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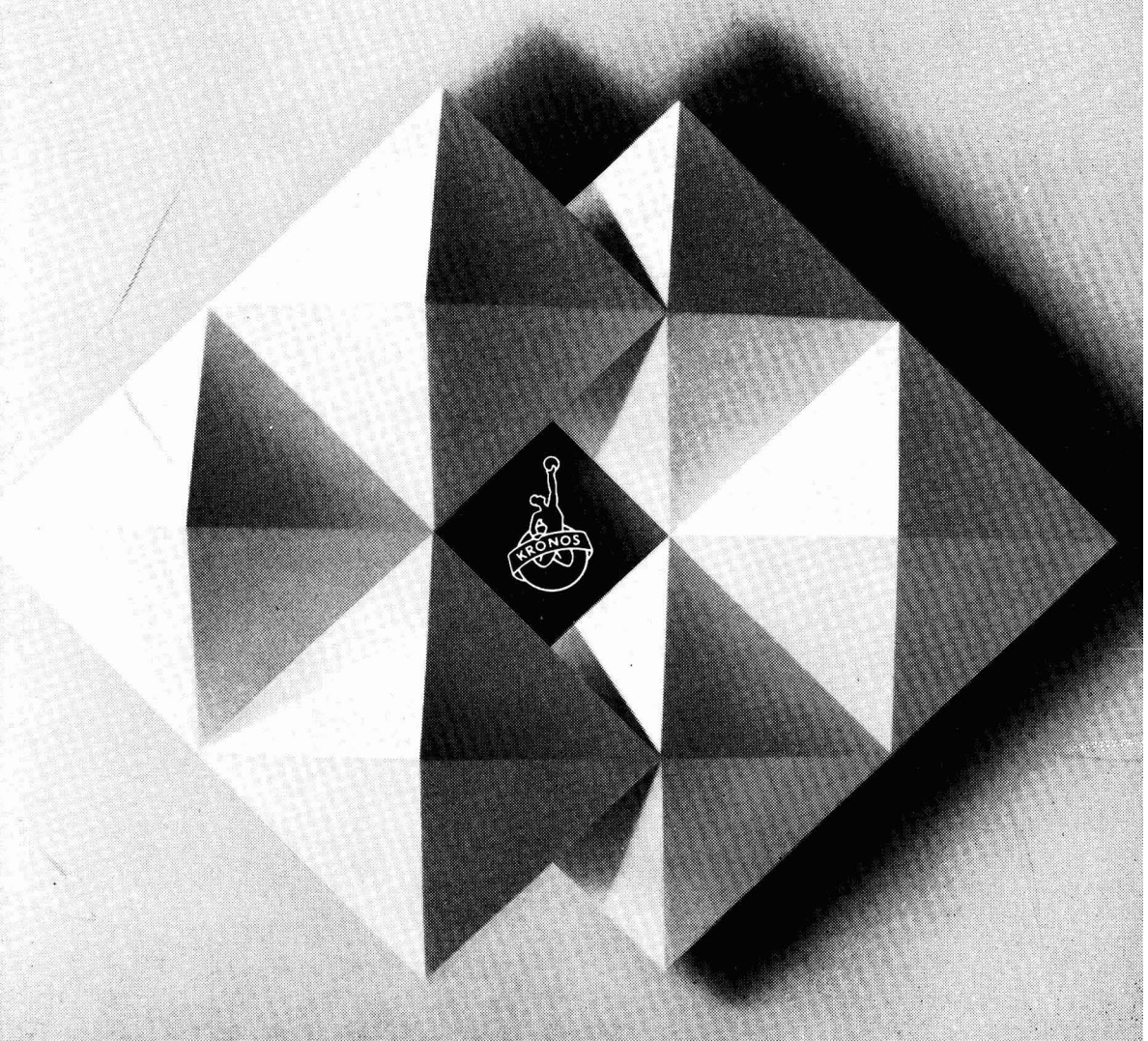

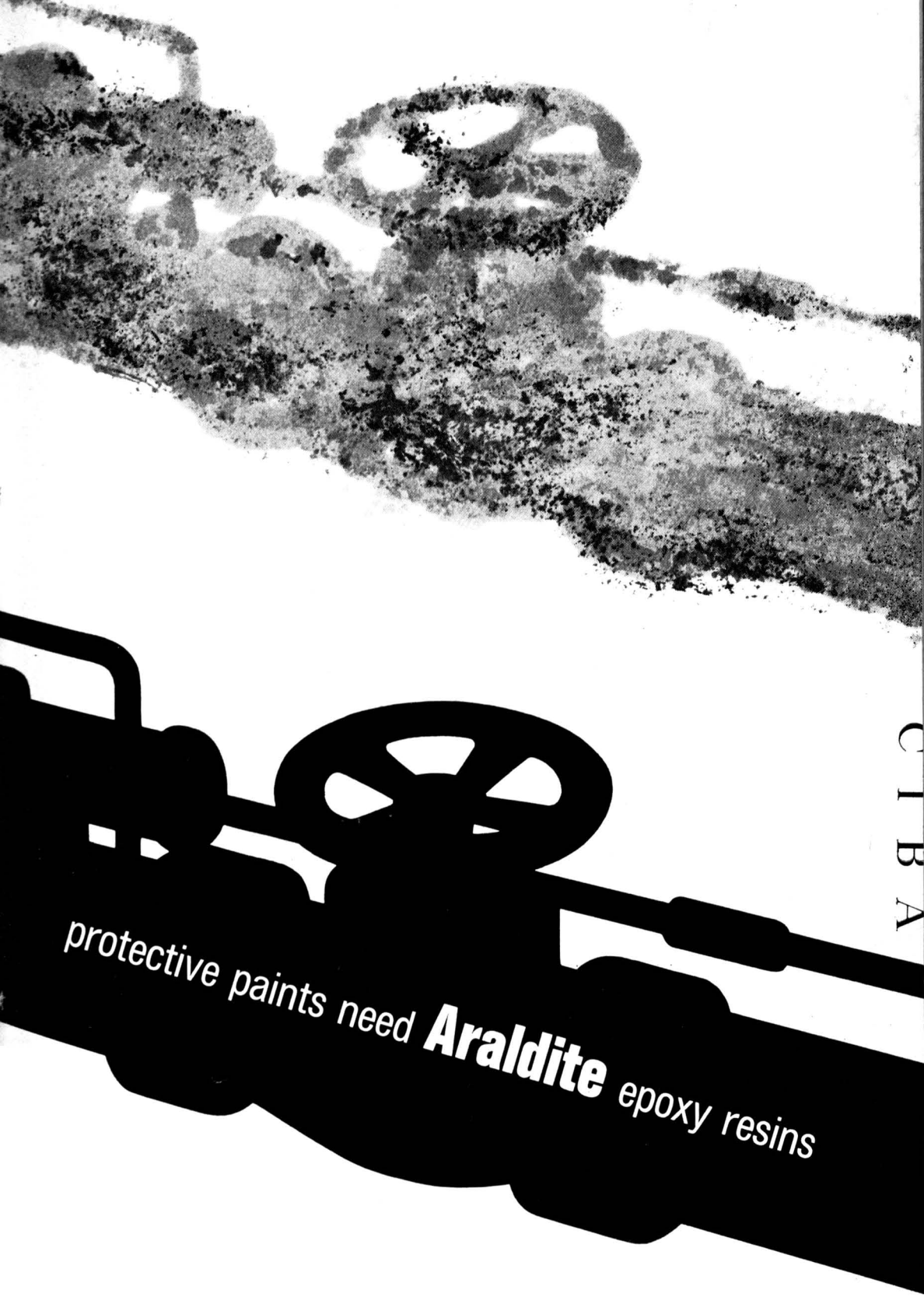

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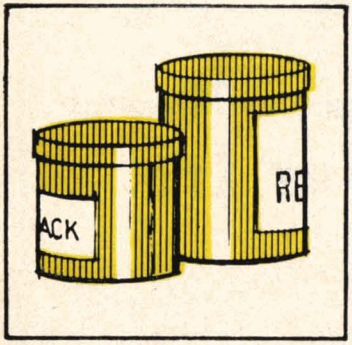

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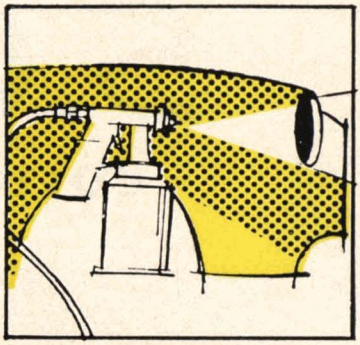

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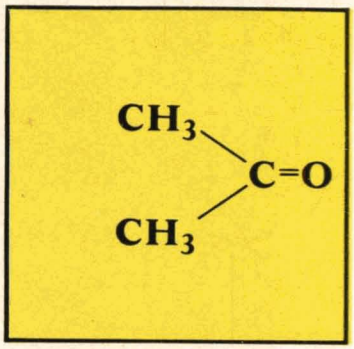

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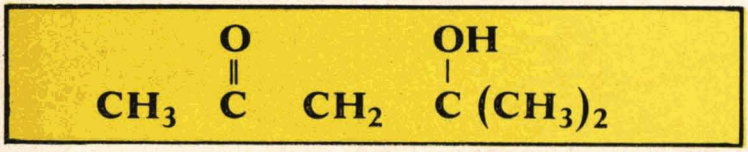

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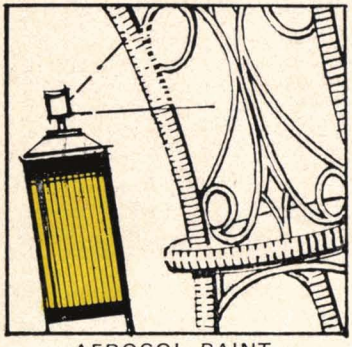

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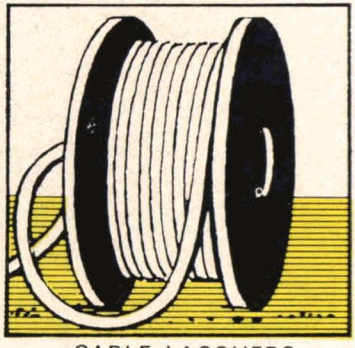

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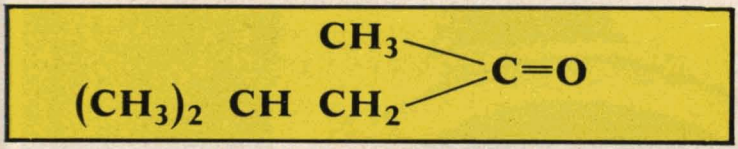

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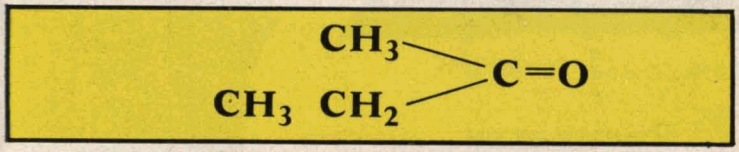

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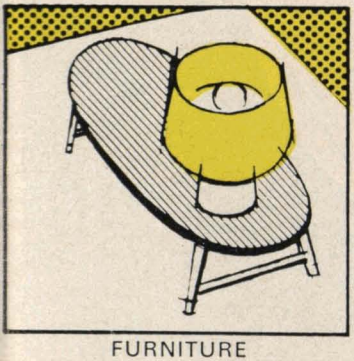

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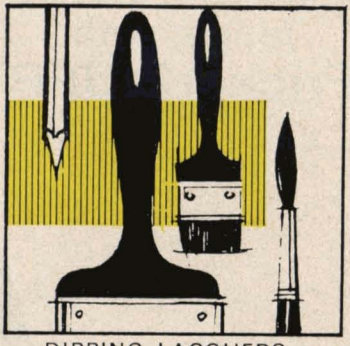

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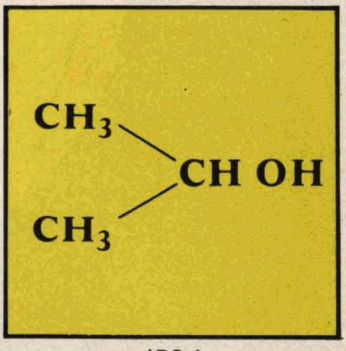

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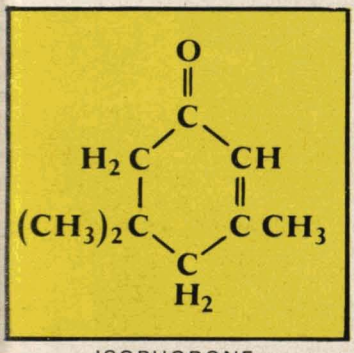

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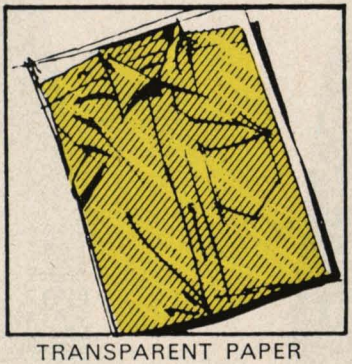

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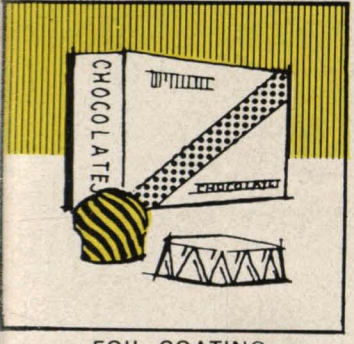

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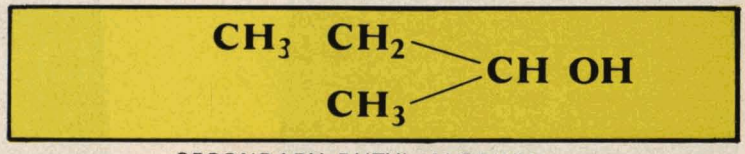

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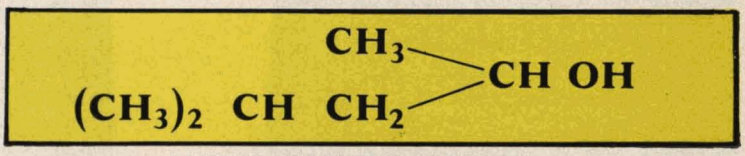

METHYL ISOBUTYL CARBINOL

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Wax Chandlers' Hall, Gresham Street, London E.C.2

Vinyl esters of synthetic branched chain fatty acids in new paint latices

By A. McIntosh and C. E. L. Reader*

"Shell" Research Ltd., Egham Industrial Chemicals Laboratory, Egham, Surrey

Summary

The performance of copolymers of vinyl acetate and the vinyl ester of synthetic branched chain fatty acids as emulsion paint media, has been examined in comparison with established polymers. From theory a 50/50 composition might be predicted as optimum, but it is shown that copolymers containing only 30 per cent of the new monomer have excellent properties. Bulk properties of latices and paints, and film properties have been evaluated. The incorporation of the branched chain structure confers good stability to the latex at neutral or slightly alkaline pH, and films formed from paints based on these latices exhibit superior resistance to scrubbing, dilute acid and alkali and exterior weathering when compared with vinyl acetate copolymers based on other comonomers. The overall level of performance resembles that of an all acrylic latex.

Peintures aux nouveaux latices contenant esters vinyliques des acides gras synthétiques de chaînes ramifiées

Résumé

On a examiné le rendement des copolymères d'acétate de vinyle et de l'ester vinylique des acides gras synthétiques de chaînes ramifiées en tant que média des peintures-émulsions en comparaison des polymères couramment utilisés. Au point de vue théorique une composition optimum de moitié/moitié doit être prédire. Cependant, on démontre que les polymères qui ne contiennent que 30% de monomère nouveau possèdent des propriétés excellentes. On a evalué les propriétés en vrac des latices et des peintures, ainsi que celles du feuil. L'introduction de la structure de chaîne ramifiée rend au latex une bonne stabilité sous les conditions de pH neutre ou légèrement alcaline. Feuils des peintures à base de ces latices démontrent meilleurs résistances à frottement humide, aux acides et alcalis dilués, et aux intempéries. Le rendement global des nouveaux copolymères ressemblent à celui d'un latex à base entièrement acrylique.

