President.

CITY AND GUILDS ADVISORY COMMITTEE NO. 9—TECHNOLOGY OF PIGMENTS, PAINTS, VARNISHES AND LACQUERS : Dr. J. G. Gillan.

EAST HAM TECHNICAL COLLEGE ADVISORY COMMITTEE : Dr. H. G. Rains.

ASSOCIATION OF EXHIBITION ORGANISERS : The General Secretary.

PROGRAMME LIAISON COMMITTEE : The Honorary Secretary and the General Secretary.

THE PAINTMAKERS ASSOCIATION TECHNICAL EDUCATION COMMITTEE : Dr. J. E. Arnold or the Honorary Secretary.

THE SOCIETY OF DYERS AND COLOURISTS TERMS AND DEFINITIONS COMMITTEE : Dr. F. M. Smith and Mr. H. Cook.

JOINT POLYMER CONFERENCE COMMITTEE : The Hon. Research and Development Officer and the General Secretary.

BRITISH STANDARDS INSTITUTION :

Committee		
PVC	Pigments, Paints and Varnishes Industry Committee	Dr. J. B. Harrison
PVC/1	Pigments	Mr. A. S. Lewis
PVC/1/9	Black Pigments	Mr. J. S. Marsh
PVC/1/11	Extenders	Mr. S. A. Ray
PVC/1/18	Zinc Dust Pigments	Mr. D. S. Newton
PVC/3	Oils, Varnishes, Putty, etc.	Dr. H. Jasperson
PVC/4 & PVC/4/1	Lac	Mr. A. J. Gibson
PVC/6	Cement Paints	Mr. W. O. Nutt
PVC/8	Plastic Wood	Mr. V. P. Gellay
PVC/10	Test Methods for Paints	Mr. T. E. Johnson until June. Mr. A. D. C. Hamilton subsequently appointed as Association's representative.
PVC/15	Water Paints and Distempers	Mr. T. W. Wilkinson
PVC/16	Ready Mixed Paints	Mr. G. A. Newell
PVC/19	Bituminous Paint	Dr. H. B. Footner
PVC/20	Calcium Plumbate Priming Paints	Mr. A. G. Walker
LGE/9	Artificial Daylight for Colour Matching	Miss D. L. Tilleard
C/17	Viscosity	Miss D. L. Tilleard
C/17/2	Revision of BS.188 (Drafting)	Miss D. L. Tilleard
CHE/43	Test Sieves	Mr. A. Pass until his death in November. Mr. M. J. F. Meason subsequently appointed as Association's representative.
CHE/50	Test Methods for Powder Properties	Dr. J. G. Rigg
CIC/4	Solvents and Allied Products	Dr. L. A. O'Neill
OSC/6	Glycerine	Mr. M. R. Mills
OSC/7	Sampling Oils and Fats	Mr. M. R. Mills
OSC/12	Vegetable Oils	Mr. W. V. Lee
OSC/24	Analysis of Oils, Fats and Soaps	Mr. W. V. Lee
ELE/16/6	Varnishes	Mr. L. Massey
ACE/44	Aircraft Finishes	Mr. J. B. G. Lewin
BLCP/18	Code of Practice : Painting	Mr. P. J. Gay
BLCP/18/1	Code of Practice : Painting on Wood	Mr. P. J. Gay
M/36	Artists' Materials	Mr. J. A. L. Hawkey
RDE/25	Road Marking Compounds	Mr. T. R. Bullett
	Floor Seals	Mr. D. G. McKay

Reports of these representatives may be seen by members at the Association's offices.

The Association was also represented on overseas organisations as follows :

ADVISORY COMMITTEE OF NATAL TECHNICAL COLLEGE : Mr. P. A. Draper and Mr. K. R. McDonald.

AUCKLAND BRANCH OF THE AUSTRALASIAN CORROSION ASSOCIATION : Mr. P. B. Hunt.

PAINT AND VARNISH SECTIONAL COMMITTEE OF STANDARDS ASSOCIATION OF AUSTRALIA : Mr. M. J. Leahey.

POLYMER SCIENCE ADVISORY PANEL OF UNIVERSITY OF NEW SOUTH WALES : Mr. J. H. Foxton.

SOUTH AFRICAN BUREAU OF STANDARDS COMMITTEE : CODE OF PAINTING : Mr. K. M. Engelbert and Mr. G. Myers.

WEST AUSTRALIAN PAINT MANUFACTURERS' ASSOCIATION : Mr. D. Cole.

1 January 1966.

Oil and Colour Chemists' Association

(Incorporated 1 January 1963 as a company limited by guarantee and not having a share capital)

BALANCE SHEET as at 31 December 1965

1964 £				£	£
		ACCUMULATED FUND—			
		Balance at 31 December 1964		33,727	
		Add Overestimate on Deficit of A.F.C. 1964 Accounts		505	
		Capital Dividend		10	
		Sale of Fractional Share ..		1	
			£	34,243	
		Deduct A.F.C. Audit Fees 1964	160		
		Accounting Charges 1964	94		
		Deficit for the year	2,415	2,669	
33,727					31,574
2,442		ETHEL BEHRENS FUND—			2,442
		CURRENT LIABILITIES—			
	1,296	Creditors and Accrued Liabilities		4,678	
	17,127	Receipts in Advance ..		20,988	
	1,560	Provision for Research Project		1,620	
	517	Provision for Paint Technology Manuals ..		184	
	—	Provision for History of the Association ..		200	
	252	Provision for United Kingdom Income Tax		344	
20,752					28,014
£56,921					£62,030

Note :
Exchange :
Currency assets and liabilities have been converted at the rates ruling at 31 December 1965

S. H. BELL
President
F. SOWERBUTTS
Hon. Treasurer

1964 £	£			£	£
		FIXED ASSETS			
	4,924	Furniture, Fittings and Office Machines at Cost		4,998	
	3,004	*Less* Accumulated Depreciation		3,427	
1,920					1,571
		INVESTMENTS—			
	17,908	British and Local Authority Securities at Cost (Market Value £17,748)		17,908	
	9,860	Other Investments (Market Value £10,208)		9,860	
27,768					27,768
		ETHEL BEHRENS FUND—			
2,442		Local Authority Security at Cost (Market Value £2,182)			2,442
		CURRENT ASSETS—			
	396	STOCK OF UNSOLD PUBLICATIONS—valued at Cost		240	
	1,353	PAPER—Stock in Hand at Cost		1,798	
	5,064	DEBTORS AND PAYMENTS IN ADVANCE		5,665	
	519	FUNDS HELD BY AUSTRALIAN FEDERAL COMMITTEE		1,358	
		CASH AND BANK BALANCES—			
		United Kingdom	£17,860		
		Overseas Sections :			
		New South Wales	663		
		Queensland	160		
		South Australian	143		
		Victorian	536		
		West Australian	100		
		South African	547		
		Auckland	162		
		Wellington	149		
	16,659			20,320	
23,991					29,381
		ADDITIONAL FUNDS HELD BY OVERSEAS SECTIONS—			
	392	Victorian	427		
	384	New South Wales	409		
	—	Auckland	32		
	24	Queensland	—		
800					868
£56,921					£62,030

REPORT OF THE AUDITORS TO THE MEMBERS

The above balance sheet and annexed income and expenditure account are in agreement with the books which, in our opinion, have been properly kept. Annual accounts submitted by the United Kingdom and Overseas Sections have been incorporated in these accounts but the assets, liabilities and expenditure included therein have not been verified by us. Subject to this remark we obtained the information and explanations we required and in our opinion the accounts comply with the Companies Act, 1948, and give a true and fair view of the state of affairs and the results of the Association.

London, 5 May 1966.

COOPER BROTHERS & CO
Chartered Accountants.