Vinylester synthetischer, verzweigte Ketten enthaltender Fettsäuren in neuen Latexen für Anstrichzwecke

Zusammenfassung

Das Verhalten von Mischpolymeren aus Vinylazetat und den Vinylestern synthetischer, verzweigte Ketten enthaltender Fettsäuren wurde mit dem von gebräuchlichen Polymeren als Bindemittel für Dispersionsfarben verglichen. Auf Grund theoretischer Berechnung würde man als Optimum ein Mengenverhältnis von 50 : 50 gewärtigen, es wird jedoch dargelegt, dass Mischpolymere mit nur 30% des neuen Monomers ausgezeichnete Eigenschaften besitzen. Sowohl die Eigenschaften von Latexen und Lacken als solche, als auch die ihrer Filme wurden bewertet. In Verbindung mit neutraler oder leicht alkalischer pH verleiht der Einbau der verzweigten Kettenstruktur dem Latex gute Stabilität. Filme von auf anderen Co-Monomeren

* "Shell" Research N.V., Delft Plastics Laboratory, Delft, Netherlands

aufgebauten, Vinylazetatmischpolymere enthaltenden Anstrichmitteln sind denen auf obigen Latexen basierenden hinsichtlich Beständigkeit gegen Abscheuern, verdünnte Säuren und Alkalien, sowie bezüglich Wetterfestigkeit unterlegen. Im Grossen und Ganzen ähnelt ihr Verhalten dem eines reinen Acryllatexes.

Виниловые эфиры синтетических разветвленных цепных жирных кислот в новых красочных латексах.

Резюме

Рассматривается действие кополимеров винил ацетата и винилого эфира синтетических разветвленных цепных жирных кислот в качестве эмульсионных красочных сред по сравнению с установленными полимерами. Из теории можно предсказать что состав в пропорциях 50:50 является оптимумом, однако доказано что кополимеры содержащие толко 30% нового мономера дают отличные свойства. Оцениваются объемные свойства латексов и красок, также свойства пленок. Включение разветвленной цепной структуры придает хорошую устойчивость латексу при нейтральной или слегка щелочной pH, и пленки изготовленные из красок на базе этих латексов предъявляют высокое сопротивление трению, раздавленной кислоте и щелочи и атмосферному выветриванию по сравнению с винило–ацетатовыми кополимерами на основе других комономеров. Общий уровень их действия носит характер полного акрилового латекса.

Introduction

This paper describes an evaluation of a new monomer, the vinyl ester of a synthetic branched carboxylic acid. The monomer is particularly suitable for copolymerisation in emulsion with vinyl acetate to produce high quality latices for emulsion paints. In this paper it will be shown that the unique structure of the comonomer is responsible for many of the attractive properties of the latices and of emulsion paints formulated from them. Physical properties of these copolymers such as glass transition points and minimum film forming temperatures will be discussed. The bulk properties of latices and the formulation of paints will be examined and a comparison of the properties of these paints with those of similar paints based on established types of latices will be made.

Nature of comonomer

Recent papers have described the improvements obtainable in alkyd resins by the incorporation of a branched chain carboxylic acid structure[1, 2]. Particular features of these alkyd resins, it will be recalled, were their excellent out-door durability, especially colour and gloss retention, and their alkali resistance. It was felt that the introduction of the same structural group into polyvinyl acetate might improve on the general performance of currently available vinyl acetate paint latices.

This structure is most conveniently introduced by copolymerising the vinyl ester of the acid with vinyl acetate. The preparation route for the vinyl ester is shown schematically below.

$$R{-}CH{=}CH_2 \xrightarrow{CO,\ H_2O} R_1{-}\overset{\displaystyle R_2}{\underset{\displaystyle R_3}{\overset{|}{\underset{|}{C}}}}{-}COOH \xrightarrow{C_2H_2} R_1{-}\overset{\displaystyle R_2}{\underset{\displaystyle R_3}{\overset{|}{\underset{|}{C}}}}{-}COOCH{=}CH_2$$

The olefins are obtained from the cracking of petroleum-derived wax and the acids are produced from them by addition of carbon monoxide and water in the

presence of a strong acid catalyst, according to a modification of the reaction discovered by Dr. H. Koch of the Max-Planck Institut für Kohlenforschung in Mülheim. The acids produced in this reaction are mainly of a tertiary nature. The vinyl ester is prepared from the acids by reaction with acetylene. Although this process is widely applicable, the work described here has been carried out on copolymers containing the vinyl ester of carboxylic acids derived from a cut of olefins with eight to ten carbon atoms, the acids therefore having nine to eleven carbon atoms. These acids and their vinyl ester are marketed under the trade names of " Versatic " 911 and " VeoVa " 911 respectively. In this paper these trade names will be used for convenience. A very similar material is available in certain areas which is the vinyl ester of the carboxylic acid derived from propylene trimer. This acid is marketed under the trade name " Versatic " 10 and its vinyl ester as " VeoVa " 10. Extensive laboratory work has shown that the properties of latices based on the vinyl esters produced from these two slightly different materials are very nearly identical.

These monomers are well adapted to copolymerisation in emulsion with vinyl acetate to give latices suitable for use as binders for high quality emulsion paints. Homogeneous, random copolymers are formed from all proportions of vinyl acetate and comonomer. The various methods of latex manufacture, however, will not be discussed here.

Physical properties

Before choosing the most desirable ratios in which the two monomers should be copolymerised, it is necessary to consider certain physical properties of polymer latices which are related to film forming.

Several theories concerning the mechanism of film formation have been put forward but perhaps the most accepted is that of Brown[3]. The hardness of the polymer at the temperature of application is one of the major factors influencing film formation. In order that coalescence may take place, the polymer should be soft enough to deform under the influence of surface tension forces but it should not be so soft that under service conditions there is excessive dirt pick-up or tackiness. In fact it is preferable that the polymer should have a glass transition temperature (T_g) about or slightly above room temperature.

The T_g of unmodified vinyl acetate homopolymer is rather too high to enable it to be used as a latex paint binder. The T_g may be lowered by externally plasticising the polymer, with dibutyl phthalate for example, but a better method is to copolymerise vinyl acetate with a " soft " comonomer.

It has been found that for many pairs of monomers a roughly linear relationship exists between T_g and copolymer composition by weight. This is the case, for example, with copolymers of vinyl acetate and " VeoVa " 911. It is also the case with copolymers of vinyl acetate and 2-ethylhexyl acrylate. The relation between T_g and polymer composition for these two pairs of monomers is shown in the diagram below.

It is evident that more " VeoVa " 911 than 2-ethylhexyl acrylate is required to effect the same reduction in T_g. (T_g has been taken here to be the point of inflexion on the graph of G-modulus, measured by means of a torsional pendulum with period 0.1 second, against temperature). However, it must be remembered

that T_g is a property of the polymer and for emulsion paints not only the polymer but also the latex, i.e. the polymer together with its environment of water, surfactants etc., must be considered. More important than the actual glass transition temperature is the minimum temperature at which the polymer particles in the latex will coalesce to form a continuous crack-free film.

The relationship between minimum film forming temperature (mft) and polymer composition is not linear like the T_g/composition relationship. A graph of mft and copolymer composition for latices based on vinyl acetate and " VeoVa " 911 is shown below.

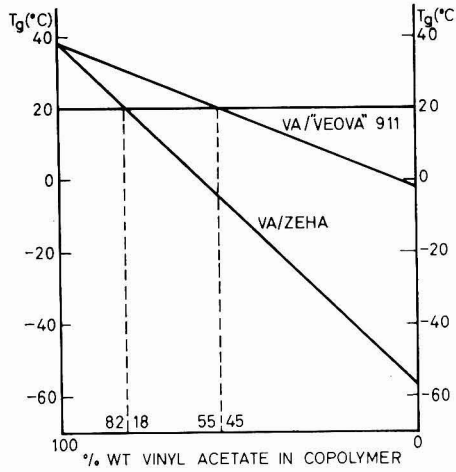

Fig. 1. Relations between T_g and polymer composition for VA/2-EHA and VA/"VeoVa" 911

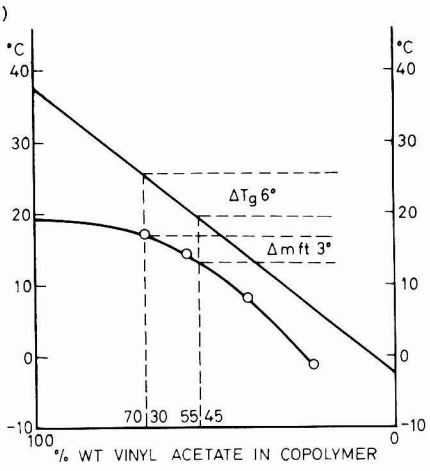

Fig. 2. Relationship between T_g and minimum temperature of film formation (mft) for VA/ "VeoVa " 911 copolymers

It can be seen that, within the range of interest for paint latices, the difference between T_g and mft increases with the vinyl acetate content. This may be explained as follows. The " hard " component of the copolymer, vinyl acetate, is a more polar entity than the tertiary ester comonomer and the polarity of the copolymer therefore increases with vinyl acetate content. It is thus more softened by the environmental water, and film formation can take place at a lower temperature than might be apparent from the position of the polymer's glass transition point.

From the graph of T_g and composition one might predict than an optimum copolymer composition should be roughly half vinyl acetate and half " VeoVa " 911. Furthermore a composition containing 70 per cent vinyl acetate might be considered unsuitable. However, whereas an increase of from 55 per cent to 70 per cent in vinyl acetate content causes an increase in T_g of 6° the increase in mft is only 3°. Small increases in mft can be easily compensated by the use of additives.

There is of course a considerable economic incentive to employ lower comonomer levels. Nevertheless, the comonomer content should not be reduced to such a low level that the desirable properties it may impart to the polymer as a

whole are lost. This can be the case with some comonomers which are only present in small amounts.

Emulsion paint is used almost exclusively in the decorative market and may be applied by technically untrained personnel or " do-it-yourself " painters who do not appreciate the fact that below a certain temperature a good film will not form. A method of preventing failure caused by application at too low a temperature is to base the paint on a soft latex with a low mft ; many such latices are available but, in addition to being comparatively expensive, the paints based on them often show unacceptably high dirt pick-up in service. Such paints can be formulated to chalk and thereby retain a clean appearance but this is also unsatisfactory, especially for coloured paints. An alternative method, and one which shows very good results in practice, is to use a harder latex and to depress the mft by means of a small amount of a coalescence agent. A coalescence agent temporarily softens the polymer particles thereby enabling film formation at lower temperatures than the mft of the latex. After application this film-forming aid disappears from the film, the rate of its disappearance depending on its volatility and chemical nature. Addition of coalescence agents is, of course, common in the formulation of emulsion paints ; their use not only reduces the mft of the paint but also improves general film properties.

Compared with many currently available paint latices the mft of a latex based on a copolymer of 70 per cent vinyl acetate and 30 per cent " VeoVa " 911 is high. Nevertheless the mft of paints based on such a latex can be reduced sufficiently by small amounts of coalescence agents. The choice of these materials is discussed later.

Bulk properties of latices

Apart from the performance of the end product, the actual emulsion paint, consideration must be given to certain important bulk properties of latices. Latices should have good storage stability within a reasonable temperature range since quite lengthy periods of time may elapse between the manufacture of the latex and its incorporation into a paint. A latex may also freeze during storage and the ability to recover from this change of state is desirable. Mechanical agitation occasioned during transport of the latex or in the manufacture of paint should not cause any coagulation or greatly alter the viscosity of the system. The ability to tolerate addition of electrolytes is also a necessary property since ionic materials are often introduced during pigmentation or processing.

Like most products currently marketed, latices containing the branched acid structure also have good mechanical, electrolyte and storage stability. An advantage offered by the incorporation of this structure is the good pH stability at neutral or slightly alkaline pH which enables resistance to repetitive cycles of freezing and thawing to be obtained.

Latices may be stabilised by the presence of protective colloid or surfactants, or both. Protective colloids, such as polyvinyl alcohol, or cellulose derivates, like hydroxyethyl cellulose, act by enveloping the polymer particles thereby preventing their coalescence under storage conditions. This type of protection, however, is often insufficient to prevent coagulation during freezing and thawing. An anionic surfactant functions by imparting a negative charge to

the polymer particles which are then prevented from coalescing by electrical forces of repulsion. This type of protection also often fails to provide sufficient stability to withstand freezing and thawing. In this latter case it seems that the effectiveness of the anionic surfactant decreases at low temperatures possibly due to alterations in solubility thereby removing or diminishing the charge on the particles. An anionic surfactant fixed to the polymer surface, i.e. by copolymerisation, would be less affected by reduction of temperature and freeze-thaw stability can be imparted to certain types of latices, notably " all-acrylic " latices in this way. This method is also applicable to latices containing " VeoVa " 911. If a small quantity of acrylic acid is copolymerised with the vinyl esters, a

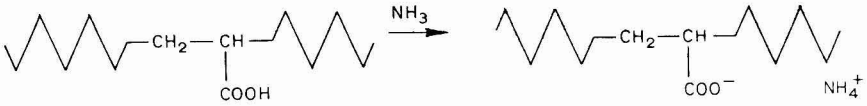

Fig. 3. Idealised diagram of the effect of incorporating acrylic acid

latex of idealised structure as shown in the diagram is obtained. One per cent of acrylic acid calculated on monomers is about optimum but care must be taken to ensure that it is suitably incorporated into the polymer, ideally at regular intervals along the polymer chain. Methods are available which enable this to be done. After manufacture the pH of the latex must be raised to 7-7½ to ensure sufficient dissociation of the carboxyl groups. As long as the latex remains at approximately neutral or alkaline pH it will be freeze-thaw stable.

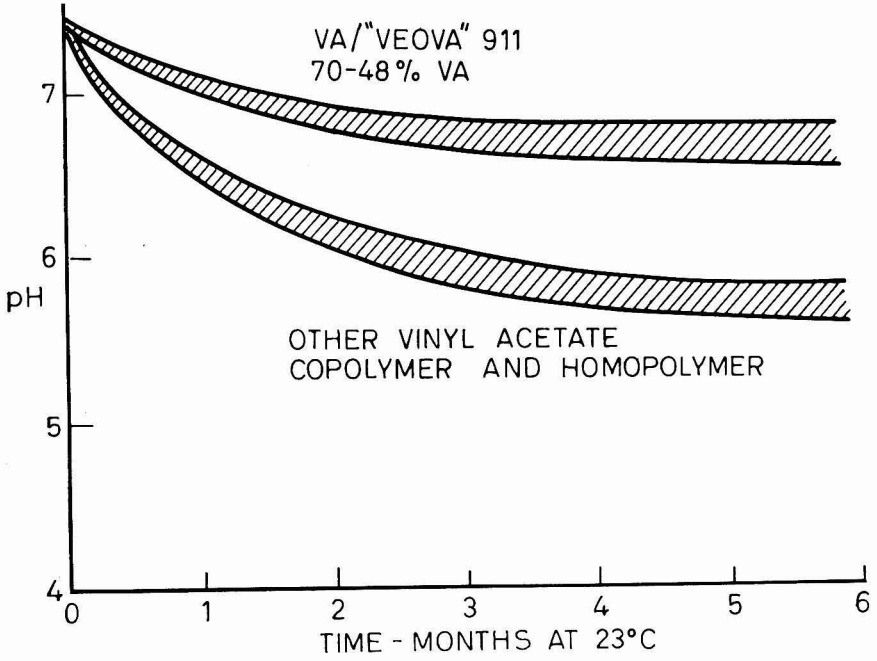

Fig. 4. Change of pH with time—vinyl acetate copolymers

This method can also be applied to conventional vinyl acetate based latices but, as shown in Fig. 4, these latices exhibit a poorer pH stability than the " VeoVa " 911 containing copolymers and hence the freeze-thaw stability can be lost.

Some properties of two typical latices containing " VeoVa " 911 are given in Table 1.

Table 1

Properties of two typical latices containing VV911 (VeoVa 911)

Percentage composition		
vinyl acetate	52½	69
" VeoVa " 911	46½	30
acrylic acid	1	1
Properties		
solids content, % wt	53.4	49.5
specific gravity, latex	1.05	1.06
specific gravity, solids	1.10	1.13
average particle size, μ	0.27	0.20
viscosity, Brookfield, 60 r.p.m., cps	82	82
odour	slight ester	slight ester
min. film forming temp., C	13	17
mechanical stability	good	good
ion stability	good	good
freeze-thaw stability	good	good
settling or syneresis after one year	none	none

Clear-film properties of latices

The properties of unpigmented latex films were discussed, inter alia, in some detail in a recent paper[4]. Mechanical properties, such as stress-strain behaviour and hardness, and chemical properties, such as UV stability, water and alkali sensitivity were considered and it is not intended to discuss these properties again here.