INCOME AND EXPENDITURE ACCOUNT FOR THE YEAR ENDED 31 DECEMBER 1965

1964 £		£	£
	Journal Expenses—		
	Printing and Publication	17,585	
	Postage	1,887	
16,725			19,472
	Publication Expenses—		
	Printing	916	
	Reprints	1,182	
	Editorial	6	
1,187			2,104
	Section Expenditure		
	Bristol	161	
	(Inc. Irish Branch £93)		
	Hull	149	
	London	563	
	(Inc. Thames Valley Branch £68)		
	(Inc. Southern Branch £50)		
	Manchester	202	
	Midlands	194	
	(Inc. Trent Valley Branch £64)		
	Newcastle	116	
	Scottish	136	
	(Inc. Eastern Branch £78)		
	West Riding	80	
	New South Wales	753	
	Queensland	85	
	South Australian	160	
	Victorian	624	
	West Australian	72	
	South African	242	
	Auckland	119	
	Wellington	156	
3,066			3,812
550	Australian Federal Committee Expenses—(1964 Estimated—1965 See Income)		—
—	Torquay Conference		171
24	Dinner Dance		—
—	Dinner to Board of Federation of Societies for Paint Technology		147
152	Past Presidents' Dinner		177
135	Provision for President's FATIPEC Expenses		135
	Administrative Expenses—		
10,818	Salaries, National Insurance and Staff Pension Scheme	11,693	
278	Temporary Staff	644	
577	Welfare	578	
2,475	Rent, Rates, Lighting, Telephone	2,532	
1,411	Printing and Stationery	1,212	
986	Postages	1,123	
1,962	Other Expenses	1,803	
18,507			19,585
—	Section Chairmen's and A.F.C. Insignia		122
121	Audit Fees		255
12	Incorporation Expenses		—
1,560	Provision for Research Project		1,620
524	Depreciation on Furniture and Fittings and Office Machines		422
106	Foundation Lecture		—
26	Provision for bad Debts		—
100	Provision for Paint Technology Manuals		—
50	Provision for Dilapidations		150
39	Provision for History of Association		200
685	Provision for United Kingdom Income Tax		832
£43,569			£49,204

1964 £		£	£
11,788	Members' Subscriptions		12,058
147	Entrance Fees		245
13,952	Advertisement Revenue		14,134
	Sales:		
6,589	*Journal*	7,106	
441	Other Publications	387	
787	Reprints	1,679	
7,817			9,172
1,578	Income from Investments (Gross)		1,822
135	Ethel Behrens Fund Income from Investments (Gross)		135
7,798	Exhibition Excess of Receipts over Payments		8,078
106	Royalties and Editorial Work in Respect of Paint Technology Manuals		769
—	Australian Federal Committee Income Less Expenditure		321
	Income from Additional Funds Held by Overseas Sections		
	Auckland	30	
	New South Wales	27	
	Victorian	24	
		81	
—	*Less* Expenditure—Queensland	26	
			55
16	Surplus on Sale of Office Furniture		—
18	Provision Against Debt No Longer Required		—
214	Deficit for the Year		2,415
£43,569			£49,204

Annual General Meeting

Since the Annual General Meeting took place on 30 June, the report of this will not appear until the September issue of the *Journal*.

Section Annual Reports

Following a recommendation from the Publications Committee, Council has decided that the Section Annual Reports will no longer be published in the August issue of the *Journal* and this space will be used for additional Transactions and Communications, However, so that Section Committees are fully aware of the activities of other Sections which have not already been reported in the *Journal*, copies of all Section Annual Reports are being sent to the Chairmen and Honorary Secretaries of all Sections and Branches.

London

Thames Valley Branch

Technical meeting

The seventh technical meeting of the 1965-66 session was held at the Royal White Hart Hotel, Beaconsfield, on 24 February 1966, when Dr. H. A. Hampton, formerly of ICI Ltd., Dyestuffs Division, now retired, gave a talk entitled " Looking Back."

Dr. Hampton opened by saying that his talk would be a survey of his experiences during three and a half decades of endeavour in the surface coatings industry. He supposed that there were few men in industry who had been more connected than he with the introduction, development and adoption of synthetic resins for surface coatings.

He joined the British Dyestuffs Corporation Section of ICI in 1930, in the Resin Section of Research Department, where his first job was to help to prepare a report on the patent position of glyptal resins, the name alkyd not having been yet introduced. This proved to be an invaluable experience, providing a useful background for future endeavours. When, after a few months, he got back to the bench, he was concerned with water soluble glyptals. It is interesting to note that more than 30 years ago work was being carried out on this currently topical subject. These aqueous solutions were used mostly as adhesives ; the difficulty was that they lacked adequate storage stability. Dr. Hampton said that from his early experience he is convinced that for real progress to be made on water soluble alkyds, some device to eliminate hydrolysis is necessary—the use of trimellitic anhydride may be the answer.

In the very early 1930s work was going on to produce drying oil modified glyptals and also stoving media based on urea resins. A stoved urea-formaldehyde resin film, plasticised with a non-drying oil modified glyptal, gave a firmly adherent, flexible, scratch-proof, colourless finish, which had good water resistance, which was quite something in 1931.

When BDC decided to use phthalic anhydride to make resins it was first thought that development would lie in the electrical and moulding powder field, and consequently a good deal of work was first done on glyceryl phthalate itself. It soon became evident, however, that neither of these directions offered the real potential for large-scale development, and attention was turned to oil modification, particularly with drying oils. Among the 19 experimental oil modified glyptals evaluated by Dr. Hampton were three which were developed as saleable products and which remained as such until August 1965 when one was withdrawn, the other two still selling in significant amounts, quite an achievement.

After this early work on alkyds, Dr. Hampton left the Research Department to do technical service work. Up to this time alkyds for brushing finishes had been offered as solutions in genuine American turpentine and much effort was devoted to turpentine quality, and the adverse effects obtainable by deviations from the genuine. With the advent of higher viscosity alkyds to eliminate skin drying and improve flow, a thorough investigation of solvents was necessary, and it was concluded that a good grade of high flash white spirit used alone gave the best compromise for brushing alkyd paints. It was, however, quite a problem to get the paint trade to use white spirit ; they were slow to appreciate that different grades existed, and that the results could be varied enormously by changing the grade. This was a simple fact, but it was tremendously important.

Up to the time of the Second World War, alkyds and other synthetic resins were beginning to make an impact on the surface coatings industry, but they had not made deep inroads into the raw material consumption figures. Wartime conditions, with all their shortages, restrictions and limitations, undoubtedly taught the industry much about the value of these new products, so that when supplies became freer, their consumption figures went up by leaps and bounds. Dr. Hampton told of his experiences with wartime surface coating problems, particularly with the formulation of anti-gas paints, an activity which seemed to go on and on to cope with developing raw material shortages.

In the early 1930s, BDC Research Department had as a target the production of a resin which would be much superior to NC as an interlayer for safety glass. This was regarded as such an important target that finally all the chemists in the laboratory were put on the project. The whole project collapsed almost overnight because of the discovery of toughened glass, but a good deal of work had been put in and this led for the first time to polymerised methyl methacrylate. Here was a crystal clear, colourless, tough, high-melting and light-fast polymer which, although this was not known at the time, turned out to be very durable. Early work showed it should be useful as a moulding composition, and this was one of the reasons why its development as such went to another division of ICI. The Dyestuffs Division, however, retained the right to develop methacrylate ester polymers in the surface coatings industry. The latest issue of the *Surface Coatings Resin Index* lists at least 31 products which are based on or contain methacrylate esters. The major paint outlet now is for finishes for automobiles in which the retention of gloss and general appearance, particularly of metallics, is outstanding.

In an endeavour to extend the usefulness of methacrylate ester polymers in surface coatings, a study was made of the effects which could be obtained by using trichlorethylene as the solvent. It soon became clear that very rapid air-drying finishes could be obtained and that a detailed investigation would be worthwhile. Trichlorethylene is the cheapest of the commonly used industrial solvents and in terms of tonnage used or of any other yardstick is one of the major solvents currently used in British industry. It has now been found to give paints with a unique combination of properties —non-inflammable, fast drying, and when used as dipping paints, giving better edge coverage, less runs, tears and fat edges, and less wastage by draining. The weight of trichlorethylene vapour, 4½ times heavier than air, means that a vapour blanket is readily formed over a suitably designed dip tank, and virtually eliminates loss due to evaporation.

Finally, Dr. Hampton dealt with developments in polyurethanes used in rigid and flexible forms, surface coatings, rubbers and adhesives, speaking particularly of the means of overcoming the toxic hazards associated with tolylene di-isocyanate by forming higher molecular weight prepolymers with polyols or by using higher molecular weight isocyanates.

In the discussion which followed, Mr. Drummond thanked Dr. Hampton for taking his mind back to the 'thirties and pointed out that the Thames Valley Branch had been formed mainly because of Dr. Hampton's stimulation and interest.

W. S.

Notes and News

Foundation Lecture and Dinner

Photo by *Lawson & Co.*

(Left to right) E. P. G. Wright (Master, Painter Stainers), A. T. S. Rudram (Hon. Research and Development Officer), Dr. S. H. Bell (President), Dr. N. F. Astbury (Foundation Lecturer), B. G. Wilson (Clerk, Painter Stainers) and R. H. Hamblin (General Secretary)

The Biennial Foundation Lecture followed the Association's Annual General Meeting on 30 June 1966 and was held at Painter Stainers Hall, London, E.C.4. Over 80 members and guests assembled to hear Dr. N. F. Astbury (Director of Research of the British Ceramic Association) present the lecture, which he entitled " Craft Against Craft Makes No Living."

The President (Dr. S. H. Bell), in introducing the lecturer, said that Dr. Astbury came to the Association with a wealth of experience of research, technical education, and of the ways in which such activities were controlled and developed, nationally and by various collective or co-operative arrangements. Dr. Astbury not only knew the background and trends but he had helped to shape them. The President referred to the lecturer's position as Chairman of the Committee of Directors of Research Associations and to his membership of the National

Council for Technological Awards 1958-64 and more recently of the Council for National Academic Awards. Dr. Astbury was also a member of the Advisory Committee for Scientific and Technical Information and a Vice-President of the Parliamentary and Scientific Committee.

The President then gave a brief history of the Foundation Lectures and noted how their titles had all seemed to be highly intriguing! In the choice of title for his lecture and by the synopsis published in the *Journal*, Dr. Astbury had already stimulated the audience to think and the President was sure that everyone would be interested in the development of the theme.

Since the full text of this important lecture will be published in the September issue of the *Journal*, it is not intended to give a report in this issue.

After Dr. Astbury had spoken, the President called upon Dr. H. H. Morgan (Founder Member, Honorary Member and President 1924-26) to propose a vote of thanks. Dr. Morgan stated that he expressed the feelings of all present when he said the lecture had been of absorbing interest and had indeed provided much food for thought. It gave him much pleasure in calling on the members to give Dr. Astbury a hearty vote of thanks, which was shown by prolonged applause.

After the lecture the President conducted Dr. Astbury, the Master and Clerk of the Painter Stainers Company to the Painted Chamber, where an informal reception was held with Past Presidents, Founder Members, Honorary Members and Members of Council. The remainder of the audience enjoyed an informal reception in the Court Room, after which they proceeded to the Dining Hall, where an excellent dinner was served.

After the Loyal Toast, the President commenced his speech by saying that it seemed odd in this late stage of the proceedings to have an Address of Welcome! He was delighted to welcome five Past Presidents : Mr. L. O. Kekwick (1951-53), Mr. C. W. A. Mundy (1955-57), Mr. N. A. Bennett (1957-59), Dr. J. E. Arnold (1963-65) and Dr. H. H. Morgan (1924-26), whose early activities had been bound up with the Foundation of the Association, of which he was both a Founder Member and an Honorary Member. Mr. G. Copping (Honorary Member), who had been so active in recording the events of the early Association and in writing the Association's History, which was to be published in the Jubilee Year, was also present.

The President then welcomed the other guests, beginning with Dr. Astbury, and mentioning also the three Founder Members present, Dr. H. H. Morgan, Mr. A. Z. Molteni and Mr. R. P. L. Britton. He was especially pleased to welcome both the Master and Clerk of the Painter Stainers Company, and addressing Mr. E. P. G. Wright (Master) said that OCCA with its forthcoming Jubilee of 50 years must appear as a mere beginner, compared to his Company, with an ultimate history of at least 700 years. However, the President referred to the Leonardo motif on the insignia and said further that our technologies go back at least as far as cave paintings—a remarkable example of durability! The President then extended a general welcome to all present and offered thanks on behalf of them all to Mr. R. H. Hamblin (General Secretary) and his staff for the excellent arrangements, to the Clerk of the Worshipful Company for his assistance, and through the Master to the Worshipful Company for permission to hold the Foundation Lecture and Dinner at the Hall.

The President proposed a toast of goodwill to the Worshipful Company of Painter Stainers in the dual role of hosts and guests, to which all responded.

Mr. E. P. G. Wright (Master, Painter Stainers), in his reply, thanked the Association for its generous and most enjoyable hospitality. He went on to recall the history of the Company and how its activities had once included disputes with the Saddlers Company as to who should paint saddles and with the Plasterers as to who should stain walls, arguments which went on long after the companies concerned wanted to do either of these!

The Company had then spent over 200 years in conflict with the College of Arms over the achievement of arms.

The later history of the Company, however, had been of devotion to charitable objects. For 250 years they had been involved in charities for the blind and latterly in the educational field. Soon the Worshipful Company would celebrate the quincentenary of the granting of their charter and the Court was very pleased that modern day scientific societies such as OCCA came to the Painter Stainers Hall; he looked forward to increasing co-operation between the Worshipful Company and OCCA in the future.

After speeches, the company dispersed.

OCCA 19

The Association's Nineteenth Technical Exhibition, 1967

OCCA 1967
Technical
Exhibition

The closing date for application to exhibit at next year's Technical Exhibition will be **2 September 1966** and already a large number of applications have been received, not only from the United Kingdom, but also from Continental sources. The Exhibition will take place from 13 to 17 March 1967 at Alexandra Palace, London (the hours of opening being given in the last issue of the *Journal*). Leaflets, a map and directions in six languages (English, French, German, Italian, Russian and Spanish) have been prepared for wide distribution on the Continent. It may be possible to supply copies to members who wish to send copies to their colleagues and associates abroad, if written application is made to the General Secretary. Similarly copies of the *Official Guide*, which will be available early in the New Year, may be obtained without charge by those intending to visit the Exhibition; all members of the Association wherever resident will be sent copies upon publication. It is felt that members may like to read extracts from some of the reports on the last Exhibition which appeared in other journals, which show the high regard in which the Exhibition is held, and some are given below:

> " The very valuable things which the OCCA Exhibition can offer to the colour industry: it offers interesting information on development trends in every sphere of our industry "
> —*Färg och Lack*, April 1966.

Industrial Finishing (April 1966) published some viewpoints of exhibitors, from which the following are extracts:-

> " The OCCA Exhibition is clearly becoming well known and respected abroad. A larger number of overseas visitors were present than in any previous year, and this is certainly an encouraging sign for future exhibitors "—*Laporte Industries Ltd.*

> " When the doors of OCCA are opened the visitors see a magnificent, very professional and orderly display of highly technical exhibits from raw materials to heavy equipment. Compliments from our many overseas friends are continuously free flowing on this first-class example of British enterprise "—*D. H. Industries Ltd.*

> " In general we found the Exhibition to be extremely productive and we were impressed by the high proportion of visitors anxious to discuss the technical aspect of our products "—*Pfizer Ltd.*

> " The many new and important developments and novelties were enough to satisfy the appetite of the most voracious technical visitor.

" This was a very fine Exhibition, unique of its kind and bigger and better than ever. It provides a valuable forum for the collection and exchange of technical information on new developments here and abroad. It must also give a great stimulus to international trade "—*Chemistry and Industry*, 26 March 1966.

" For the European paint and varnish industry the Exhibition held by the ' Oil and Colour Chemists' Association ' has become an important event. This is emphasised not only by the greater number of exhibitors from the Common Market countries, but—above all—by the fact that the number of visitors from the countries the other side of the Channel had increased " —*Fette Seifen Anstrichmittel*, June 1966.

The Exhibition is not confined to British companies, and the Exhibition Committee will be pleased to consider applications for stand space from companies abroad. These should be sent to the General Secretary at the address given at the front of the *Journal*.

8th FATIPEC Congress

The eighth FATIPEC Congress was held between 6-10 June at Scheveningen, Holland, an impression of which is given by Mr. T. R. Bullett in his guest editorial on page 670. Shown above at the farewell party are, standing, left to right : Dr. S. H. Bell (President of OCCA) and Dr. H. W. Talen (Chairman of the Technical Committee of the Congress). Sitting, left to right, are : Ir. P. Ernotte (President-Elect of FATIPEC), Dr. W. J. Nijveld (President of FATIPEC) and F. J. M. A. Heierman (Secretary of the Congress Organising Committee).

Bristol Section

Skittles match

The Annual Skittles Match between Bristol Section and Birmingham Paint, Varnish and Lacquer Club was played this year on a fresh alley at the Royal William, Painswick, on 19 May. There were other unique features about the match this year, for the Bristol Section representatives outnumbered those of the Birmingham PV and L Club and included a number of young ladies whose presence and skill at skittles enlivened the proceedings considerably. The " Alkyd Cup " was won for the third time in succession by the Birmingham Club, sustained by members on loan from the Bristol Section, by eight points in 380.

The light refreshments and convivial evening were very much enjoyed by all who attended.

Hull Section

Annual dinner-dance

The 1966 Annual Dinner-Dance of the Hull Section will be held on Friday 14 October 1966. The venue for this occasion has been changed this year to the Eden Hotel, Willerby, Nr. Hull, Yorkshire. Tickets, 35s. each, will be available from the Hon. Social Secretary : T. A. Fillingham, Esq., 37 Southfield, Hessle, Yorkshire.

Newcastle Section

Symposium : " Technical Training in the Paint Industry : The Industrial Training Act "

More than 40 members and visitors, including some from other Sections, attended this symposium, which was held at Rutherford College of Technology, Newcastle-upon-Tyne, on the afternoon of 25 May 1966. Mr. E. L. Farrow was in the chair and there were eight speakers. There was a lively discussion after each paper.