For copolymers of vinyl acetate with plasticising comonomers the hardness of the polymer obviously increases with vinyl acetate content. This is shown in Table 2 from which it is evident that a copolymer of 70 per cent vinyl acetate with 30 per cent " VeoVa " 911 has roughly the same hardness as a copolymer

Table 2

Pendulum hardness of clear latex films (Koenig, seconds)

Ratio of VA to comonomer	comonomer	
	VV911	2-EHA
85-15	—	52
80-20	90	35
75-25	—	18
70-30	50	—
54-46	23	—
40-60	8	—

of 85 per cent vinyl acetate with 15 per cent 2-ethylhexyl acrylate. Free films of these latices exhibit similar stress strain behaviour though the minimum film forming temperature of the former is somewhat higher due probably to its less polar nature.

Latex paint evaluation programme

In an extensive evaluation of emulsion paints based on different types of latices, two copolymers containing the branched acid structure have been compared with six commercially available products representative of the major chemical types of paint latices currently marketed.

The two copolymers containing the branched acid structure were of the following percentage compositions by weight :

vinyl acetate	52½	69
" VeoVa " 911	46½	30
acrylic acid	1	1

The bulk properties of these latices were given in Table 1. It should be pointed out here that the levels of comonomer in these two polymers do not represent the full useful range of polymer composition. From latex copolymers containing 20 per cent of " VeoVa " 911 emulsion paints can be formulated which are at least equivalent to similar paints based upon established types of vinyl acetate copolymers of acknowledged quality.

The commercially available products included in the evaluation as standards represent a fair cross section of the major types of paint latices at present marketed. They are listed in Table 3.

Table 3

Latices included in the paint evaluation programme

Latex	VA-VV911 (52½% VA)	VA-VV911 (69% VA)	VA-DBM	VA-DOM	VA-2EHA	VA-homo (10% plast.)	Acrylic	S-B
Particle size, μ. (average)	0.27	0.20	1	—	—	0.4-1	—	0.20
Solids content, %	53.4	49.5	56.0	49.8	55.9	56.8	44.8	49.0
pH as received	7.0	7.3	5.1	5.4	4.3	4.5	9.1	10.8
Viscosity, cps (Brookfield, 60 rpm)	82	82	214	250	1620	1300	390	31

Paints based on these latices and varying in pigment volume concentration (PVC) from 30 per cent to 70 per cent have been tested in the laboratory but attention here will be confined to paints of 40 per cent and 60 per cent PVC. Forty per cent may be regarded as a typical PVC for general purpose emulsion paints for both indoor and outdoor application ; 60 per cent is a suitable PVC for good quality interior emulsion paints.

Formulation of paints

A considerable amount of attention has been given to the development of formulations in which the different types of latices can be fairly evaluated and

compared. As emulsion paints generally contain a dozen or more ingredients this is not easy ; a good formulation for one latex may not be suitable for use with another. For example, a coalescence agent which is recommended for one type of latex may have less effect on a latex of a different chemical type.

In a comparative evaluation certain questions inevitably pose themselves : should each latex be tested in its optimum formulation, or in the same formulation, or in formulations at price parity, or what criteria should be adopted ? Perhaps a comparison in optimum formulations would be best but these are probably not known and, if they were, they would vary depending on the envisaged end-use and might well be unacceptable commercially. On the other hand, evaluation of each latex in precisely the same formulation also does not afford a wholly fair basis for comparison. The authors consider that a good comparative evaluation of a number of latices can be made by making the minimum number of alterations to one basic formulation which should be fair for all the products under test. The paint formulations discussed here have been developed with this philosophy in mind.

In a given emulsion paint formulation there are four basic variables : pigment volume concentration, non-volatile content, hiding pigment loading and viscosity. Unfortunately it is extremely difficult or impossible to keep all these four quantities constant in a series of paints based on different polymers. An attempt has been made to approach this ideal as closely as possible in the following way.

At both pigment volume concentrations discussed here, 40 per cent and 60 per cent, one large batch of pigment paste was mixed and dispersed. To portions of the pigment paste the precise quantities of latices required for the chosen PVC were added and the coalescence agent and second increment of preservative then stirred in very carefully. The semi-finished paints were next diluted to equal volumes with water and thickener solution. The ratio of water to thickener solution was chosen so that the finished paints should have the same initial viscosity. After preparation, adjustments of pH were made if necessary, with ammonia, to give pH of 7-7½ for vinyl acetate based paints and 9-9½ for " all acrylic " or styrene-butadiene based paints. Thus all the paints prepared had the same pigment volume concentration, hiding pigment loading and initial viscosity. They differed slightly in solids content by weight. The blue tints were prepared by adding 2 per cent by weight of a pigment dispersion, " Monastral " Blue BNVS, to the white paints.

The 60 per cent PVC formulations were similar in many respects to the 40 per cent PVC ones. A lower level of titanium dioxide was chosen because, at these higher pigment loadings, there is an increased contribution to opacity from " dry-hiding." The 60 per cent PVC paints were also formulated at higher solids contents.

Although the precise reasons for choosing each individual ingredient will not be discussed, some attention must be paid to coalescence agents, the main point of difference between the various formulations.

For the commercially available latices the choice of coalescence agent was guided mainly by the recommendations in the manufacturers' literature. Thus ethylene glycol monobutyl ether acetate was chosen for use with the two

maleate copolymers, the 2-ethylhexyl acrylate copolymer and the externally plasticised homopolymer. No active coalescence agent was used with the all-acrylic latex. Hexylene glycol was used with the styrene-butadiene latex, as it has been found to have a beneficial effect on the film properties of paint based on such latices[5].

Coalescence agents for use with latices containing " VeoVa " 911

To find the optimum coalescence agents for use with latices containing the branched acid structure, a number of experiments were carried out to determine the effect of various solvents on the minimum film forming temperature, storage and freeze-thaw stability, flow and application properties, water spotting, scrubbability and cleansability (stain removal) of paints. Several points emerged from this investigation.

On its own, ethylene glycol is a poor coalescence agent for this type of latex but its presence is necessary to restore the freeze-thaw stability that is generally lost when a more effective coalescence agent is included in the formulation. Ethylene glycol is also stated to improve the wet-edge properties of paints and was included in all formulations. Acetates such as the acetates of the monobutyl ethers of ethylene and diethylene glycols are very efficient coalescence agents but tend to reduce pH stability. This may not be particularly important in some systems but, as reduction in pH can jeopardise freeze-thaw stability when this has been obtained by the technique described earlier, this type of product has not been used with the " VeoVa " 911 copolymer latices. The commercially available vinyl acetate latices did not have good freeze-thaw stability and so this objection was less applicable.

In addition to the obvious effect on low temperature film formation, the presence of coalescence agents improved scrubbability, cleansability (stain removal) and water spot resistance. Some results of this work are given in Table 4.

Table 4

Choice of coalescence agent for VA-VeoVa 911 (52½% VA) paints of 40% PVC

coalescence agent	depression of mft	freeze-thaw stability	ΔpH (after four weeks 50°)	water-spot (15 minutes)*
none	—	good	0.9	4
ethylene glycol (EG)	poor	good	0.8	6
hexylene glycol (HG)	fairly good	poor	0.8	8
EG/HG 1/1	fairly good	good	0.9	9
ethylene glycol monobutyl ether (BO)	good	poor	0.8	8
ethylene glycol monobutyl ether acetate (BOA)	good	poor	1.3	8
diethylene glycol monobutyl ether acetate	good	poor	1.5	6
BO/EG 1/1	good	good	0.7	8
BOA/EG 1/1	good	good	1.1	8

* Rating 10—Unaffected

From this investigation ethylene glycol monobutyl ether 2 per cent on paint weight was chosen for use with the latex containing 46 per cent " VeoVa " 911.

With the latex containing 30 per cent " VeoVa " 911, 2 per cent of ethylene glycol monobutyl ether lowers the mft of a 40 per cent PVC paint to about 11° or 12° and not to 5° which is often considered to be a desirable level. But to reduce the number of variables to a minimum, ethylene glycol monobutyl ether was nevertheless used with the higher vinyl acetate content latex. Improved performance is of course obtained by the use of more efficient coalescence agents and for this reason it is appropriate to mention some of them here.

At the 2 per cent level, based on total weight of paint, many of the commonly employed coalescence agents such as the glycol ethers and hexylene glycol are not particularly effective. Diethylene glycol monobutyl ether acetate is a good coalescence agent but, as mentioned before, tends to reduce pH stability. Benzyl alcohol, tributyl phosphate, diethylene glycol dibutyl ether, 2-ethylhexanediol-1,3 and the proprietary products " Dalpad " and " Texanol " are examples of suitable coalescence agents for use with lower " VeoVa " 911 content copolymers. In the formulations examined, between 1 per cent and 2 per cent of these products will reduce the mft of 40 per cent PVC paints to 5°C or less without adverse effects on storage stability. Film properties such as scrubbability increase considerably when good coalescence agents are used. As with the lower vinyl acetate content copolymers the incorporation of ethylene glycol is also recommended.

Bulk paint properties

The experimental paints were subjected to a series of tests designed to evaluate their storage properties in terms of pH, viscosity and freeze-thaw stability. To this end, samples were stored for one year and their properties were determined at appropriate intervals. The freeze-thaw stability test, carried out on 350 ml samples, consisted of five cycles, each of 24 hours freezing at −20° followed by 24 hours recovery at 23°.

The diagrams (Figs. 5 and 6) shows the results obtained for pH and viscosity

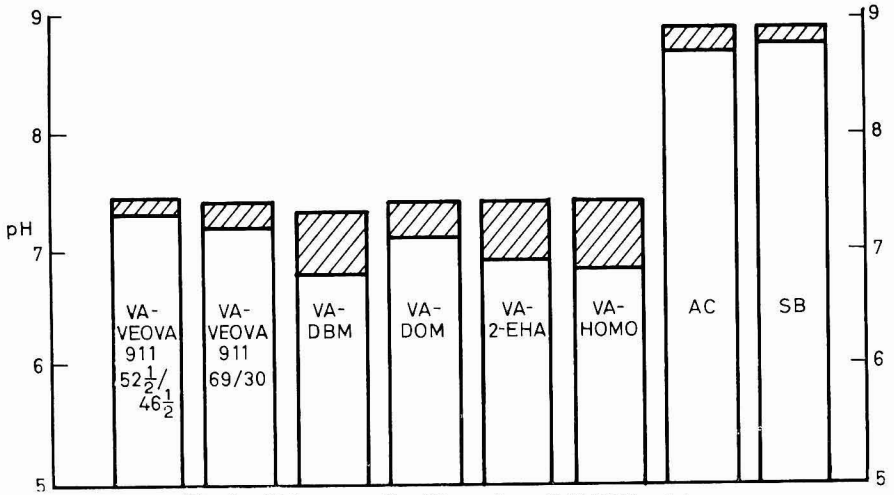

Fig. 5. pH decrease after 12 months—40% PVC paints

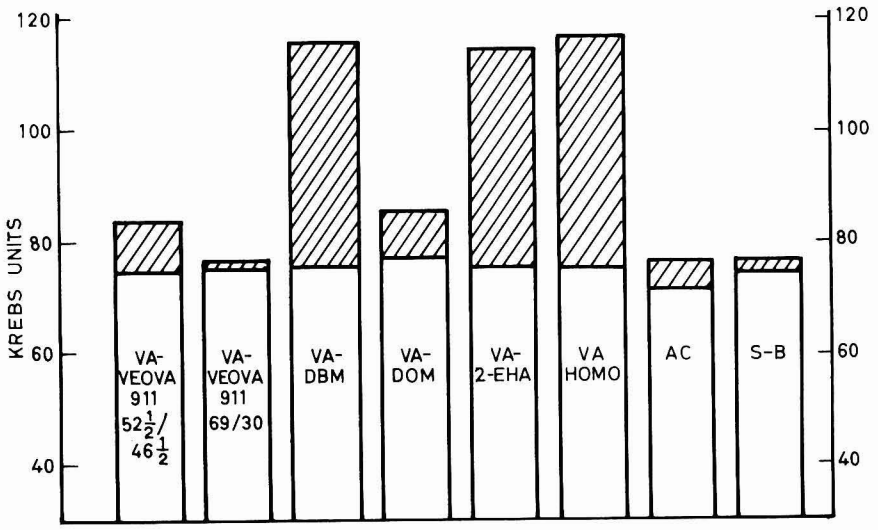

Fig. 6. Viscosity increase after 12 months—40% PVC paints

stability for the 40 per cent PVC paints. The 60 per cent PVC series shows a generally similar pattern of performance. Compared with the paints containing the branched acid structure most other vinyl acetate based paints showed poorer storage stability. The two " VeoVa " copolymer latices, the acrylic and the styrene butadiene latices all gave paints of excellent freeze-thaw stability ; most paints based on the other vinyl acetate latices gelled after one cycle.

Film properties of paints

It is always difficult to select criteria for assessing paint performance which will satisfy all possible requirements without involving an inordinate volume of laboratory work. It is felt, however, that the tests reported in the following sections of this paper are sufficiently broad in character to be able to claim that the results are meaningful.

Hardness : This property is mainly of interest in the context that too soft a film will lead to excessive dirt retention on exposure. In the systems under review the differences in König hardness of the 40 per cent PVC paints were not very large. The acrylic and styrene-butadiene based paints had the lowest film hardness of 17 seconds. The hardest films were those of the two " VeoVa " based paints. Of these, that based on the copolymer containing only 30 per cent comonomer was noticeably harder. This is evident in the early stages of exposure testing, when the initial dirt retention of the harder polymer is appreciably less. It is often held that too hard a polymer will lead to cracking on substrates such as wood which tend to " move ". This has not been found in practice with the harder copolymers ; this is referred to in the section on exposure testing.

Scrubbability : The original tests were performed on sand-blasted glass panels but results on this substrate showed very poor reproducibility and therefore the method was modified. A film of blue-tinted alkyd paint was applied to

the sand-blasted glass and dried for four days. This film was then lightly sanded before applying one coat of the latex paint by doctor blade. After seven days drying at 23°, the scrubbabilities were measured using an R.E.L. Scrub Tester. The weight on the nylon brushes was kept constant at 500 g throughout the tests and the paint films were continuously wetted with 0.5 per cent " Teepol " solution. The results are shown in Fig. 7.

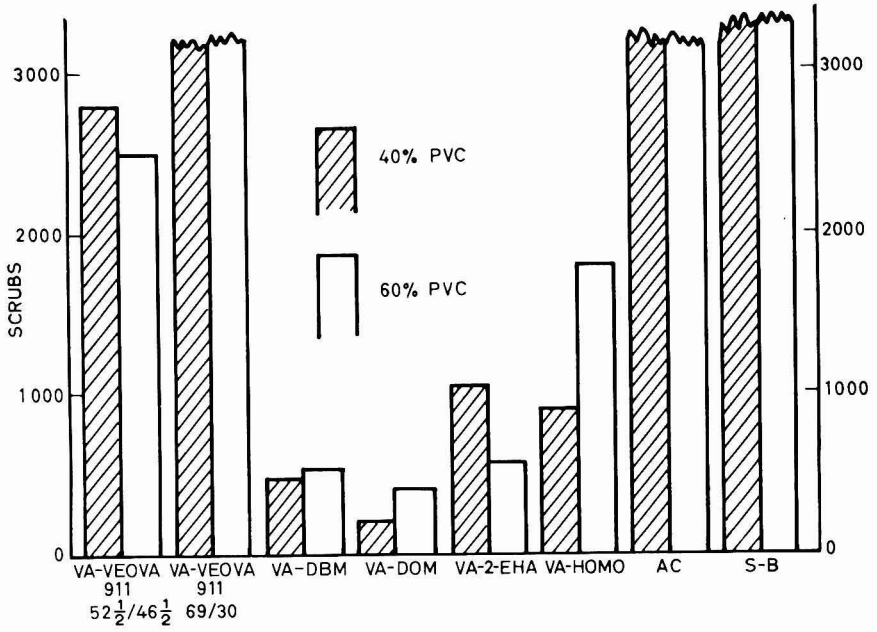

Fig. 7. Scrubbability of white 40% and 60% PVC paints

It is evident that the scrubbabilities of both the " VeoVa " paints, the acrylic, and the styrene-butadiene paints are markedly superior to all the commercial vinyl acetate based paints examined. The scrubbability of the " VeoVa " based paints improves with increasing hardness of polymer within the range investigated. In other tests we have found that scrubbability performance is very related to the degree of completeness of coalescence and for optimum results the most effective coalescence agents must be used.