Mr. A. Tenick, of the Ministry of Labour Regional Office, spoke on the Industrial Training Act in general. The Training Board covering the paint industry would probably cover chemicals, oils, plastics and rubber, and be set up early next year. The operation of the levy and grant systems was explained, and Mr. Tenick explained in detail the functions of the Boards and the committees set up by them. Regarding the cost to the industry, he was most emphatic that, from his own experience, the cost was amply repaid, not only by a long-term rise in productivity, but also by immediate savings due to elimination of waste.

Mr. G. J. Lumb, of the Paint Makers Association, described the history of technical education in the industry over the past decade. The attitude of the Technical Education Committee of the PMA was that basic science should come first and education in technology afterwards. Arrangements for technical and technological education, through the National Certificate and City and Guilds courses, were now quite good, and his Committee was presently concerned with devising courses at a lower level, for operatives, salesmen and others. The PMA had been in touch with the Ministry about the Act, but so far had heard nothing definite.

Mr. J. J. Lawler, of East Ham Technical College, described the former City and Guilds and National Certificates courses, and went on to the revised schemes. The important change in the City and Guilds courses was that it followed on from the Chemical Technicians' Certificate, and therefore specialisation in paint technology was deferred to a later age than hitherto.

Mr. L. K. Street, of the College of Further Education, Newcastle-upon-Tyne, spoke about training courses for operatives. Such courses were rare in the science-based industries, and he had had great difficulty in interesting certain employers (dentists, for example) in them. The courses would have to be based either on the college or on industry, with day- or block-release from one to the other. Three types of course might be run : a basic course for operatives, with emphasis on covering a wide range of operations ; a higher grade for people with supervisory potentialities ; and an intermediate type for those concerned with buying and selling rather than production.

Mr. L. H. W. Hallett, of Rutherford College, spoke on the courses available for technologists, starting at GCE " A " level or ONC, and going up to Hons. Degree standard. The paint industry was not strong enough to support its own institution : training would therefore have to be based on the College, with help from the industry. After describing the courses leading to HNC, LRIC, GRIC and B.Sc.(Tech.), Mr. Hallett considered post-graduate study. Locally there had been attempts to run advanced courses on special subjects, and nationally it was probable that arrangements would be made for people to gain degrees equivalent to M.Sc. and Ph.D. without registering as students at a university. Two existing problems were the rather poor communication between the colleges and industry—it was up to employers to make more enquiries about available facilities ; and the supply of teachers for the technological subjects—teaching, it appeared, did not attract people from industry.

Mr. G. L. Lewis, of International Paints Ltd., started by criticising both the Ministry of Labour and the Paint Makers' Association for dilatoriness in implementing the proposals for training in the industry. Referring to a point of view which had been expressed, that some workers were ineducable, he dissented : even the shop-floor sweeper could be trained to be a better sweeper. The industry must realise the benefits of training, and take it up with enthusiasm. The Training Officer and the personnel department were the most important factors in any training scheme. The first thing was to appoint and train a Training Officer : then he must survey the organisation. Training of supervisory management was a most important step. However, a Training Officer must know how much money he had available, and the delay in setting up a Board made this difficult at present.

Mr. N. Richardson, of British Paints Ltd., said that he had been appointed Training Officer, at present on a part-time basis, and he described the results of his preparatory work. He estimated the levy to the industry as probably in the region of 2.5 per cent of the payroll. Regarding courses, he counselled delay, because the Board would probably demand that they conform to its own patterns. The first job was to keep records of the training that is actually being done and the cost of it. Training of supervisors, particularly in techniques of instruction and communication, was the key to works training. The best place for such training was within industry, rather than in a college, because of the diversity of supervisory jobs. Training of managers, for example for overseas factories, was done in his company by taking people from the laboratories and putting them in the works to do method studies ; also by entering them into college courses in management.

Dr. H. W. Keenan wound up the symposium. He said that 50 years ago he was the first apprentice indentured in the paint industry. After paying a special tribute to the contribution of the college representatives to the symposium, he referred to the problem of providing teachers. He then went on to outline his vision of the industry in 30 years' time, one of the most interesting features being an end of the " rat-race of useless competition." In ending the symposium he congratulated the Section on organising it, and said that its only fault was in being too short.

Programme Liaison Committee

The Liaison Committee, comprising two representatives each from the Institution of the Rubber Industry (*IRI*), the Plastics Institute (*PI*), the Plastics and Polymer Group of the Society of Chemical Industry (*SCI*), the Society of Dyers and Colourists (*SODAC*) and *OCCA*, was formed in order to obviate the inconvenience caused by the clashing of dates of symposia, conferences, exhibitions, etc., of the different bodies. Forthcoming major meetings or conferences on polymers or related topics planned by the above societies and others are given below ; they will be held in London, unless otherwise stated.

1966

5-9 September : Summer School in Leicester, *SODAC*.

19 September : Lecture on the life and work of Professor H. Staudinger, by Dr. V. E. Yarsley, *SCI*.

20-22 September : Joint Conference on " Advances in Polymer Science and Technology," *SCI/IRI/PI*, in association with *OCCA* and *SODAC*.

27 September : AGM in association with North-Eastern Section Symposium on " Cellular Plastics in Insulation," *PI*.

29 September : Mercer Lecture in Manchester, *SODAC*.

November (date not yet advised) : Fourth Swinburne Award Address, *PI*.

15 and 16 November : Conference on " The Coating of Plastics with Metals," *PI*.

1967

4-6 January : Teaching Seminar on " Plastics Design Engineering—Theory and Practice " (at Cranfield), *PI*.

13-17 March : 19th Technical Exhibition, *OCCA*.

11 and 12 April : Conference on " Plastics in Telecommunication Cables," *PI*.

28 April : AGM and Dinner in Manchester, *SODAC*.

15-18 May : International Rubber Conference and Exhibition (at Brighton), *IRI*.

19 and 20 June : Joint Conference on " Plastics and the Production Engineer," *IProdE/PI*

20-24 June : Biennial Conference at Scarborough on " Interfacial Behaviour," *OCCA*.

October : RPG Conference on " Filament Winding," *PI*.

15 and 16 November : Conference on Packaging with Plastics," *PI*.

More standard methods of tests for paints

The latest additions to B.S. 3900 : METHODS OF TESTS FOR PAINTS published by the British Standards Institution include optical, mechanical, durability and environmental tests and tests for drying times. The first parts of the standard were published in June 1965, and with the present series bring the number of tests issued to 20.

The new parts are as follows :

Group C	—Tests associated with paint film formation	
Part C2	—Surface drying time ..	1s.
Part C3	—Hard drying time ..	4s.
Group D	—Optical tests on paint films	
Part D1	—Colour comparison ..	1s.
Group E	—Mechanical tests on paint films	
Part E1	—Bend test	4s.
Part E2	—Scratch resistance ..	5s.
Part E3	—Resistance to impact (falling weight)	4s.
Group F	—Durability tests on paint films	
Part F1	—Alkali resistance of plaster primer-sealer	2s.
Part F2	—Resistance to humidity under condensation conditions	2s.

Part F3 —Resistance to artificial weathering (enclosed carbon arc) .. .. .. 4s.

Group G—Environmental tests on paint films

Part G3 —Resistance to spotting by liquids .. .. .. 1s.

Copies of B.S. 3900 may be obtained from the BSI Sales Branch, 2 Park Street, London W.1. (Postage 6d. will be charged extra to non-subscribers).

Association tie

Members are reminded that the Association's tie is available at 20s. sterling, post free (by air mail to addresses outside Europe 5s. extra) from Thresher & Glenny Ltd., Lancaster Place, Strand, London, W.C.2. The tie, which is in dark blue "Terylene," carries a reproduction of the Association's insignia in red and gold silk, and is available only to members of the Association.

New Zealand Sections' Convention

The 1966 Convention of the Wellington and Auckland Sections will again be held at Wairakei from 12 to 14 August. This year the Wellington Section are responsible for the organisation and any members of the Association who intend to visit New Zealand during August and wish to attend the Convention will be welcomed. Application should be sent by cable to W. J. James, P.O. 5019, Wellington, New Zealand.

News of Members

Mr. W. T. Branscombe, an Associate Member attached to London Section, has retired as deputy chairman of Pinchin Johnson and Associates Limited, the Courtaulds paint subsidiary.

Mr. D. Hastilow, an Ordinary Member attached to the General Overseas Section, has been appointed Chief Chemist of Valvoline (Australia) Pty. Ltd. with effect from 1 July 1966.

Mr. A. D. Hibberd, a past Chairman of Newcastle Section, has joined the East Anglia Lacquer Co. Ltd., where he will have full responsibility for the overall progress of the company in both technical and administrative sections.