Cleansability : This is in some respects another aspect of scrubbability, but instead of measuring the deterioration of the film, the ease (or otherwise) of removal of a variety of stains is determined. The same apparatus is employed with the nylon brushes replaced by polyurethane foam sponges. A proprietary brand of soap was used as the cleaning medium and the sponges were soaped after each 25 cycles of cleaning. The staining media (writing ink, HB pencil, red wax crayon, black-currant juice and carbon paper) were applied in parallel stripes approximately 5 mm apart at right-angles to the path traversed by the sponge. One hour was allowed before testing. The figures in Table 5 are the average of two determinations, five representing the maximum possible and

indicating that all five different staining media were removed in 100 cycles of cleaning. It can be seen that most of the paints showed very good cleansability.

Table 5

Cleansability of paints of 40% and 60% PVC (max 5)

Base latex	40% PVC	60% PVC
VA-VeoVa911 52½-46½	4½	5
VA-VeoVa911 69-30	5	4¼
VA-DBM	3	4½
VA-DOM	4¾	4¾
VA-2EHA	5	5
VA homopolymer	4¾	4¾
Acrylic	4½	4¾
Styrene-butadiene	3¼	4

Water spot, water soak and chemical resistance : A variety of tests were carried out to evaluate the behaviour of these latex paints on exposure to water or aqueous environments.

In water spotting tests no significant differences in performance among the latices were found.

In resistance to dilute acetic acid and caustic soda the acrylic acid and high " VeoVa " 911 content latices were best overall, followed by the styrene-butadiene and lower " VeoVa " 911 contents latices. The other vinyl acetate polymers were all markedly poorer in these tests.

The results for coatings on concrete blocks partially immersed in water for three months are shown in Table 6. This is in effect an alkali-resistance test since the pH of the water may rise to 10 or 11 under the conditions of immersion. The values shown were obtained by numerically rating, from 10 (unaffected) to nil (complete failure), softening and loss of binding power above the water line, and blistering and yellowing below the water line. Where loss of binding power was total and the film disintegrated, obviously an overall rating of nil was justified. These individual numerical ratings have been averaged to give the final figures.

Table 6

Alkaline water soak—white paints—three months

Base latex	40% PVC		60% PVC	
	condition above waterline	condition below waterline	condition above waterline	condition below waterline
VA-VeoVa911 52½-46½	5	8	10	9
VA-VeoVa911 69-30	8	8	10	8
VA-DBM	0	6	10	7
VA-DOM	0	6	10	8
VA-2EHA	6	5	10	8
VA homopolymer	0	4	10	7
Acrylic	4	8	8	7
Styrene-butadiene	8	9	10	9

10—perfect 0—complete disintegration

The behaviour of the blue paints closely paralleled that of the white paints, except that in general the failures were rather more emphasised with the blue paints. This is most probably due to the extra surfactant introduced in the blue tinter. Differences in performance are less noticeable at 60 per cent PVC since the films are much more porous thus permitting easier transmission of water vapour. At the lower PVC the alkali resistance of the polymer is playing a greater role and here the good alkali resistance of the branched chain ester can be detected.

Exposure testing

The importance of exterior durability of emulsion paints is increasing substantially. Large quantities are already applied on exterior surfaces in the United States while the trend in Europe is also towards greater exterior usage. Recognising this, a great deal of importance has been placed on exterior durability. At our exposure station in The Netherlands nearly two thousand panels coated with various emulsion paints are on test. Particular attention has been paid to the performance on alkaline substrates such as concrete and asbestos cement. These are materials that are being increasingly used in the building industry and they are also most liable to require decoration. Wood is also used as a construction material in many countries and oil-primed and unprimed pine have therefore been included as substrates in our tests.

To obtain a preliminary estimate of relative performance "Weatherometer" tests were carried out. A prime coat (one volume paint plus half volume water) and two full coats were brushed on asbestos cement panels and subjected to 1,500 hours wet-and-dry exposure. The cycles consisted of 102 minutes ultraviolet light exposure followed by 18 minutes UV plus demineralised water spray directed at the panels. Some results are shown in Fig. 8. The superior performance of the "VeoVa" containing polymers is clearly demonstrated although too much reliance should not be placed on artificial weathering results since the relative emphasis on various types of film failure differs from that found in natural weathering. The results however, are indicative of the general level of UV resistance.

For the purpose of this paper the exterior weathering results have been confined to 40 per cent PVC paints since, as mentioned previously, this is a typical PVC for outdoor/indoor paints. A large number of results have been obtained in exterior durability tests of two years duration on oil primed and unprimed Oregon pine, air-dried asbestos cement and concrete blocks. In all cases except oil-primed wood, one dilute coat followed by two full coats of paint were applied by brush. On primed wood the diluted coat was omitted. All the panels quoted have been mounted vertically facing south as it was considered that this is more representative of practical conditions than 45° exposure. It is obviously not possible to present all the data from such series in a tabular form which is easily assimilable; representative results have therefore been selected to illustrate the performance of the paints. Exposures at 45° (not reported here) have been carried out as well and the results in general follow the same pattern as for the vertical exposures. The failures are, as expected, slightly more severe.

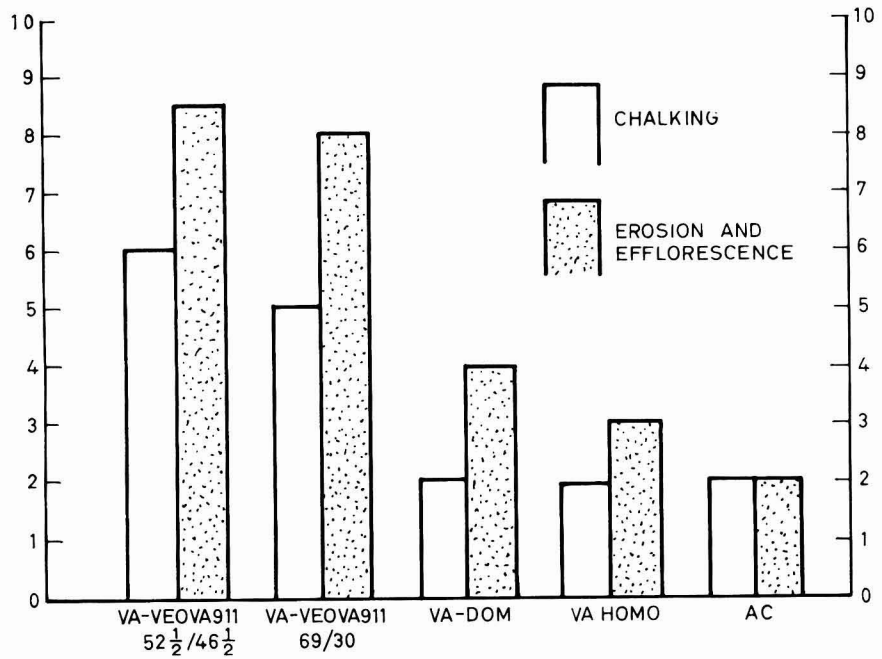

Fig. 8. Condition of blue 40% paints after 1,500 hours " weatherometer " exposure

The following observations may be made on these results :

Wood : On primed wood most of the paints are still in good condition although the styrene-butadiene based paint is chalking very heavily and has cracked. The higher rates of chalking of the homopolymer and acrylic paints are more noticeable in the blue series.

Most of the unprimed wooden panels (Fig. 9) show cracking of the paint film which is most evident with the homopolymer paint. The interesting feature apparent from these results is that the paints based on the harder experimental latex containing only 30 per cent branched chain ester are not showing signs of great failure. This result is in accordance with those obtained by Oosterhof who showed that neither tensile strength nor hardness of the films seems to have a direct influence on exterior durability[4].

Concrete : On matured concrete many of the paints based on the conventional vinyl acetate latices especially the externally plasticised homopolymer are showing erosion and efflorescence. The acrylic and styrene butadiene paints are in fairly good condition but chalking heavily; this is especially noticeable with the blue panels which have lost their colour strength. The paints containing the branched acid structure remain in excellent condition and are only chalking slightly.

Asbestos cement : On this substrate only the " VeoVa " 911 based paints show uniformly high performance (Fig. 10). Most of the paints based on con-

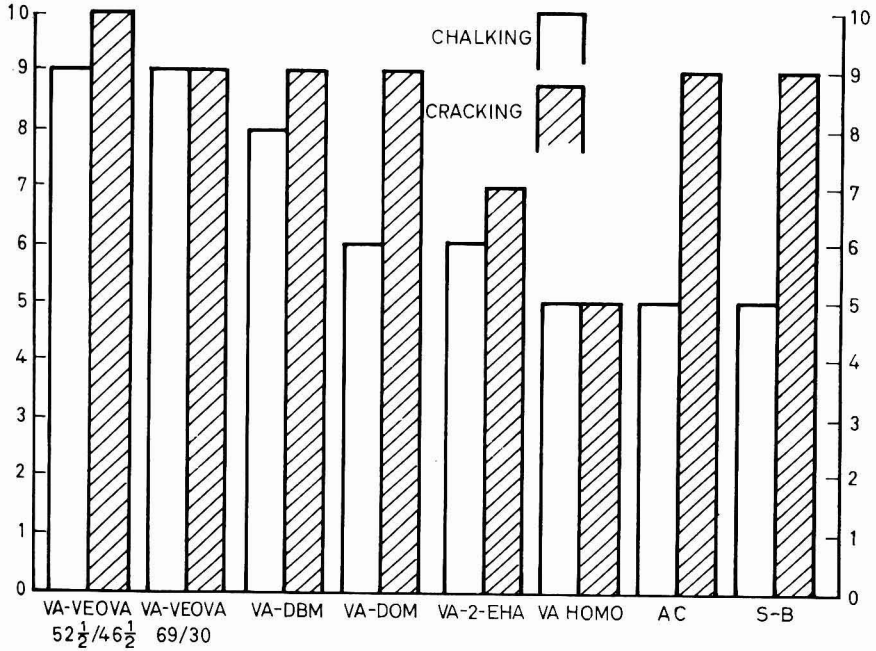

Fig. 9. Two years exterior exposure on unprimed wood—white 40% PVC paints

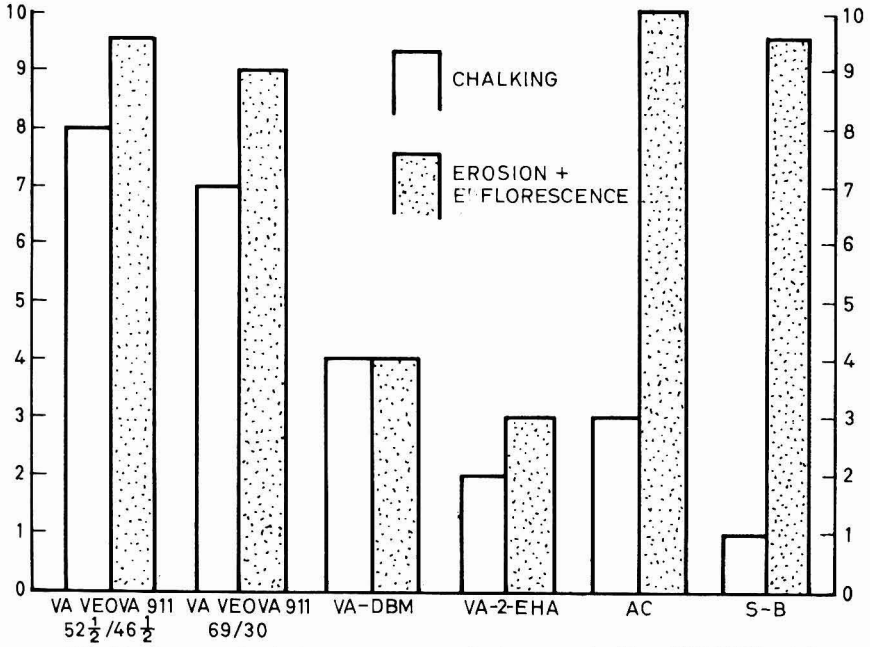

Fig. 10. Two years' exterior exposure on asbestos cement—blue 40% PVC paints

ventional vinyl acetate latices have failed very badly, often within less than a year. The acrylic and styrene-butadiene based paints chalk heavily and this is more noticeable with the blue paints which appear to have faded considerably ; the alkali resistance of these paints, however, is of a very high level.

Conclusions

From consideration of glass transition temperature it can be argued that for paint latices based on copolymers of " VeoVa " 911 and vinyl acetate the optimum polymer composition is roughly 50 per cent of each component. However, emulsion paints based on copolymers containing only 30 per cent " VeoVa " 911 have excellent properties for both outdoor and indoor application. The minimum film forming temperatures of such copolymers are high compared with those of the more established types of latices and small quantities of coalescence agents are required. Very little overall decrease in paint performance is found when the comonomer content of the polymer is decreased from 50 per cent to 30 per cent. Indeed some properties are improved by the use of a harder binder.

Due to the presence of the branched acid structure, latices have good stability at neutral or slightly alkaline pH. Freeze-thaw stability can be built into the polymer. Paints based on these latices also have excellent storage and freeze-thaw stabilities.

Particular advantages offered by the incorporation of the " VeoVa " structure in emulsion paint films include superior scrubbability, dilute acid and alkali resistance compared with conventional vinyl acetate type latices. The performance is comparable with that of all-acrylic latices.

In exterior weathering tests only the " VeoVa" based paints have shown a uniformly high standard of performance on all substrates. Their rates of chalking are low and tint retention is therefore good. Their resistance to degradation on alkaline substrates is outstanding. On unprimed wood they generally show the lowest degrees of cracking ; this is true also for the lower comonomer content latices.

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Routine measurement of the viscosity of paint samples

A cone and plate viscometer with thermostatic control

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Summary

This paper describes the development of an instrument for the routine determination of the viscosity of paint samples. It is a cone and plate viscometer which operates at a set temperature of 25 C irrespective of wide variations of room temperature. It operates at a fixed rate of shear of 10,000 sec^{-1} and has a scale range of 0-5 poises ; measurements can be made to better than ± 0.1 poises. Other rates of shear and other scale ranges can be provided by making modifications and by using cones of different dimensions. The instrument is robust and simple to use and to clean.

Mesurage en routine de la viscosité des échantillons de peinture. Un viscosimètre à cône et plaque, muni de contrôle thermostatique

Résumé

Cet article décrit la mise au point d'un instrument destiné à la détermination en routine de la viscosité des échantillons de peinture. Il s'agit d'un viscosimètre à cône et plaque qui s'opère à une température constante de 25 C sans égard pour des variations étendues de température ambiante. Il fonctionne à un taux de cisaillement fixe de 10,000 sec^{1} et il possède une échelle dont la portée est 0-5 poise. Des taux de cisaillement différents et des portées différentes de l'échelle peuvent être assurés par l'introduction des cônes d'autres dimensions et par des modifications convenables. L'instrument se trouve robuste, et facile également à utiliser et à nettoyer.

Routine Viskositätsmessungen von Anstrichmittelproben Ein Kegel und Platte Viskosimeter mit thermostatischer Kontrolle

Zusammenfassung

In dieser Veröffentlichung wird die Ausarbeitung eines Instrumentes zur Routinebestimmung der Viskosität von Farb— und Lackmustern beschrieben. Es handelt sich um ein Kegel und Platte Viskosimeter, das bei einer feststehenden Temperatur von 25 C unabhängig von weiten Schwankungen in der Zimmertemperatur arbeitet. Das Instrument arbeitet mit einer bestimmten Scherkraft-Geschwindigkeit von 10,000 $Sek.^{-1}$ und ist mit einer 0-5 Poisen umfassenden Skala versehen ; Messungen, die besser als $\pm$ 0.1 Poisen sind, können vorgenommen werden. Andere Scherkraft—Geschwindigkeiten und andere Skalenumfänge können durch Vornahme von Änderungen und Anwendung von Kegeln anderer Grösse vorgenommen werden. Das Instrument ist solid gebaut, sowie einfach zu bedienen und zu säubern.