Mr. A. T. S. Rudram, Honorary Research and Development Officer of the Association and a past Chairman of London Section, is joining the board of Sissons Brothers & Co. Ltd. on 1 September. He will take over the duties of technical director from Mr. S. S. Done, a past Chairman of the Hull Section, who will remain a member of the board until his retirement next year.

Obituary

Herbert L. Howard

Mr. Herbert L. Howard died on 3 June at the age of 68.

He worked for over 30 years in various firms dealing with the technology of oils, pigments, resins, inks and paints, and was at one time Chief Chemist to Burrell & Co. Ltd., of Millwall. His keen interest in education led him to work as a part-time lecturer in his special subject and after nine years' work at the Borough Polytechnic he became Head of the Science Department of the East Ham Technical College in 1947. During the last ten years of his life he became Lecturer in Science at the London College of Printing and Graphic Arts. Here he was able to study and to publish some articles on the history of printing and printing inks. He was also responsible for initiating a number of researches on printing processes, such as etching.

He was a Fellow of the Royal Institute of Chemistry and obtained a B.Sc. degree in Chemistry at Imperial College. He was elected an Ordinary Member of the Association and took an active part at Conferences and Section meetings. He also held office in the British Association of Chemists, first as Registrar and then as President.

G. J. C.

Section programmes for 1966-67 session

Main Association events

1967

Monday 13-Friday 17 March

OCCA 19. (Nineteenth Technical Exhibition), Alexandra Palace, London, N.22.

Tuesday 20-Saturday 24 June

ASSOCIATION BIENNIAL CONFERENCE on "INTERFACIAL BEHAVIOUR." Scarborough.

Friday 23 June

ANNUAL GENERAL MEETING Grand Hotel, Scarborough.

Bristol

Meetings will be held at the Royal Hotel, College Green, Bristol 1, at 7.15 p.m., unless otherwise stated.

1966

Friday 30 September

"Trichlorethylene Paints," by A. H. McQuillan.

Friday 28 October

"The Strip Coating of Steel with Paint and Plastic Finishes," by D. S. Newton, to be held at the Angel Hotel, Cardiff.

Friday 25 November

"Dispersion Techniques," by I. Berg. (Joint meeting with the Birmingham Paint, Varnish and Lacquer Club, to be held at the Hawthorns Hotel, Bristol.)

1967

Friday 6 January

"Measurement of Colour by Instrumentation," by P. V. Foote.

Friday 27 January

"The Use of a Computer to Control a Batch Reaction," by J. M. F. Drake.

Friday 24 February

"Solvents," by L. A. Tysall.

Friday 31 March

"Industrialised Housing," by K. F. J. Humphreys.

Friday 28 April

Annual General Meeting, to be followed by a Film Show.

Hull

Meetings will be held at the Royal Station Hotel, Hull, at 7 p.m., unless otherwise stated.

1966

Monday 3 October

"Colour," by W. B. Cork.

Friday 14 October

Twenty-third Annual Dinner Dance, at the Hotel Eden, at 7 p.m.

Monday 7 November

"Marine Coatings," by J. C. Kingcome.

Monday 5 December

"Tall Oil," by G. Winfield. (Joint meeting with SCI Oils and Fats Group).

1967

Monday 2 January
Hull Section Co-operative Research Programme.

Thursday 19 January
Discussion evening, at the Queens Hotel, George Street, Hull.

Monday 6 February
" Electronic Data Processing," by C. Campbell.

Monday 6 March
" Powder Coatings," by B. Y. Downing or F. E. Bassford.

Irish

Meetings will be held at the Clarence Hotel, Wellington Quay, Dublin, at 8 p.m., unless otherwise stated.

1966

Friday 30 September
" PVA Pigmentation," by L. Tasker.

Friday 28 October
" Developments in Media for Printing Inks-Paints," by G. H. Hutchinson.

Friday 25 November
" Carbon Black—Its Uses and Problems, by N. Scott.

Thursday 8 December
Annual Dinner Dance, at the South County Hotel.

1967

Friday 27 January
Open Forum.

Wednesday 8 February
Works Visit to Messrs. Lever Brothers, at 3 p.m.

Also in February—time and date later —Social Evening (including visit to Telefis Eireann (Studios).

Friday 31 March
Annual General Meeting, at 7.30 p.m., followed by " The Manufacture and Uses of Wallboard," by Bowaters Irish Wallboard Mills Limited (Film).

London

Meetings will be held in the Physics Department, Imperial College of Science and Technology, South Kensington, London, S.W.7, at 6.30 p.m., except for that on Wednesday, 9 November.

1966

Wednesday 14 September
" Problems Of Paint Users," by D. A. Bayliss.

Thursday 20 October
" The Behaviour Of Printing Ink On Rollers," by C. C. Mill.

Wednesday 9 November
Half-day Symposium on " Operational Research Methods."

Thursday 8 December
" Metallo-Organic Primer Coatings," by R. N. Faulkner.

1967

Wednesday 18 January
" Evaluation for Electrodeposition of Recently Developed Water Soluble Epoxy Esters," by L. A. Tysall and J. Van Westrenen.

Thursday 16 February
" The Printing of Bank of England Notes," by Dr. A. C. Healey.

Wednesday 22 March
" European Liaison Lecture."

Southern Branch

All Southampton meetings to be held at the Royal Hotel, at 7.30 p.m. All Portsmouth meetings to be held at the Keppels Head Hotel Southsea, at 7.30 p.m., unless otherwise stated.

1966

Monday 10 October

" Polyurethanes—Progress in the Surface Coating Industry," by G. Sutno, at Southampton.

Monday 14 November

" Grinding," by A. C. B. Mathews, at Portsmouth.

Friday 25 November

Discussion evening, " Paint—Fact or Fiction," at the Keppels Head Hotel, Portsmouth.

Monday 12 December

" Car Finishes," by H. A. Shelton, at Southampton.

1967

Monday 9 January

" Paint Test Methods," by T. R. Bullett, at Portsmouth.

Monday 13 February

" Electrostatic Application of Powder Coatings," by Dr. A. W. Bright, at Southampton.

Monday 13 March

" Polyesters " by A. A. K. Whitehouse at Portsmouth

Monday 10 April

Annual General Meeting, at Portsmouth.

Thames Valley Branch

All meetings will be held at the Royal White Hart Hotel, Beaconsfield, Buckinghamshire, at 7 p.m.

1966

Thursday 29 September

" Grinding," by A. C. B. Mathews.

Thursday 27 October

" Paints to meet Motorcar Manufacturers' Specifications," by I. Walker.

Wednesday 30 November

" Accelerated Weathering," by H. A. Hipwood, plus another speaker.

Thursday 15 December

" Co-Polymers with Vinyl Esters of Branched Carboxylic Acids in Thermosetting Systems," by W. H. M. Nieuwenhuis.

1967

Thursday 18 January

" The use of computers in Industry," by A. Davies.

Friday 17 February

Ladies' Night, buffet supper and dance at Great Foster Hotel, Egham, Surrey.

Tuesday 21 February

" Decorative Paints and Decoration " —a discussion group.

Wednesday 29 March

" Some Recent Developments in Paint Testing Instruments," by C. J. H. Monk.

Wednesday 12 April

Annual General Meeting

Manchester

1966

Friday 23 September

Works visit to The Wallpaper Manufacturers Ltd., Holmes Chapel Branch, and Jodrell Bank.

Friday 14 October

" Strip Coating," by D. S. Newton. (Joint meeting with the North-West Branch of the Institute of Metal Finishing), at the Manchester Literary and Philosophical Society, at 6.30 p.m.

Friday 28 October

Annual Dinner and Dance, at the Midland Hotel, Manchester.

Friday 11 November

" Electrical Methods of Evaluating the Corrosion Resistance of Painted Specimens," by H. F. Clay, at the Manchester Literary and Philosophical Society, at 6.30 p.m.

Friday 9 December

" Various Aspects of the Preparation and Testing of Recently Developed Water Soluble Epoxy Esters," by J. Van Westrenen and L. A. Tysall, at the Strand Hotel, Brunswick Street, Liverpool, at 6.30 p.m.

1967

Friday 13 January

" What Kind of Staff Does the Paint Industry Need ? " by M. H. M. Arnold, at the Manchester Literary and Philosophical Society, at 6.30 p.m.

Friday 10 February

" The Restoration of Works of Art," by A. R. Ashton, at the Manchester Literary and Philosophical Society, at 6.30 p.m.

Friday 10 March

" The Development of Alkyd based Powder Coatings," by B. Y. Downing and F. E. Bassford, at the Strand Hotel, Brunswick Street, Liverpool, at 6.30 p.m.

Friday 7 April

Annual General Meeting—provisionally in the Pavilion Suite, Lancashire County Cricket Club, Trafford Park, Manchester 16, at 6.30 p.m.