Установившийся в практике способ для измерения вязкости красочных образцов.

Вискозиметр снабженный конусом и пластинкой и термостатическим контролем.

Резюме

В статье дано описание развития инструмента для определения, установившимся в практике методом вязкости красочных образцов. Это вискозиметр конусно–пластин-

чатого типа, работающий при заданной температуре в 25°C независимо от больших изменений в комнатной температуре. Инструмент работает при установленной скорости сдвига в 10000 сек$^{-1}$ и имеет шкалу от 0 до 5 пуазов; измерения производятся с точностью до ± 0,1 пуаза. Можно обеспечить иные скорости сдвига и другие диапазоны шкал с помощью видоизменений и применением конусов различных размеров. Инструмент прочный и не предъявляет затруднений в применении и чистке

Introduction

There is a need in paint-testing laboratories for a simple instrument for the routine determination of the viscosity of paint samples. Of the various types of instruments available, cone and plate viscometers have several advantages: (*a*) they are quick to use ; (*b*) they can be made to operate at a high shear rate similar to that encountered during application ; (*c*) they need only a very small sample ; (*d*) they are very easy to clean.

A simple cone and plate viscometer embodying these advantages is the cone and plate attachment for the Rotothinner[1]. This, however, can only measure viscosity at the temperature of the instrument, as the small sample used quickly assumes the temperature of the cone and plate. Although this can be an advantage if the viscometer is housed in a constant temperature room, it considerably limits its use in the general laboratory where the temperature is not closely controlled. The present paper describes the design and development of a cone and plate viscometer with a thermostatically controlled plate which can be used to measure viscosity at a standard temperature irrespective of wide variations of room temperature. The operating temperature chosen for the experimental model is 25°C which can be maintained with room temperature variations between 10°C and 30°C. The speed of operation is such that, on homogeneous samples, a complete determination including cleaning can be made every minute.

The instrument has been used satisfactorily on paint testing in a laboratory for more than six months and has given reliable performance. It appears to be well liked by operators and is now suitable for production as a commercial instrument.

Discussion

Cone and plate viscometers in general

The cone and plate viscometer consists essentially of a motor rotating a conical plate in close proximity to a flat plate. The torque required to shear liquid between the cone and plate is proportional to the viscosity of the liquid.

Early reference to cone and plate viscometers was made by Higginbottom[2] and several commercially made instruments are now available, e.g. Ferranti[3], Weissenberg[4], Haake[5] and Brookfield[6]. However, these instruments, being rather sophisticated, are not ideally suited for making routine determinations in the paint industry.

The cone and plate system is one of the most satisfactory methods of measuring viscosities at high rates of shear, and the cone and plate attachment for the Rotothinner[1] was developed so that such measurements could be made with robust and inexpensive equipment. A disadvantage is that determinations can only be made at room temperature.

The new instrument: design considerations

The basic design of the new instrument is similar to the cone and plate attachment for the Rotothinner. The same arrangement for maintaining the spacing of the cone and plate has been adopted but it has been necessary to depart from the Rotothinner design in order to provide the thermostatic control to the plate.

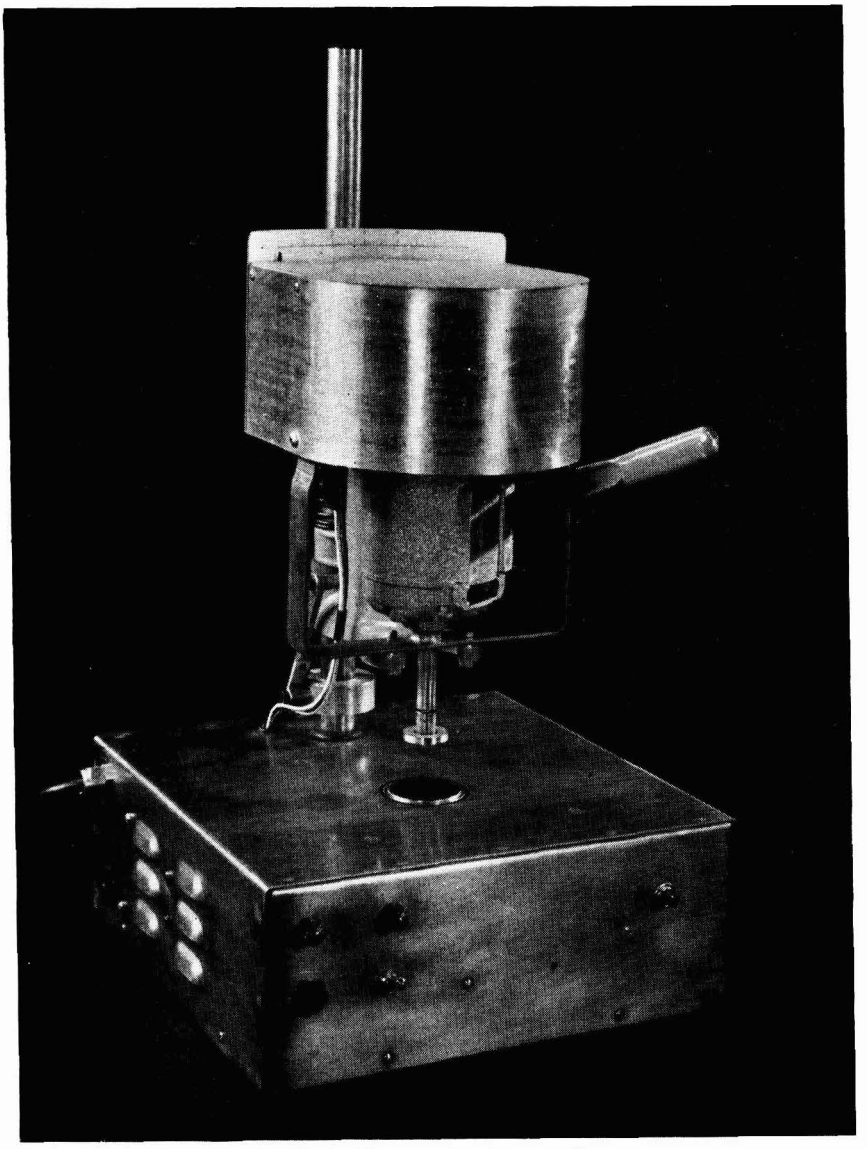

Fig. 1. The complete instrument

In the new design the plate is fitted to the base of the instrument and measurement made of the torque reaction of the motor driving the cone. This has the advantage that the motor and torque measuring part of the instrument are kept at the top while the stationary plate can form part of the base housing the thermostatting equipment. The design finally adopted is shown in Figs. 1 and 2.

The rate of shear chosen for the instrument was 10,000 sec^{-1} as this is frequently regarded as typical of application by brush. With a synchronous motor of 1,500 rpm this shear rate can be obtained with a cone angle of one degree. Other rates of shear can be obtained by using cones with different cone angles.

The torque transmitted when a cone of $1\frac{1}{8}$ in in diameter is used, is well within the capacity of the 0.01 hp sycnhronous motor chosen and yet sufficient to enable a reasonably robust torque measuring system to be used.

The viscosity scale range chosen for this model was 0-5 poises, as experience has shown that this covers the normal range of paints. This scale range can be changed to suit different materials by modifying the instruments and using cones of different diameters.

It was decided to control the temperature of the plate thermostatically at 25°C as this is commonly used for viscosity determinations in this country. However, the instrument can be modified to work at 20°C or any other temperature which may be more suitable to local conditions. It is desirable that the control temperature should be maintained irrespective of quite large room temperature variations, but in order not to make the equipment too expensive the maximum range likely to be encountered in this country was taken to be 10-30°C. Below 10°C the operator would probably make some arrangements about warming the room for reasons of personal comfort, and air temperatures above 30°C are only rarely encountered here. Such control would necessarily require some refrigeration and so a Frigistor[7] has been used ; this is a semiconductor Peltier effect device which can produce a hot or cold surface according to the direction of the DC electrical supply. It is necessary that one face of the Frigistor is kept cool by running water, or a flow of air over cooling fins. Air cooling was chosen for this instrument as the conditions do not demand the maximum cooling effect of the Frigistor and it is convenient to have an instrument which is independent of a supply of running water. It is arranged that the cone is normally in contact with the plate except for filling and cleaning. In this way the temperature of the cone keeps very near to that of the plate and the small amount of liquid required to fill the gap quickly assumes the mean temperature.

To illustrate the effectiveness of the temperature control, the instrument was tested with a standard oil of 3.9 poises at 25°C. The results obtained at different room temperatures are shown in Table 1.

The results show that the instrument performs within a tolerance of ± 0.1 poises at room temperatures from 8°C to 30°C. The range could be widened to include higher and lower temperatures, as experienced overseas, by increasing the power supply to the Frigistor.

Table 1

Temperature of room (°C)	*Indicated viscosity in poises (after 15 sec. running)*
8	4.0
11	4.0
18	4.0
22	4.0
24	3.9
25	3.9
26	3.9
28	3.9
30	3.9
32	3.8
34	3.7
36	3.6

During running, however, there is some heating of the specimen by viscous shear. This has a negligible effect on the readings below two poises but at higher viscosities it has a small but significant effect. Table 2 shows the readings obtained after various times of running when samples of oil at different temperatures are put into the viscometer. It can be seen from the figures that a time of 15 sec is required for good mixing and thermostatting of the oil but if this figure is exceeded the effect of self-heating becomes evident. Satisfactory readings within the tolerance of the instrument can be obtained between 15 and 30 sec running time.

Table 2

Running time (seconds)	*Oil at 2.4 poises at 25°C Scale reading*		*Oil at 3.9 poises at 25°C Scale reading*	
	original oil temp. 15°C	*original oil temp. 25°C*	*original oil temp. 15°C*	*original oil temp. 25°C*
5	2.5	2.4	4.2	3.95
10	2.4	2.4	4.1	3.9
15	2.4	2.4	3.9	3.85
20	2.4	2.35	3.85	3.8
25	2.35	2.35	3.85	3.8
30	2.35	2.35	3.8	3.75
60	2.3	2.3	3.7	3.7

When solutions of materials of high molecular weight are sheared between the cone and plate, normal forces are produced which tend to separate the cone from the plate ; these can be overcome by spring or dead weight loading. The instrument uses dead weight loading of approximately 50g per sq cm, sufficient to spread the sample and resist any normal forces developed during running but not so great that it would cause appreciable wear.

Mechanical construction

The instrument is shown in diagram form in Fig. 2. It consists of a base *A* which supports a vertical column *B*, and a head assembly *C* which can be raised or lowered by means of a lever *D*. The head assembly is mounted on a casting

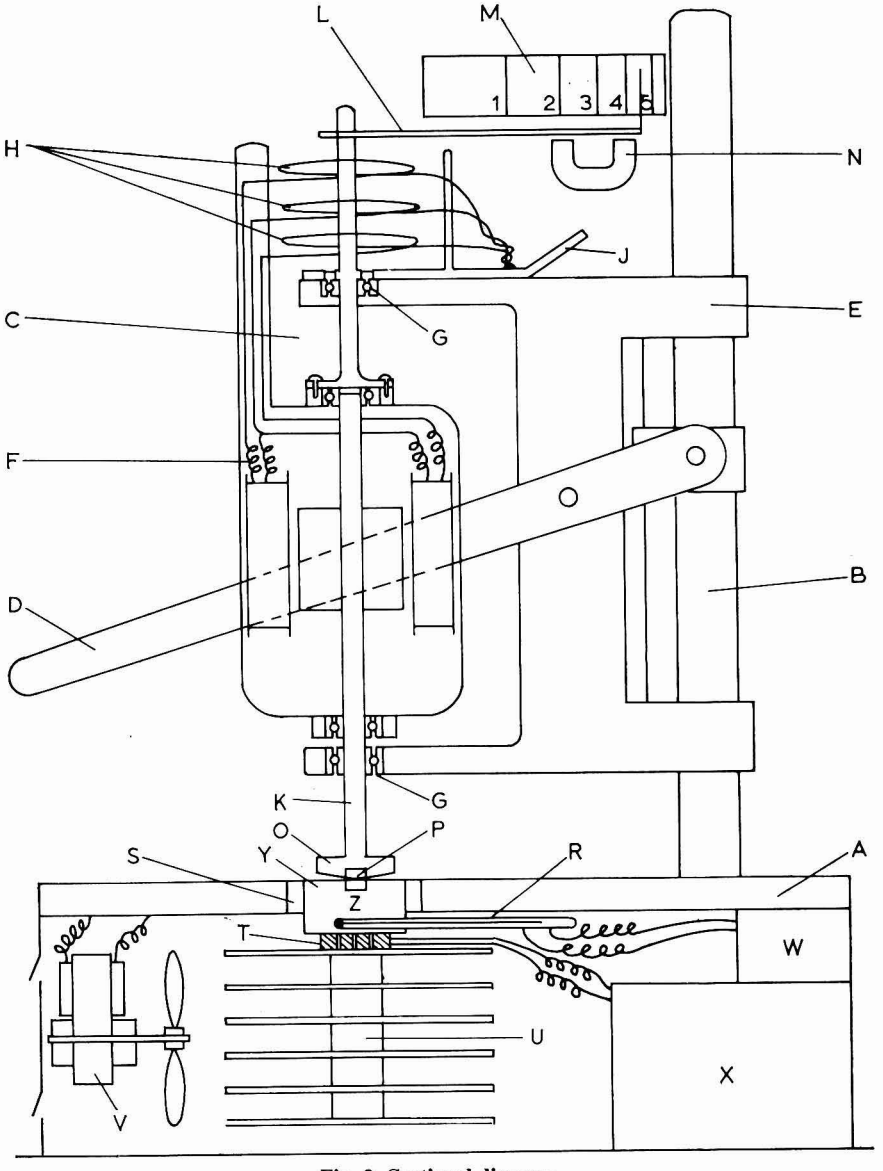

Fig. 2. Sectional diagram

E which is a sliding fit on the column. This casting supports a Framco synchronous motor *F* which has its axis vertical and parallel to the column *B*. The body of the motor is modified so that it is held in the casting by means of two ball-races *G* and is free to rotate independently of the rotation of the rotor of the motor. This freedom to rotate is restrained by torque springs *H* which are attached to the motor body at one end and to a zero adjusting lever *J* at the other. In operation the torque transmitted by the motor shaft *K* gives an equal and opposite torque to the motor body which is opposed by the springs. A pointer *L* is attached to the motor body and angular deflections of the motor due to torque are indicated on the scale *M*. Damping is arranged by means of a copper sector working in the gap of a permanent magnet *N*. The torque springs are made of 22 SWG phosphor bronze wire insulated with polythene sleeving and serve as electrical conductors to supply electricity to the motor. The lower end of the motor shaft carries the cone *O* which is $1\frac{1}{8}$ in in diameter and has an apical angle of 178°. The centre of the cone has a high speed tool steel insert *P* which forms the point and makes contact with the plate.

The base of the instrument is formed from the aluminium casting *A* into which is inserted the viscometer plate *Y*. The latter is thermally insulated from the casting by means of the plastic collar *S*. The viscometer plate *Y* which is of mild steel with a high speed tool steel insert, is mounted on a copper block *Z* which carries the pre-set contact thermometer *R*. Under the copper block is mounted the Frigistor unit *T* and under this the heat sink *U* consisting of six plates of 20 SWG copper $\frac{3}{8}$ in apart. The whole of this assembly is held to the aluminium casting by means of brackets and an adjustment is provided for levelling the plate. Heat is dissipated from the heat sink by means of the fan *V*.