For junior members

1966

Tuesday 6 September

Junior Lecture : " Basic Rheology—What it can tell you," by A. R. H. Tawn, at the Manchester Literary and Philosophical Society, 36 George Street, Manchester 1, at 4 p.m.

1967

Friday 31 March

Junior Lecture : " Synthetic Resin Manufacturing Plant," by I. C. Sime, at the Manchester Literary and Philosophical Society, at 4 p.m.

Midlands

Meetings will be held at Chamber of Commerce House, 75 Harborne Road, Birmingham 15, at 6.30 p.m., unless otherwise stated.

1966

Friday 16 September

" Continuous Strip Coating," by E. E. V. Sharpe.

Thursday 20 October

" Solvent or Solventless," Symposium, at Regency Club, Shirley, at 10.30 a.m.

Friday 18 November

" The Printing of Bank of England Notes," by Dr. A. C. Healey, at the Wolverhampton and Staffordshire Technical College, at 7.00 p.m.

1967

Thursday 5 January

Annual Ladies' Evening, at the George Hotel, Solihull, at 7.00 p.m.

Friday 20 January

" Surface Coatings in Nuclear Establishments," by W. J. Colclough.

Friday 17 February

" Polymer Characterisation," by Dr. F. W. Peaker.

Friday 10 March

Newton Friend Lecture : " Materials and Skills of the Jeweller."

Precious Metals—R. W. Dew.
Precious Stones and Gems—N. Harper.
Setting and Mounting—J. Rossiter.

Friday 21 April

Annual General Meeting, at 7 p.m. Venue to be arranged.

Trent Valley Branch

All meetings will be held at the British Rail School of Transport, London Road, Derby.

1966

Thursday 6 October

" Versatic Acids and their Derivatives," by M. V. Moore, at 7.30 p.m.

Thursday 10 November

" Interesting Developments in Emulsion Paints," by P. M. Troll, at 7.30 p.m.

1967

Thursday 12 January

" Castor Oil Chemistry," by Dr. G. Silverstone, at 7.30 p.m.

Thursday 23 February

" Electrical Methods of Assessing the Protection of Paints Against Corrosion," by H. F. Clay, at 7.30 p.m.

Thursday 6 April

Annual General Meeting, at 7 p.m.

Newcastle

Scientific meetings will be held at the Royal Turk's Head Hotel, Grey Street, Newcastle upon Tyne, at 6.30 p.m., unless otherwise stated.

1966

Thursday 6 October

" Metallo-Organic Primer Coatings," by R. N. Faulkner.

Thursday 3 November

" Recent Advances in Accelerated Weathering," by T. M. Hipwood and M. W. Northway.

Thursday 1 December

" Trends in the Building Industry," by P. B. L. Keate. (Joint meeting with the local branch of the Federation of Master Painters and Decorators.)

1967

Thursday 5 January

" Newer Methods of Paint Application," by F. G. Dunkley.

Thursday 2 February

" A Scientist Turns to Crime," by K. J. Kimber.

Tuesday 7 February

Evening Visit to the Northern Forensic Science Laboratory, at Gosforth.

Friday 24 February

Ladies' Night, at the Five Bridges Hotel, Gateshead.

Thursday 2 March

Afternoon Visit to British Oxygen Chemicals Ltd., Birtley, followed in the evening by a paper by M. R. Porter—" Tall Oil."

Thursday 6 April

Annual General Meeting.

Scottish

All Technical Meetings will be held in the Lorne Hotel, Sauchiehall Street, Glasgow, commencing at 6 p.m.

1966

Thursday 13 October

" Fish Oils, their Composition, Processing and some Industrial Applications," by S. A. Reed.

Thursday 10 November

" Quality Control of Organic Pigments," by Dr. J. D. Sanders.

Thursday 8 December

" Modern Commercial Management," by H. B. Johnson.

1967

Friday 13 January

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

Thursday 19 January

" Some Industrial Applications of Water Based Paints" by a speaker from Vinyl Products Ltd.

Thursday 9 February

"Aerosols" by a speaker from Metal Box Co. Ltd.

Thursday 9 March

" The Use of Microscopy in the Investigation of Paint Defects," by M. Wells.

Friday 7 April

Annual General Meetings, at Lorne Hotel, at 6 p.m. Followed by Annual Smoking Concert, at the Eglinton Arms Hotel, Eaglesham, at 7.30 p.m.

Eastern Branch

All Meetings will be held at the North British Hotel, Princes Street, Edinburgh, at 7.30 p.m. unless otherwise stated.

1966

Wednesday 26 October

" Tall Oil and its Products," by H. R. Porter.

Wednesday 23 November

" Photography in Industry and Commerce," by T. E. Gray.

Wednesday 14 December

" Recent Developments in the Use of Organic Titanium Compounds," by R. H. Stanley and G. H. J. Neville.

1967

Wednesday 25 January

" Electrical Methods of Assessing the Protection provided by Paints against Corrosion," by H. F. Clay.

Wednesday 22 February

" Applications of Computers in Industry," by C. T. Campbell.

Wednesday 22 March

Annual General Meeting—followed by film " Polyurethanes."

Wednesday 19 April

" Epoxy Curing Agents from Conjugated Esters," by R. V. Crawford.

Student Group

All meetings will take place at the Lorne Hotel, Sauchiehall Street, Glasgow, at 10 a.m.

Saturday 15 October

" Colour Consciousness," by F. M. Smith.

1966

Saturday 12 November

1. " Primers," by G. Scott.
2. " Epoxy Resins," by A. Pisacane.
3. " Oils and Oil Modified Resins—Polymer Systems," by G. H. Hutchinson.

Saturday 10 December

" Works Management," by J. Louden.

1967

Saturday 14 January

" Forensic Medicine," by D. Jack.

Saturday 11 February

Discussion Morning—Annual General Meeting.

Saturday 11 March

" Laboratory Equipment and its Uses," speaker from British Titan Products Ltd.

West Riding

Technical meetings will be held at the Great Northern Hotel, Leeds, at 7.30 p.m.

1966

Tuesday 13 September

SYMPOSIUM ON DISPERSION

" High Speed Mixing," by J. de Jong of Morehouse International. " Ball Milling," by I. R. Sheppard of Steel and Cowlishaw. " Sand Milling," by D. P. Sullivan of D. H. Industries.

Tuesday 11 October

" Modern Developments in Printing Inks and Paper Varnishes," by J. A. L. Hawkey.

Tuesday 8 November

" Organic Dichromates in Corrosion Protection," by C. A. Vessey.

Friday 25 November

Annual Dinner and Dance, at the Granby Hotel, Harrogate.

Tuesday 13 December

" Protection by Patents and Trademarks," by Dr. G. Gansser.

1967

Tuesday 10 January

" Plaster-type Paints from Plastic Emulsions," by Dr. Disselhoff.

Tuesday 14 February

" Plastics in Building," by J. R. Crowder.

Wednesday 8 March

" Tall Oil," by Dr. Winfield.

Tuesday 11 April

Annual General Meeting.

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

APPERLEY, THOMAS WILLIAM JAMES, B.SC., Dyeing Section, Department of Chemical Technology, Bradford Institute of Technology, Bradford, 7. (*West Riding*)
BARCLAY, JAMES FRASER ALLAN, 22 Rangeview Drive, Newton, South Australia. (*South Australia*)
BAYLIS, BERNARD MALPAS, Phoenix Lacquer Co. Pty. Ltd., 72/76 Canterbury Road, Bankstown, New South Wales, Australia. (*New South Wales*)

CAMPBELL, JOHN GERALD, 15 Bimbadeen Avenue, Lugarno, New South Wales, Australia. *(New South Wales)*

CAST, CHARLES HERBERT, 2 Paine Street, Maroubra, New South Wales, Australia. *(New South Wales)*

CASTELLO, VICTOR ANGELO, 14 Hawthorn Crescent, Hawthorn, South Australia. *(South Australia)*

CHAPMAN, RONALD THOMAS CRAWFORD, 4 Valda Avenue, Box Hill North, E.12, Victoria, Australia. *(Victoria)*

DAWSON, RONLEY JOHN, Australian Titan Products Pty. Ltd., Newson Street, Ascot Vale, Melbourne, Victoria, Australia. *(Victoria)*

ELTRINGHAM, JAMES NORMAN, 3a Kelvyn Grove, Manurewa, Auckland, New Zealand. *(Auckland)*

FLETCHER, IAN DAVID, 10 Turnbull Avenue, North Clayton, Victoria, Australia. *(Victoria)*

FOWKES, GRAHAME WILLIAM, 3 Woodman Close, Halesowen, Nr. Birmingham. *(Midlands)*

GLEN, HUGH, 2 Moidart Court, Barrhead, Nr. Glasgow. *(Scottish)*

GOOCH, COLIN, Flat 2, 52 Scenic Drive, Manurewa, Auckland, New Zealand. *(Auckland)*

HILL, ROGER FREDERICK, B.SC., A.R.I.C., The Laboratories, BIP Chemicals Ltd., Tatbank Road, Oldbury, Birmingham. *(Midlands)*

KIRK, C. J., L.R.I.C., 29 Park Road, Duffield, Derbyshire. *(Midlands)*

KIRKBRIGHT, JOHN MICHAEL, " Grange Close," Littlethorpe Road, Ripon, Yorkshire. *(West Riding)*

KNIGHTS, FREDERICK ANTHONY, 76 Lynbrooke Avenue, Auckland, S.W.3, New Zealand. *(Auckland)*

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Forthcoming Events

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

Monday 8 August

Victorian Section. " Gloss Latex Paints," by Dr. B. James, to be held at Union House, Melbourne University, Parkville.