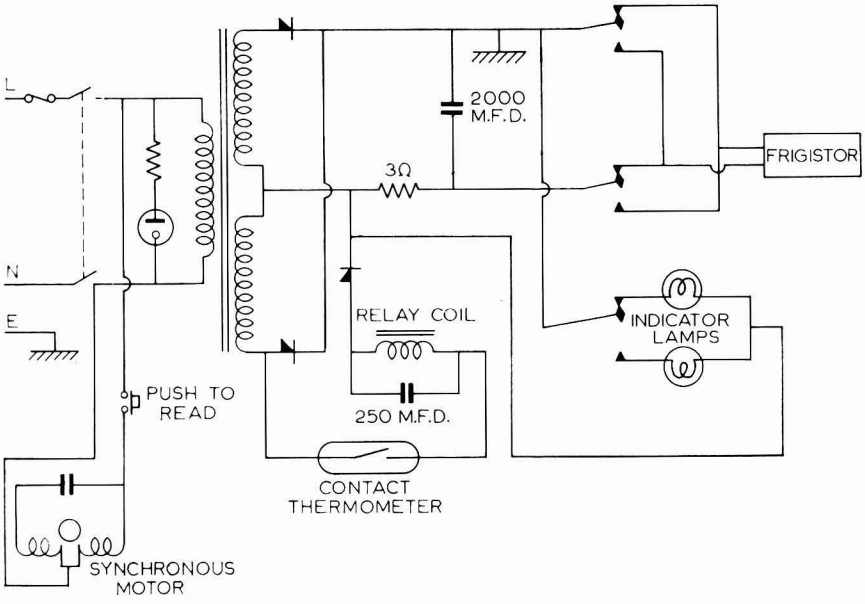

Fig. 3. Wiring diagram

The pre-set contact thermometer *R* operates a relay *W* which changes the direction of the current flowing through the Frigistor (see wiring diagram Fig. 3). The direct current supply for the Frigistor is provided by the power pack *X* consisting of a transformer and silicon rectifiers. An indication of whether the Frigistor is on the heating or cooling cycle is given by pilot lights.

Acknowledgements

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Formulation of fungus-resistant paints

1: Addition of pentachlorophenol

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Summary

The effectiveness of pentachlorophenol (PCP) as a fungicide is assessed. Panels painted with gloss and flat enamel paints were exposed at Lae, New Guinea, and houses in Melbourne were painted inside with a flat enamel paint containing PCP. The mould growth in each case is compared with that on the same paint free of fungicide. The loss of PCP from paint films exposed under different conditions was determined.

PCP is found to be of no particular use as a fungicide for outdoor application. A paint containing PCP performs better indoors than one free of it. Analytical results show that PCP is lost very quickly from the paint film, especially in humid conditions or at high temperatures.

La mise au point des formules de peintures résistant à végétation mycélienne

1: L'addition de pentachlorophénol

Résumé

L'efficacité de pentachlorophénol (PCP) en tant que fongicide est appréciée. Panneaux-éprouvettes revêtus des peintures-émaux brillantes et mates ont été exposés à Lae, Nouvelle-Guinée, et d'ailleurs quelques bâtiments à Melbourne ont été peints à l'intérieur avec une peinture mate contenant PCP. La croissance de moisissure en chaque instance est comparée auprès de celle sur la même peinture exempte de fongicide. La perte de PCP des feuils de peinture exposés sous diverses conditions a été déterminée.

On trouve que PCP ne possède pas de valeur remarquable en tant que fongicide pour l'usage extérieur. Une peinture contenant PCP se comporte mieux à l'intérieur qu'une telle peinture exempte de PCP. Résultats analytiques démontrent que PCP se perd très rapidement du feuil de peinture, notamment sous des conditions humides ou à des températures élevées.

Ausarbeitung einer Rezeptur für Pilzbeständige Anstrichfarben

1: Zusatz von Pentachlorphenol

Zusammenfassung

Die Wirksamkeit von Pentachlorphenol (PCP) als Pilzverhütungsmittel wird bewertet. Es wurden Tafeln mit glänzendem und mattem Emaillelack in Lae, Neu Guinea dem Wetter ausgesetzt, und Häuser in Melbourne wurden innen mit einem matten Emaillelack gestrichen. Diese Lacke enthielten PCP. In allen Versuchen wird das Pilzwachstum an den gleichen, aber pilzverhütungsmittelfreien Farben verglichen. Der Verlust an PCP von unter verschiedenen Bedingungen exponierten Anstrichfilmen wurde bestimmt.

Dabei wurde festgestellt, dass PCP Pilzverhüter in Aussenanstrichen keinen besonderen Zweck erfüllt. Eine für Innenanstrich verwandte PCP enthaltende Farbe verhält sich jedoch besser als eine, die davon frei ist.

Analytische Resultate ergeben, dass der Anstrichfilm PCP sehr schnell, besonders unter feuchten Bedingungen und bei hohen Temperaturen, verliert.

Формулировка красок с сопротивлением против плесени

1. Добавление Пентахлорофенола.

Резюме

Оценивается эффективность пентахлорофенола (ПХФ) как средства для истребления плесени. Панели, окрашенные блестящими и матовыми эмалевыми красками были подвержены действию лучей света в Лао, Новая Гинея, и жилища в Мельборне были окрашены внутри матовой эмалевой краской содержащей ПХФ. В каждом случае нарастание плесени сравнивалось с плесенью на одинаковой краске без фунгисида. Определялась потеря ПХФ в красочных пленках при различных условиях экспонирования. Установлено что ПХФ не проявляет особых качеств как истребитель плесени под открытым небом. Краска содержащая ПХФ дает лучшие результаты внутри дома чем краска не содержащая ПХФ.

Аналитические данные показывают что ПХФ быстро испаряется из красочной пленки, особенно в условиях высокой влажности или при высокой температуре.

Introduction

The disfigurement of painted surfaces, both indoors and outdoors, is frequent enough in Australia to justify an investigation into the formulation of mould-resistant paints. One very simple way of suppressing mould, if it worked, would be to add a fungicide to the coating. A great many compounds have been proposed for this purpose but their efficacy in practice has not been proved. In a previous paper[1] the authors reviewed the available methods for testing the fungus resistance of paint, and emphasised the unreliability of these procedures.

It was pointed out some time ago[2] that determination of the stability of a fungicide in a paint film exposed under conditions similar to those encountered in the field might help to forecast the usefulness of the compound. The present paper deals with the loss of pentachlorophenol (PCP) from paint films, and the actual performance of coatings containing PCP after exposure in both indoor and outdoor environments favourable to mould growth.

Experimental

Analytical work

Flat and gloss alkyd paints containing approximately 3 per cent PCP and the corresponding blanks were brushed out on sheets (12 in × 6in) of a polyethylene terephthalate polyester film which were then attached to hardboard panels and exposed under the following conditions :

(1) outdoors at Highett, Victoria.

(2) in a fog room at 20°C.

(3) in a constant temperature room kept at 20°C and 65 per cent R.H.

(4) in a hot room at 28°C.

(5) outdoors at Lae, New Guinea.

The panels at Highett were mounted on racks at an angle of 45° facing north; those at Lae had the painted films attached to both sides and were mounted at 45° and facing 31° east of true north.

Table 1
Incidence of mould growth in houses painted with a flat alkyd enamel paint containing PCP

Inspection*	Houses									Rooms								
	No PCP			1% PCP			2% PCP			No PCP			1% PCP			2% PCP		
	Number inspected	Number affected	% affected	Number inspected	Number affected	% affected	Number inspected	Number affected	% affected	Number inspected	Number affected	% affected	Number inspected	Number affected	% affected	Number inspected	Number affected	% affected
1	11	6	54.5	14	8	57.1	22	1	4.5	50	6	12.0	53	10	18.9	73	1	1.4
2	15	7	46.6	16	5	31.2	24	3	12.5	80	10	12.5	57	6	10.5	82	3	3.7
3	13	10	77	16	8	50	20	5	25	51	13	25.4	60	13	21.6	66	7	10.6

*Inspection made annually after each winter season.

The analyses were carried out by extracting the PCP with cyclohexane and determining it spectrophotometrically. Not all of the PCP can be extracted in this way, and the residue of the extraction was analysed for chlorine which was accounted for as PCP (non-extractable). The non-extractable part is probably chemically bound by reaction between the hydroxyl group of the PCP and the carboxyl group of the paint medium. Details of this analytical method have been described previously[3].

Evaluation indoors

Evaluation of the paint indoors was carried out in 55 dwellings in which a comparatively high degree of disfiguration by mould had necessitated redecoration. Sixteen dwellings were painted with a flat alkyd enamel containing 1 per cent PCP, 24 were painted with the same enamel containing 2 per cent PCP, and the remaining 15 were decorated with the same flat alkyd enamel without fungicide. Before painting, all walls were washed down with methylated spirit. The houses were inspected after each winter season, when mould is most prolific, and mould growth was then washed off with a hypochlorite solution. The results are summarised in Table 1.

Exposure at Lae, New Guinea

A separate set of hardboard panels was exposed at Lae, New Guinea. Both sides of the panels were painted with a paint containing approximately 3 per cent PCP or with a paint without PCP. The intensity and extent of mould growth was assessed at intervals of about three months. The results are summarised in Tables 2 and 3.

Table 2

Assessment of mould growth on paint films exposed outdoors at Lae : upper side of panels

Paint	Replicate number	Exposure											
		4 months		7 months		10 months		14 months		17 months		20 months	
		I	C	I	C	I	C	I	C	I	C	I	C
G	1	2	50	2-3	100	2-3	100	2	100	3	100	3	100
	2	2	50	2-3	100	2-3	100	2	100	3	100	3	100
GP	1	2	5	2	50	2	50	2	100	3	100	3	100
	2	2	5	2	100	2	50	2	100	3	100	3	100
	3	2	5	2	50	2	50	2	100	4	100	4	100
	4	2	5	2	75	2	50	2	100	—	—	—	—
F	1	4	10	3-4	100	3	100	3	100	3	100	3	100
	2	4	10	3-4	100	3	100	3	100	3-4	100	4	100
FP	1	0	0	0	0	0	0	3	100	3	100	3	100
	2	0	0	1-2	100	3	50	3	100	3	100	3	100
	3	2	10	1-2	100	1-2	20	3	100	3	100	3	100
	4	2	10	1-2	100	3	1	—	—	—	—	—	—

G, gloss paint without PCP ; GP, gloss paint with PCP ; F, flat enamel paint without PCP ; FP, flat enamel paint with PCP.

Each panel was assessed by noting the percentage area " C " covered by mould and by the colour intensity " I " according to the following scale, 9(black)—→0(white).

Table 3

Assessment of mould growth on paint films exposed outdoors at Lae : under side of panels

Paint	Replicate number	Exposure											
		4 months		7 months		10 months		14 months		17 months		20 months	
		I	C	I	C	I	C	I	C	I	C	I	C
G	1	2	1	7	15	3	100	2-3	100	2-4	100	4	95
	2	2	1	7	20	3	100	2-3	100	2-4	100	4	90
GP	1	1-2	50	3	1	3	1	2	100	3-4	80	4	80
	2	0	0	3	1	3	100	2-3	100	3-4	80	2-3	100
	3	1-2	50	3	1	3	100	2	100	3-4	90	4	80
	4	0	0	3	1	3	100	3	100	—	—	—	—
F	1	2-3	75	3	100	2-3	100	2-3	100	2-4	100	2-3	90
	2	4-5	85	3-4	100	2-3	100	2-3	100	3-5	100	3-7	100
FP	1	1	50	1	100	1	100	1-2	100	1	100	9	1
	2	1	50	1	100	1	100	1-2	100	1-2	100	8	1
	3	1	50	1	100	1-2	100	1-2	100	1	100	8	2
	4	1	50	1	100	5	1	—	—	—	—	—	—

G, gloss paint without PCP ; GP, gloss paint with PCP ; F, flat enamel paint without PCP ; FP, flat enamel paint with PCP.

Each panel was assessed by noting the percentage area " C " covered by mould and by the colour intensity " I " according to the following scale, 9(black)—→ 0(white).

Discussion

Analytical results

The samples were analysed for extractable and non-extractable PCP. The latter always increases on exposure sometimes by as much as 50 per cent. However, only the extractable PCP is likely to be of any value as a fungicide, since to be effective it must be able to diffuse to the surface. Figs. 1 and 2 show the values for the extractable PCP, which decreases in all cases.

The highest rates of decrease were in the fog room and in the hot room (curves 4 and 6). A loss of between 80 and 90 per cent occurred after only 14 months exposure at Lae (curves 1 and 2), but in the constant temperature room the losses amounted to 50 per cent after two years (curve 5). The losses from films exposed outdoors at Highett (curve 3) were similar to those in the constant temperature room.

The loss occurring between 20°C and 38°C showed that a large amount of the loss was due to volatilisation. No decomposition was likely to occur under these conditions. On the other hand, the high rate of loss in the fog room at only 20°C was probably due to hydrolysis of PCP in a film saturated with water.

Visual assessment—indoors

Results of the survey made over three seasons are summarised in Table 1. A statistical analysis shows that the addition of 1 per cent PCP to the paint does not increase the mould resistance, but that it is significantly increased ($P<0.05$) by adding 2 per cent PCP.

Visual assessment—outdoors

The results of the assessments of the panels at Lae are given in Tables 2 and 3. On the upper side (Table 2), coatings containing PCP had a slightly better appearance than the controls up to ten months, but there was no difference thereafter. On the under side (Table 3), the gloss paint film was slightly better up to seven months, whereas the flat coating containing PCP had a slightly better appearance throughout the test.

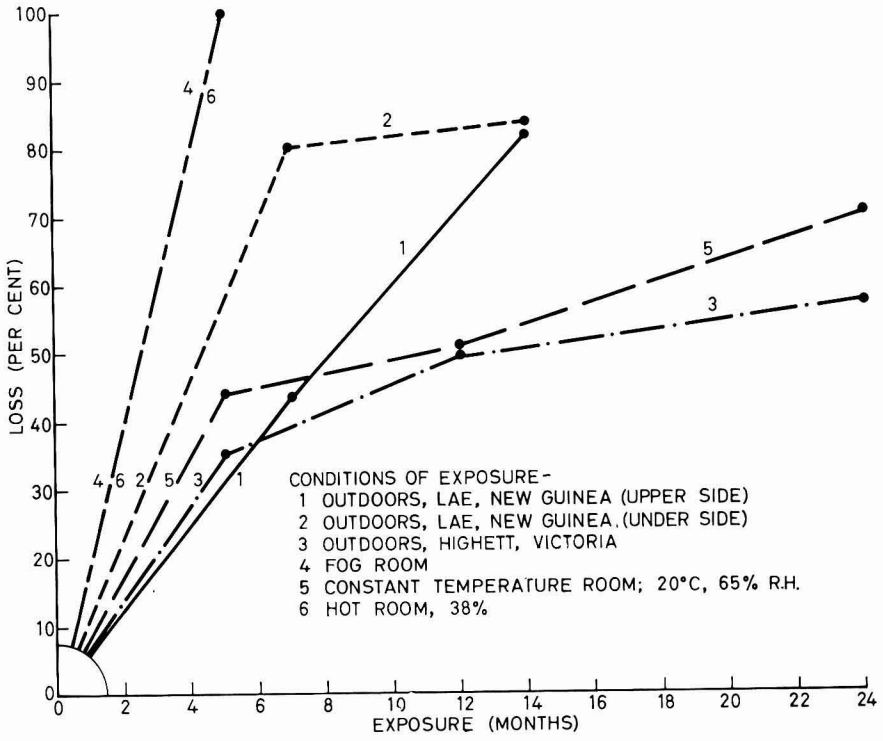

Fig. 1. Loss of extractable PCP from gloss enamel paint film. Initial analysis—extractable PCP, 2.20 per cent ; non-extractable PCP, 1.10 per cent

Conditions of exposure :

1. Outdoors, Lae, New Guinea (upper side)
2. Outdoors, Lae, New Guinea (under side)
3. Outdoors, Highett, Victoria
4. Fog room
5. Constant temperature room ; 20°C, 65 per cent RH
6. Hot room, 38°C

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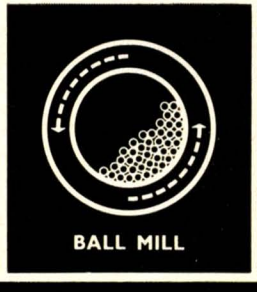