Tuesday 16 August

Western Australian Section. Golf Day to be held at the Western Australian Golf Club. Details from Section Secretary, G. D. Parsons, c/o Polymer Corporation Interstate Pty. Ltd., 1141 Hay Street, Perth.

Thursday 18 August

New South Wales Section. " A Chemical View of the Tanning Industry," by Mr. G. Turner (Richard Hodgson & Sons (Aust.) Pty. Ltd.).

Tuesday 6 September

Manchester Section. Junior Lecture : " Basic Rheology—What it can tell you," by Mr. A. R. H. Tawn (Cray Valley Products Ltd.) to be held at Manchester Literary and Philosophical Society, 36 George Street, Manchester, 1, at 4 p.m.

Saturday 10 September

Scottish Section—Student Group. Film Show **has been cancelled**

Monday 12 September

Victorian Section. " Security Printing," by Mr. W. Steggles, to be held at Union House, Melbourne University, Parkville.

Tuesday 13 September

West Riding Section. Symposium on Dispersion. " High Speed Mixing," by Mr. J. de Jong (Morehouse International). " Ball Milling," by Mr. I. R. Sheppard (Steele & Cowlishaw). " Sand Milling," by Mr. D. P. Sullivan (D. H. Industries).

Wednesday 14 September

London Section. " Problems of Paint Users," by Mr. D. A. Bayliss (CEGB) to be held at Physics Dept., Imperial College of Science and Technology, South Kensington, London, S.W.7, at 6.30 p.m.

Thursday 15 September

New South Wales Section. " Silicone Co-polymers—their manufacture and use," by Mr. J. L. Vinson and Mr. H. G. Stephen.

Friday 16 September

Midlands Section. " Continuous Strip Coating," by Mr. E. E. V. Sharpe to be held at Chamber of Commerce House, 75 Harborne Road, Birmingham, 15, at 6.30 p.m.

Monday 19 September

Western Australian Section. Lecture (to be arranged).

Friday 23 September

Manchester Section. Works Visit—The Wallpaper Manufacturers Ltd., Holmes Chapel Branch and Jodrell Bank.

Thursday 29 September

London Section—Thames Valley Branch. " Grinding," by Mr. A. C. B. Mathews, to be held at Royal White Hart Hotel, Beaconsfield, Bucks., at 7 p.m.

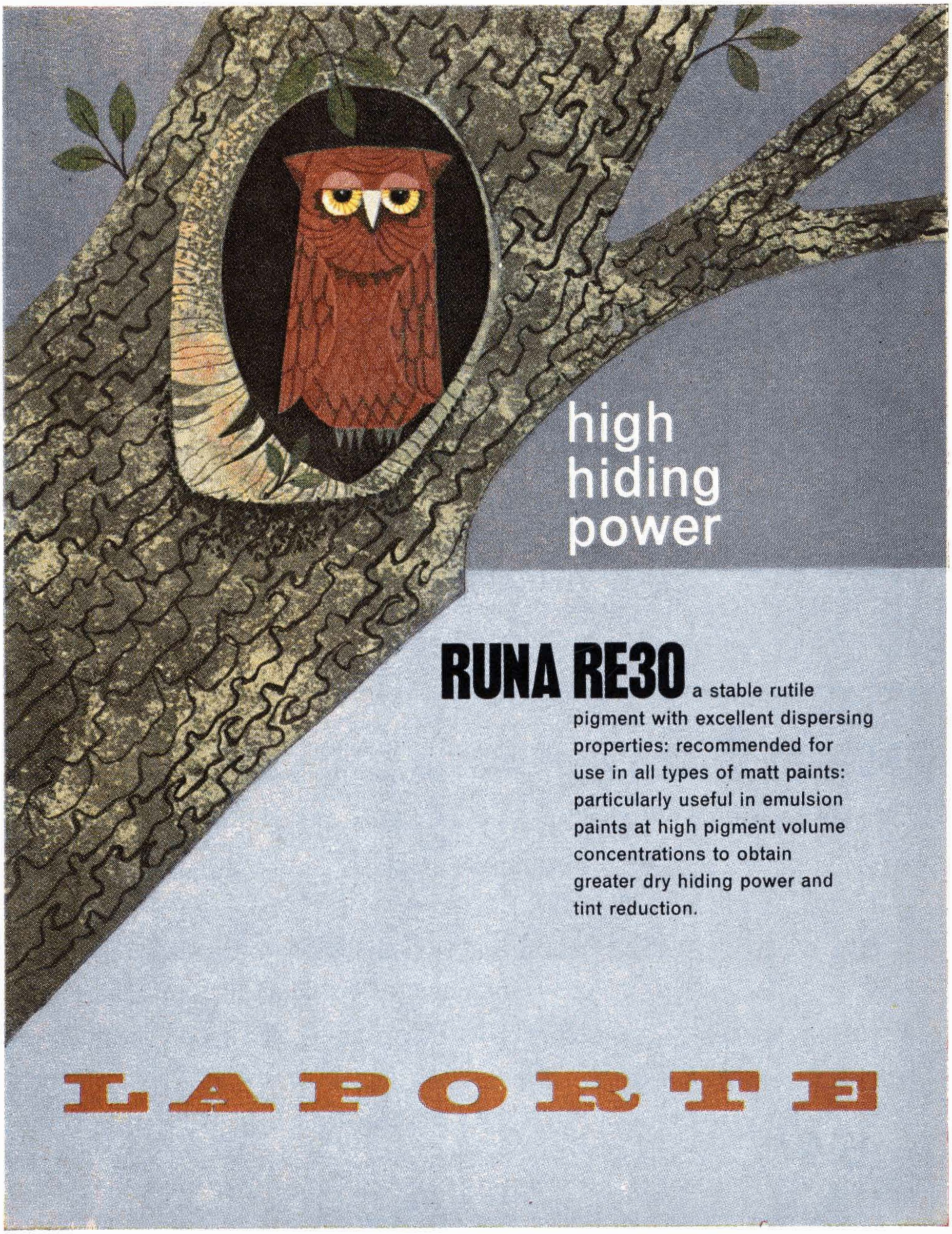
Friday 30 September

Bristol Section. " Trichlorethylene Paints," by Mr. A. H. McQuillan (ICI Ltd.), to be held at the Royal Hotel, College Green, Bristol, 1, at 7.15 p.m.

Irish Section. " PVA Pigmentation," by Mr. L. Tasker, to be held at the Clarence Hotel, Wellington Quay, Dublin, at 8 p.m.

Thursday 20 October

New South Wales Section. " Metal protection with Vinyls," by Mr. B. Godard, (Union Carbide Inc., USA).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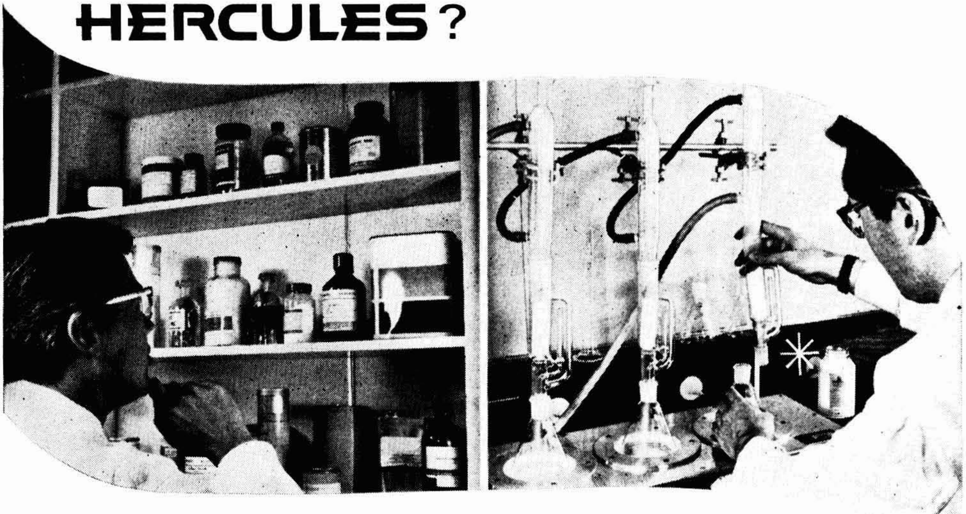