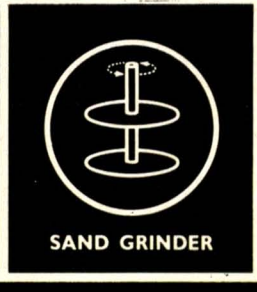

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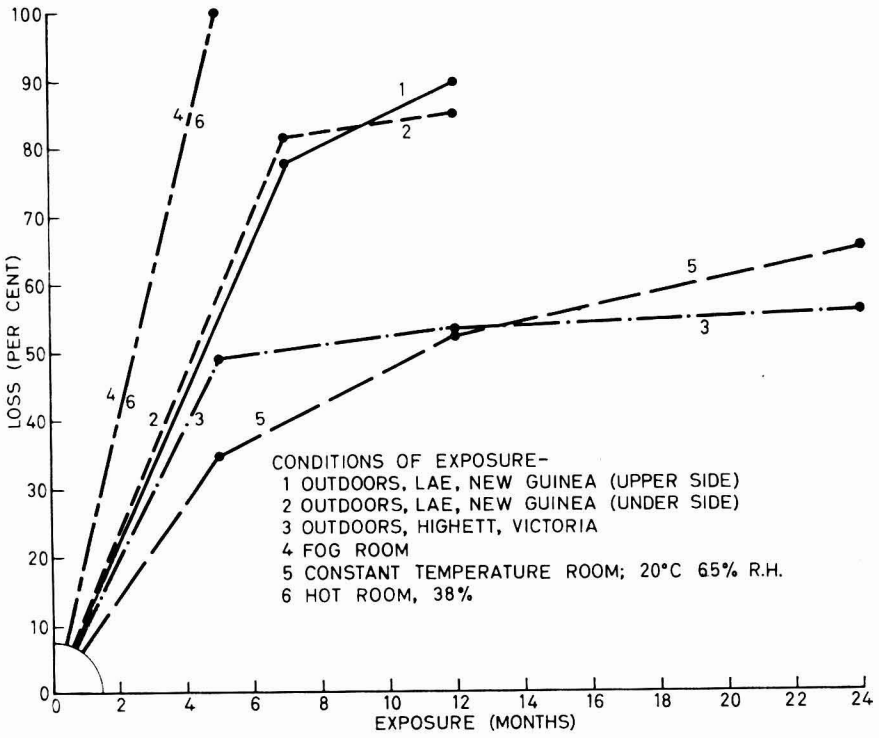

Fig. 2. Loss of extractable PCP from flat enamel paint film. Initial analysis—extractable PCP, 3.2 per cent ; non-extractable PCP, 0.8 per cent

Conditions of exposure :

1. Outdoors, Lae, New Guinea (upper side)
2. Outdoors, Lae, New Guinea (under side)
3. Outdoors, Highett, Victoria
4. Fog room
5. Constant temperature room ; 20 C, 65 per cent RH
6. Hot room ; 38 C

Conclusions

The results show that addition of PCP to paint even at the comparatively high level of about 3 per cent does not increase the mould resistance sufficiently to be of any value in outdoor exposure. (This quantity will produce a paint film containing a higher percentage of PCP depending on the solid content of the paint.)

The analytical results show that this could be due to the loss of fungicide from the paint film, probably by volatilisation and hydrolysis of the PCP under humid conditions. From the fact that 1 per cent of PCP in paint does not increase the fungus resistance indoors, it can reasonably be assumed that more

than 1 per cent extractable phenol must be present in a paint film to make it fungus-resistant. A coating should therefore contain at least 6 per cent extractable PCP if the surface is to be mould-resistant even for 14 months in the humid tropics.

The addition of 2 per cent PCP to paint used indoors gives a significant improvement in the mould resistance, but this appears to decrease as the PCP is lost from the paint film. The rate of loss is much lower than in paint exposed outdoors, and this is the reason for its longer effectiveness. A paint that would stay active indoors for five to six years would need from 4 to 5 per cent PCP to give the desired effect.

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New developments in carbon black technology in relation to pigmentation*

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Summary

The significance of the particle size distribution of carbon blacks is discussed in relation to structure, potential jetness (Blackness) and undertone. Surface area is shown to affect the amount of protective colloid type dispersing agents as well as the pigment-to-binder ratio to ensure stability in mill pastes. Conversely the minimum requirement of ionic dispersant is influenced by the concentration of acidic oxygen on the carbon black surface. Oil absorption is governed by the superficial surface area independent of porosity.

Characteristic differences in the proportions of different oxidised groupings on the black surface are indicated for carbon blacks manufactured by different processes. Carboxylic groupings play a major part in determining the rheology of dispersion in polar media. Thermal after-treatment increases the amount of oxygen on the surface, but at the expense of increased porosity evidenced by high nitrogen surface areas. Newer methods of after-treatment give relatively high proportions of strongly acidic surface groups without appreciably increasing porosity. This allows better dispersed properties and enhanced optical properties in the dry film.

Application is made of some of these characteristics in the case of the formulating of a mill paste for maximum colour development of a ball milled baking enamel. Additionally the vehicle itself is shown to influence dispersibility, rheology and colour development as exemplified in a series of linseed stand oil lithographic inks.

Développements récents à l'égard de pigmentation dans la technologie de noir de carbone

Résumé

L'importance de la composition granulométrique des noirs de carbone est discuté au point de vue de structure, de noirceur absolu potentiel, et de sous-ton. On demontre l'effet exercé par l'aire superficielle sur la quantité d'agents de dispersion du type colloide protecteur ainsi que le rapport pigment/liant afin d'assurer la stabilité des masses broyantes. Au contraire, la teneur minimum d'agent dispersant ionique est influencée par la concentration d'oxygène acide à la surface du noir de carbone. La prise d'huile est régie par l'aire superficielle de la surface ne tenant aucun compte de porosité.

On mentionne des différents caractéristiques en ce qui concerne les proportions de divers groupements oxydés sur la surface des noirs de carbone preparés au moyen des procédés différents. Groupements carboxyliques jouent un role majeur en déterminant la rheologie de dispersions dans milieux polaires. Post traitement thermique augmente, à la fois, la quantité d'oxygène sur la surface et la porosité que témoignent les valeurs élevées d'aire superficielle rendues par la méthode d'absorption d'azote. Méthodes perfectionnées de post traitement produisent des proportions assez élevées de groupements superficiels fortement acides, sans augmenter sensiblement la porosité, qui assurent dans le feuil sec un meilleur degré de dispersion et des propriétés optiques augmentées.

On peut utiliser certains de ces caractéristiques pendant la mise au point de formules d'une masse broyante pour rendre le développement maximum de couleur dans un émail au four

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achevé par broyage à billes. En outre on demontre, au moyen d'une série d'encres lithographiques à base de standolie, l'influence éxercée par le véhicule, soi-même, sur l'aptitude à disperser, la rhéologie et le développement de couleur.

Neuentwicklungen auf dem Gebiet der Carbon Black Technologie hinsichtlich Pigmentierung

Zusammenfassung

In dieser Abhandlung wird die Bedeutung der Teilchengrössenverteilung von Carbon Blacks im Zusammenhang mit Struktur, grösstmöglichster Tiefschwärze und Unterton besprochen. Es wird gezeigt, dass es zur Erhaltung der Stabilität von Mahlpasten erforderlich ist, sowohl die Abhängigkeit der Quantität schutzkolloidartiger Dispergierungsmittel, als auch des Pigment-Binder Verhältnisses von der Grösse der Oberfläche zu beachten. Umgekehrt wird der erforderliche Mindestzusatz eines ionischen Dispergierungsmittels von der Konzentration säurebedingten Sauerstoffs an der Carbon Black Oberfläche beeinflusst. Das Ölaufnahmevermögen hängt unabhängig von Porosität von der Grösse der Oberflächenoberschicht ab.

Die durch verschiedenartige Herstellungsprozesse bedingten charakteristischen Unterschiede im Mengenverhältnis der verschiedenen an der schwarzen Oberfläche der Russe befindlichen oxidierten Gruppierungen werden aufgezeigt. In polaren Bindemitteln spielen Carboxylgruppen insofern eine grössere Rolle, als sie die Rheologie der Dispersion bestimmen. Thermische Nachbehandlung erhöht zwar den Sauerstoffgehalt an der Oberfläche, jedoch, wie der hohe Stickstoffgehalt an der Oberfläche beweist, auf Kosten vergrösserter Porosität. Neuere Nachbehandlungsmethoden ergeben verhältnismässig hohe Anteile an stark sauren, an der Oberfläche befindlichen Gruppen, ohne die Porosität wesentlich zu erhöhen. Dadurch wird der Grad der Dispergierung erhöht, und die optischen Eigenschaften des trockenen Films werden verbessert.

Einige dieser charakteristischen Eigenschaften werden als Beispeil in einer für höchste Farbkraftentwicklung formulierten Mühlenpasta mit einer mittels Kugelmühle gemahlenen Ofenemaille zur Anwendung gebracht. Ausserdem wird an Hand einer Serie von Leinölstandöl enthaltenden lithographischen Druckfarben aufgezeigt, dass das Bindemittel selbst Dispergierbarkeit, Rheologie und das Herausbringen der Farbtiefe beeinflusst.

Новые усовершенствования в технологии сажи по отношению к пигментации

Резюме

Обсуждается значение распределения частиц по величине сажи по отношению к структуре, потенциальной черноте и полутону. Показано что поверхностная площадь влияет на количество дисперсионных веществ защитного коллоидного типа и также на отношение грунтовой краски к связывающему веществу с целью обеспечения устойчивости заводских паст. Напротив, минимальное требование ионного дисперсионного вещества зависит от вияния консентрации окисленного кислорода на поверхности сажи. Абсорбция масла регулируется площадью поверхности независимо от пористости.

Даются характеристические различия пропорций разных окисленных групп на черной поверхности в производстве сажи по различным процессам. Карбоскильные группы играют более важную роль в определении реологии дисперсии в полярных средах. Вторичная термическая обработка увеличивает количество кислорода на поверхности но за счет повышения пористости, которая проявляется в повышении количества азота на поверхностных площадях.

Современные методы вторичной обработки дают сравнительно высокую пропорцию сильно кислотных поверхностных групп без значительного увеличения пористости. Это дает возможность улучшения дисперсных свойств и повышения оптических свойств сухой пленки.

Некоторые из этих характеристик применяются в формулировке заводской пасты для наибольшего развития цвета в эмали горячей сушки, приготовленной из шарикового краскотертого материала. Помимо того, показано что само связывающее вещество влияет на дисперсность, реологию и развитие цвета, что иллюстрируется в ряде льяноолифных литографических чернил.

Introduction

The rheology of a carbon black dispersion and the ultimate optical properties of the pigmented surface are both affected by the extent and nature of the carbon black surface. In the past these properties have been described in terms of average particle size derived from actual counts of electron micrographs, surface area derived from low temperature nitrogen adsorption isotherms, volatile content and oil absorption[1]. Anomalous behaviour of certain blacks or families of blacks have made it necessary to reconsider these simple criteria, as a result of which new concepts are being evolved to explain these difficulties and to modify properties in order to tailor blacks for specific purposes.

In the past few years several carbon blacks with novel properties have appeared commercially and many more are in various stages of development. These developments have been indicated by increasing knowledge and have in turn added to the understanding of the subject. Because of the introduction of these newer carbon blacks and the fact that they can be handled rather more scientifically, it is perhaps timely to review the present state of the knowledge.

The effect of particle size

Pigmenting blacks range in particle size from about 4,000 Å down to about 100 Å and it is generally recognised that potential jetness increases as particle size decreases. It is at once obvious that actual jetness will be limited by the degree of dispersion attained and this in turn will be affected by both the vehicle and the dispersing equipment, since both govern the shearing forces available to break up pigment agglomerates. Other complicating factors include the affinity of pigment and vehicle as determined by the surface chemistry of the carbon black and the dispersibility as affected by the physical state of the surface.

Apart from these factors which will be analysed subsequently, the importance of particle size distribution as distinct from average particle size became apparent soon after the advent of oil furnace blacks. Despite an overlap of particle sizes of the finer oil furnace blacks and the coarser channel blacks, the latter were jetter and browner. Comparison of size distribution curves calculated from electron micrographs showed that the oil furnace blacks had a " skew " distribution, i.e. they had a " tail " of coarse particles. Whether these were really ultimate particles or arose as a result of the higher " structure " of these blacks, the optical result was the same, *viz.* increased diffuse reflectance corresponding to somewhat coarser particles and slightly greater reflectance at the blue end of the spectrum.

The particle size distribution curves for several blacks are presented in Fig. 1. Carbolac 1 is the finest of the series of channel blacks with an arithmetic mean diameter of 100 Å, whereas at the other end of the spectrum Spheron 9 has an arithmetic mean diameter of 290 Å ; these channel blacks have a fairly normal distribution. The high structure oil furnace black, Vulcan 3, has an average particle diameter slightly finer than Spheron 9, but it also has a proportion of coarse particle in the 600-900 Å range. As a result of this, Vulcan 3 appears less jet than Spheron 9 and is rated at 90 Scale (corresponding with

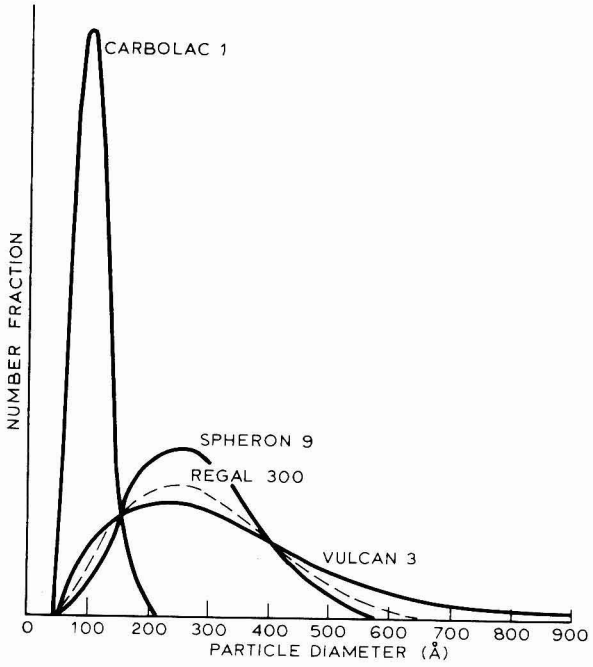


Fig. 1. Particle size distribution of typical carbon blacks

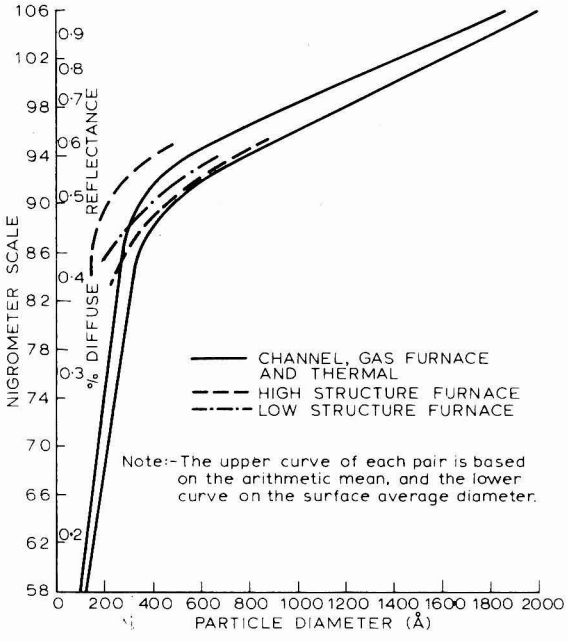


Fig. 2. Relation of potential jetness to particle size

a diffuse reflectance of 0.5 per cent) in the Cabot Nigrometer test as compared with 84 Nigrometer Scale (0.4 per cent diffuse reflectance) for Spheron 9.

The relation between potential jetness and particle size is illustrated in Fig. 2. Nigrometer Scale (or diffuse reflectance) is plotted against particle diameter for the whole spectrum of carbon blacks. The upper curve of each pair is based on the arithmetic mean diameters, the full curve representing the relationship for channel, gas furnace and thermal blacks. The upper broken curve is that for high structure oil furnace blacks and it is confirmed that they are lighter than their arithmetic mean diameters would indicate.

By replotting the curves in terms of *surface average diameter*

$$\frac{\Sigma nd^3}{\Sigma nd^2}$$

(lower pair of curves) the effect of the " skew " distribution is partially taken into account and the curves lie closer together. The significance of this parameter can be envisaged if it is postulated that the scattering of incident light by the pigment is proportional to the extent of the surface of the individual particles and thus the net optical effect of an assembly of particles of different sizes will be the same as that of particles at the diameter corresponding with the average surface area.