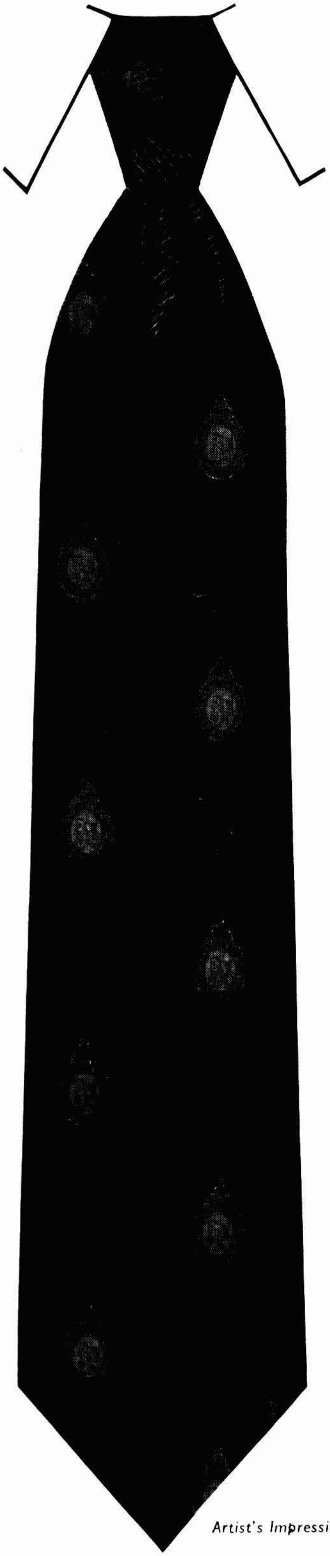

Artist's Impression

O.C.C.A. TIE

The Council has authorised the production of an exclusive tie for Members of the Oil & Colour Chemists' Association. The Association's insignia is woven in red and gold silk on a dark blue terylene background.

The tie is sold only to Members of the Association for **20s.** (sterling) post free. If Members resident outside the continent of Europe wish the tie to be sent by airmail **5s.** (sterling) should be added to cover postage and insurance. The tie is available only from :

THRESHER & GLENNY

Lancaster Place, Strand, London, W.C.2

To : Thresher and Glenny Ltd.,
Lancaster Place, Strand, London, W.C.2.

As a Member of the Association please accept my order for an O.C.C.A. Tie. Remittance to the value of **20s.** (sterling) (if required to be sent by **airmail** to addresses outside Europe, please add **5s.** (sterling) to cover postage and insurance) is enclosed.

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August 1966

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A Castrol Company

is concerned with the development and manufacture of jointing and sealing compounds for buildings, roads, runways, the motor industry and in anti-corrosion coatings for use throughout the world. The following vacancies exist in our Development and Works Laboratories in London, N.W.10, and Bracknell New Town, Berkshire.

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LIL

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LAPORTE TITANIUM LIMITED
(a member of the Laporte Group of Companies)

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A WELL-KNOWN
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Requires an
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To sell to the Paint, Varnish
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Technical background desirable
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Good salary and prospects.
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OCCA 19
13-17 MARCH 1967

The closing date for application to exhibit at the next Tech. Exhibition: 2 September 1966

CLASSIFIED ADVERTISEMENTS

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British Titan Products Co Ltd

invite applications from experienced science graduates, for a limited number of posts as

TECHNICAL OFFICERS

to give increased support to our flourishing export market of approximately £10 million per annum.

The Company, which is one of the world's largest producers of Titanium Dioxide, has an impressive growth record. Its products are universally used for whitening and opacifying all kinds of surface coatings and allied materials, e.g. paints, plastics, paper, printing inks, vitreous enamels and floor coverings.

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If you possess

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- Good appearance and positive personality

we are interested in you, and if you would like further details, write in confidence to :—

The Personnel Manager,
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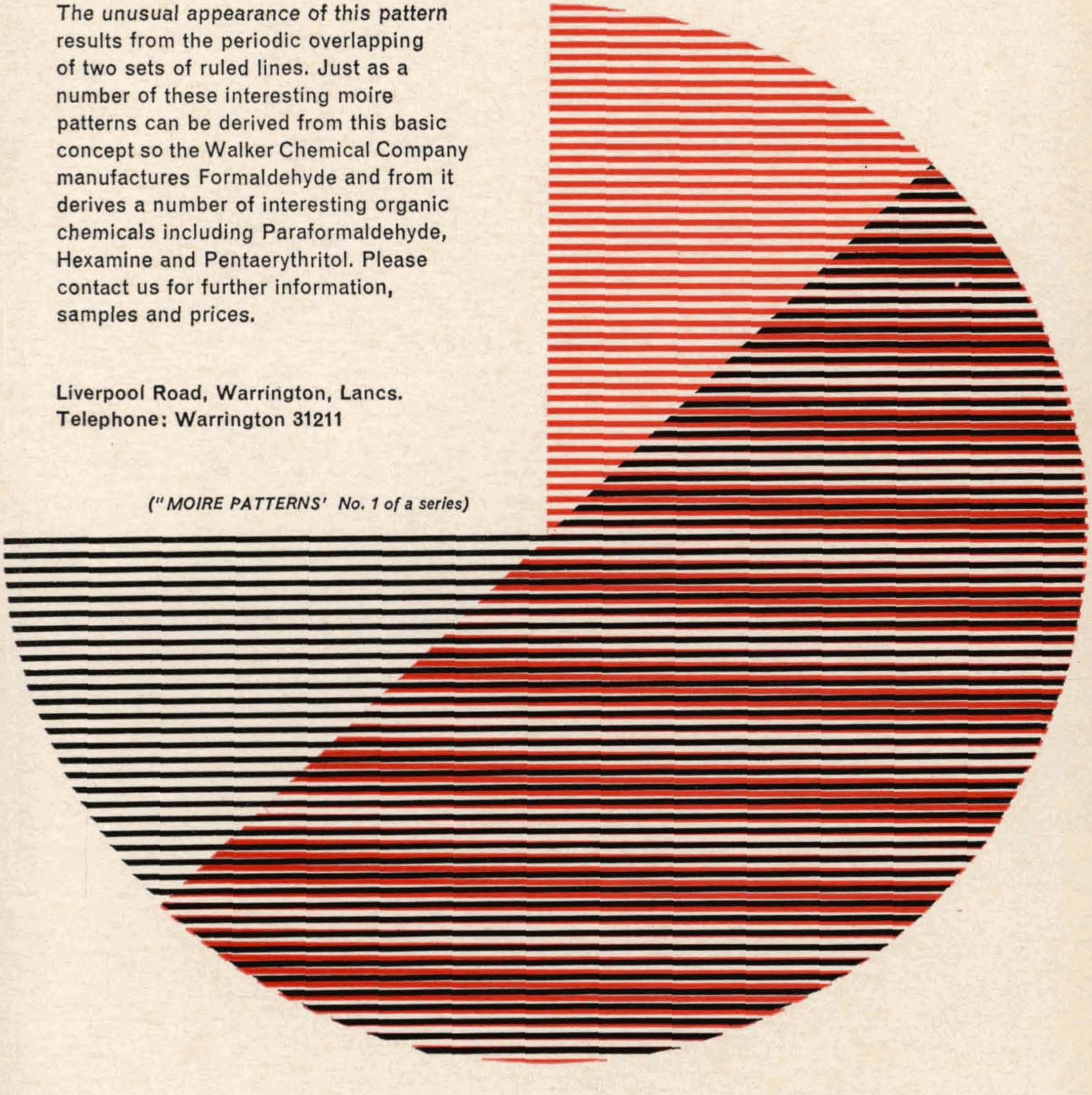
Young graduate wanted for varied development and technical service work in expanding industrial division concerned with new surface coating processes. Commercial aptitude an advantage. Excellent salary and conditions. Write with full details of career to date to Personnel Officer, Lewis Berger (Great Britain) Ltd., Freshwater Road, Chadwell Heath, Essex.

BOOKS WANTED

MARCH and November 1965, *JOCCA*, Nos. 3 and 11, Volume 48, and March 1966, No. 3, Volume 49, urgently required. Enquiries please to Assistant Secretary, Wax Chandlers' Hall, Gresham Street, London, E.C.2. Telephone No.: MON 1439.



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