On the other hand, referring back to Fig. 1, the low structure oil furnace blacks typified by Regal 300 are less skewed than higher structure blacks and therefore appear darker. Extremely low structure blacks fall below the full curve in Fig. 2, suggesting that the larger " particles " of high or even normal structure blacks are indeed agglomerates.

Contribution of surface area

The surface area of a given weight of smooth spheres is inversely proportional to their mean surface diameter. Thus Vulcan 3 with a mean surface diameter of 436 Å is calculated to have a superficial area of 74 m^2/gm, whilst Carbolac 1 with a diameter of 122 Å should have 264 m^2/gm of surface. Calculated from nitrogen adsorption isotherms by the method of Brunauer, Emmett and Teller, Carbolac 1 surface area is close to 1,000 m^2/gm, although Vulcan 3 is only 77 m^2/gm. The obvious inference is that Vulcan 3 particles are relatively smooth and non-porous whereas Carbolac 1 particles are highly porous. The manner in which these pores arise will be outlined later in this discussion, but it will be seen that the ratio of nitrogen surface area to calculated surface area gives a measure of porosity.

For the present the main effects arising from these relatively large surface areas in comparison with other pigments will be considered. Since wetting involves the replacing of a pigment/air interface with a pigment/vehicle interface at the surface of agglomerates, and dispersion involves the breakdown of these agglomerates and coating the surface thus exposed with vehicle, the energy required to achieve dispersion increases as the surface area increases, other things being equal. In practice the surface chemistry will also have an effect. In view of the relatively large surface areas of carbon blacks in comparison with other pigments they are, in general, more difficult to disperse and milling under conditions of high shear is recommended.

Surface area is also important in relation to dispersion in so far as it influences the amount of dispersing agent, protective colloid, or vehicle solids required to ensure stability. In the case of ionic dispersants the effect is modified by the level of volatile as will be shown subsequently.

The physical adsorption of a soap or an amine, or a polymeric dispersant or even of the resin itself, however, is governed by the amount of surface available.

Table 1

Optimum dispersing agent requirement for nitrocellulose lacquers

	N_2 Surface area (m^2/g)	Volatile (%)	% Copper oleate on black
Supercarbovar	400	5	12½
Carbolac 2	850	13	25
Carbolac 1	950	16	30

In Table 1 the optimum requirement of copper oleate to achieve the jettest nitrocellulose lacquers for each of three blacks is tabulated. The dispersing agent requirement is proportional to the surface area and independent of the amount of volatile. In the case of polymeric material the actual weight percentage required to satisfy the surface will be of a different order from the above, but the same proportionality will hold.

Table 2

Requirement of anionic dispersing agent to produce stable 15 per cent aqueous dispersions

	N_2 Surface area (m^2/g)	Volatile (%)	Volatile concentration (g/m^2)	Dispersing agent requirement (% on black)	
				Experimental	Calculated*
Black Pearls 46 ..	720	14.9	.00021	22	23.3
Black Pearls 607 ..	593	16.3	.00028	6	14.5
Black Pearls 70 ..	545	10.1	.00019	15	19.5
Black Pearls 74 ..	320	5.1	.00016	12	13.6
Spheron 9	105	5.0	.00048	1.5	1.5
Vulcan 3	74	1.0	.00014	3.7	3.6
Sterling SO ..	42	0.8	.00019	1.5	1.5
Regal SRF	30	0.5	.00017	1.2	—

*Calculated DAR for black = DAR for Regal SRF $\times \frac{\text{surface area of black}}{\text{surface area of Regal SRF}} \times \frac{\text{volatile concentration of Regal SRF}}{\text{volatile concentration of black}}$

The influence of surface chemistry on the requirement of anionic dispersing agent to produce stable aqueous dispersions for a series of blacks is illustrated in Table 2. It has long been recognised that the oxidised surface groupings on carbon blacks act as natural dispersing agents in polar media and the present data demonstrate the roughly quantitative relationship that can be deduced. On the basis of the dispersing agent requirement of (say) Regal SRF, a first approximation of that for any other black may be calculated as shown. Deviations may be explained in terms of the actual constituents of the volatile, since the after-treated channel blacks in particular have a higher proportion of strongly acidic groupings which would be expected to exert a greater dispersing activity in comparison with other blacks. This aspect is discussed in more detail below, but, in particular, the unique properties of Black Pearls 607 are attributable to its having double the carboxylic-type acidity of Black Pearls 46.

Changes in viscosity or drying rate on storage of black dispersions are attributable to incomplete development of all the available surface during initial grinding, with subsequent adsorption of vehicle or dryers. Lower surface area blacks are obviously more viscosity stable and have less tendency to adsorb components. To offset loss of drying the inclusion of zinc or calcium naphthenate during the grind will minimise subsequent adsorption, or alternatively the use of " feeder " dryers is indicated.

Not all the surface available to the nitrogen molecule will be effective from the point of view of the effects noted above. Surface areas calculated from the adsorption of larger molecules may be more meaningful in this regard.

Vehicle demand and structure

The relation of surface area to oil absorption or vehicle demand in the case of channel blacks is illustrated in Fig. 3. As surface area increases oil absorption increases in the case of blacks of the same physical form (i.e. fluffy or pelletised) and the same approximate volatile content. Thus the upper left curve is that for unpelletised, low volatile (4-7 per cent) blacks. Pelletising causes a reduction in oil absorption, either due to a loss of structure or to the difficulty of achieving the same degree of dispersion, as in the case of fluffy blacks. The difference is more marked at the higher oil absorption end.

Oxidation of the surface to give volatiles in the order of 10-16 per cent has only a secondary effect on the oil absorption since it appears that the increased surface due to the formation of internal pores is not available to the oil. Thus the highly oxidised, unpelletised Moguls and Carbolacs lie on a lower curve than the low volatile blacks considered above. Once again the pelletised counterparts are still lower.

The oil furnace blacks are not shown on the same figure as the channel blacks since no such simple relationship exists, being complicated by varying degrees of structure. High structure blacks have a much higher oil absorption than their surface area would indicate. Thus Sterling SO with a surface area of 40 m^2/g has an oil absorption of the same level as a channel black of 200 m^2/g surface area.

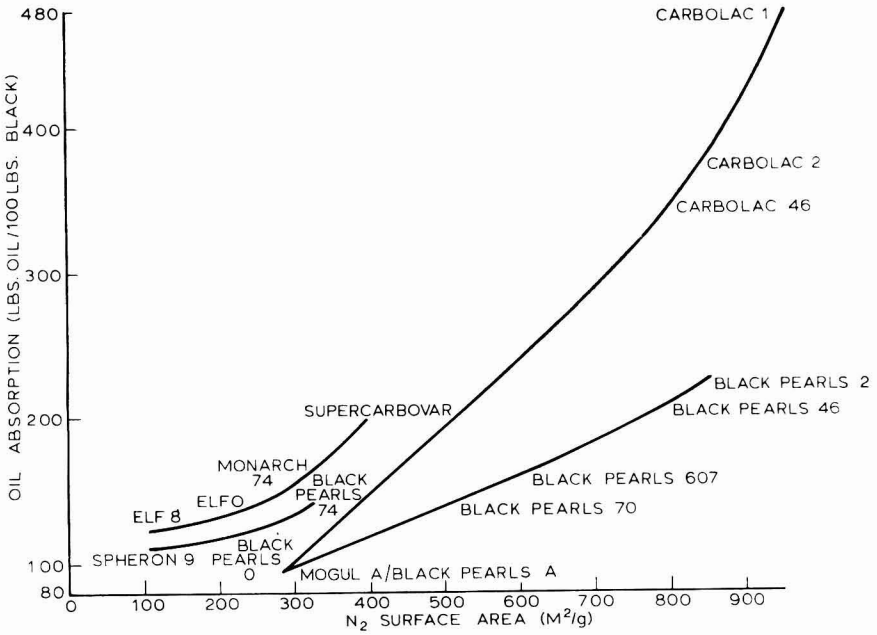


Fig. 3. Relation of oil absorption to surface area

In liquid dispersions such as inks and paints lower oil absorption carbons are often desirable. Originally high structure was thought to be inherent in the oil furnace process, but by injecting alkali metal salts into the furnace and thereby lowering the flame ionisation potential, structure was reduced to the same or even lower levels than that found in channel blacks. Thus the Regal series of blacks impart similar rheological properties to channel blacks of comparable particle size. This effect is achieved by virtue of a much lower oil absorption which counterbalances the influence of the higher volatile of the channel blacks. At the coarser end of the oil furnace black spectrum it has been possible to produce blacks similar to the gas furnace blacks.

There is some evidence to indicate that structure arises from the manner in which the graphitic crystallites are oriented in the surface of the carbon black particles. Regal blacks tend to have a slightly lower volatile content than higher structure oil furnace blacks of the same particle size. This indicates that they have a lower ratio of crystallite edges to crystallite planes lying in the surface.

Structure influences the behaviour of carbon black in several ways. Low structure blacks are more difficult to disperse than high structure blacks since more energy is required to achieve breakdown of agglomerates into discrete particles rather than into the clusters characteristic of structure. The apparent difficulty is compounded if comparisons are made at equal pigment loading, since the dispersion is being carried out at different viscosities and consequently

under different shear conditions. For adequate development of the properties of low structure blacks it is therefore necessary to increase the pigment loading during milling in comparison with that of a high structure black in inverse proportion to the respective oil absorptions.

Having achieved adequate dispersion, the colour strength and tinctorial power of low structure blacks is superior to higher structure blacks of equivalent particle size. As pointed out in a previous section, this arises from the somewhat narrower particle size distribution.

Two self-obvious effects of the low oil absorption levels of the Regal blacks at a given pigment/binder ratio in the final formulation are the low viscosity and good flow of the paint or ink, and the improved gloss of the finish because of the additional free vehicle.

Chemical nature of the carbon black surface

At those points on the surface of the carbon black particle where the edge of a graphitic crystallite is exposed, adsorption or chemisorption may take place. Most importantly in the present context is the chemisorption of oxygen or oxygen containing groups. These have been measured in the past by devolatilising at 950°C in a muffle furnace and recording the weight per cent loss as volatile content. More meaningful characterisation of these chemical groupings and their concentration is among the more important developments in carbon black technology.

The clean, devolatilised surface of any carbon black has a basic reaction in view of its aromatic nature. Thermal blacks have a volatile content of the order of 0.5 per cent and an aqueous slurry of these blacks has a pH of about 9.5. Oil furnace blacks produced with a limited amount of air have a volatile content of approximately 1 per cent and a pH of 8.5. On the other hand channel blacks as originally manufactured have 4-5 per cent volatile and give an aqueous pH of about 5.

To increase the flow of low colour channel blacks especially for use in lithographic inks, and to improve the dispersibility of high colour channel blacks, these are further after-treated by heating to red heat in air. This increases the volatile content to 10 per cent or more corresponding with a pH of 3.5 or lower. Air oxidation also increases porosity as measured by the nitrogen surface area.

Analysis of the volatile constituents of carbon black has been published by Rivin[2]. He postulated that oxidation took place stepwise from phenols or hydroquinones (weakly acidic), through quinones (neutral), to either lactones (neutral) or carboxylic groups (strongly acidic). Further oxidation of the carboxylic groups liberated CO_2 and caused porosity. Relative proportions of the various oxidised groupings for a range of carbon blacks are given in Table 3. The after-treated channel blacks are seen to have a comparatively high proportion of carboxylic groups. On the other hand, oil furnace black volatiles are mainly weakly acidic.

The importance of the oxygen functionality depends on the vehicle. In polar vehicles the oxygenated surface assists wetting and dispersion and consequently colour development. For a given particle size, increasing volatile gives lower

Table 3

Pigment	AM dia. (Å)	N_2 S/A (m^2/g)	Vol. (%)	C—OH phenols or hydro-quinones (me/g)	C=O quinones (me/g)	O ‖ C—OH car-boxylic (me/g)	O ‖ C—OR lactones or esters (me/g)
Black Pearls 46	130	720	14.5	1.90	2.55	0.43	0.05
Black Pearls 70	150	545	10.7	1.63	2.65	0.32	0.17
Black Pearls 71	160	380	4.9	0.89	0.49	0.06	0.12
Black Pearls A	280	299	12.6	1.30	2.29	0.28	0.24
Spheron 6 ..	250	112	6.9	0.78	1.21	0.16	0.08
Regal 600 ..	230	108	2.2	0.54	0.02	0.02	0.11
Vulcan 6 ..	230	114	2.5	0.56	0	0.02	0.16

viscosities and longer flows in general. As mentioned above relative to the dispersing agent requirement of different carbon blacks in aqueous dispersions, an increase in the concentration of oxygen on the carbon surface results in improved dispersibility and this is determined not merely by the volatile per unit surface area but also by the proportion of carboxylic groups. Air oxidised blacks have both a high volatile content and a high proportion of carboxylic groups, but this is partly offset by the increased surface due to porosity.

Table 4

	AM diameter (Å)	N_2 S/A (m^2/g)	Vol. (%)	Weak acids (me/g)	Strong acids (me/g)
Black Pearls 71 ..	160	380	4.9	0.89	0.06
Black Pearls 70 ..	150	545	10.1	1.63	0.32
Black Pearls 607 ..	130	593	16.3	1.33	0.86
Regal 330	250	82	0.8	0.37	0.02
Regal 400	250	96	2.0	0.80	0.05

To overcome this objection and allow increased surface acidity with minimum increase in porosity, methods of after-treatment other than thermal oxidation have been devised. An interesting comparison of the effects of these newer manufacturing techniques is concerned with the series of blacks tabulated in Table 4. Black Pearls 71 is a conventional medium colour channel black with a volatile content of 5 per cent. An air after-treated black of approximately the same particle size, Black Pearls 70, shows a 45 per cent increase in surface

area for a twofold increase in volatile. This net gain in oxygen concentration results in better viscosity stability of enamels based on Black Pearls 70 in comparison with Black Pearls 71. After-treatment by these newer techniques of a black of similar particle size yields Black Pearls 607, which shows only the same degree of porosity as Black Pearls 70 even though its volatile content is of the order of 16.5 per cent. In addition, the proportion of strongly acidic groups is higher. As a result of its surface chemistry, Black Pearls 607 gives colour of the same order and in many instances better than high colour blacks such as Black Pearls 46. Stability and gloss of enamels pigmented with this black is outstanding.

At the coarser end of the carbon black spectrum, oxidation of a low structure oil furnace black yields Regal 400 at the same surface area as Regal 330, but double the volatile content. The combination of moderate volatile and very low oil absorption imparts flow properties in letterpress and lithographic vehicles similar to the medium flow channel blacks air after-treated to 7.5 per cent volatile.

Optimising ball milling conditions

As carbon blacks have become more sophisticated the formulation of mill pastes to achieve the best performance from these newer pigments has become more critical. Part-for-part replacement of an existing black seldom achieves the desired result and it is therefore essential that the mill paste and the dispersing cycle be modified in line with the surface characteristics outlined earlier. As an indication of the order of differences to be expected from different blacks we have examined some of the variables in a ball milled alkyd/melamine baking enamel.

The procedure adopted was to take various resin solutions from 10 per cent to 40 per cent using a blend of resins in the same proportions as in the final formulation. Into these we milled the several blacks in the study at pigment loadings between $7\frac{1}{2}$ per cent and 20 per cent. The rate and degree of dispersion as measured by fineness-of-grind gauge and colour development was compared with the mill paste viscosity and pigment/binder ratio.

For simplicity, only the more significant results in relation to the effect of volatile and surface area on rheology and milling efficiency are presented.

In Fig. 4 mill paste viscosity expressed in Stormer Krebs Units is plotted against pigment loading in a 25 per cent resin solution and a 40 per cent resin solution for both Regal 330 and Regal 400. In a given vehicle the effect of increased volatile at the same surface area is to lower the viscosity of the mill paste. As pointed out earlier, Regal 400 requires a higher black loading in the mill paste to achieve the same degree of shear.

The 40 per cent resin solution results in a lower pigment/binder ratio than necessary for blacks of this surface area with the net result that grinding is slower. Furthermore at the 20 per cent black loading the mill paste viscosity is too high for efficient ball milling under our laboratory conditions, giving a very poor grind.

With the 25 per cent resin solution the best results were achieved at 20 per cent black loading (i.e. a pigment/binder ratio of 1 : 1), although, once again,

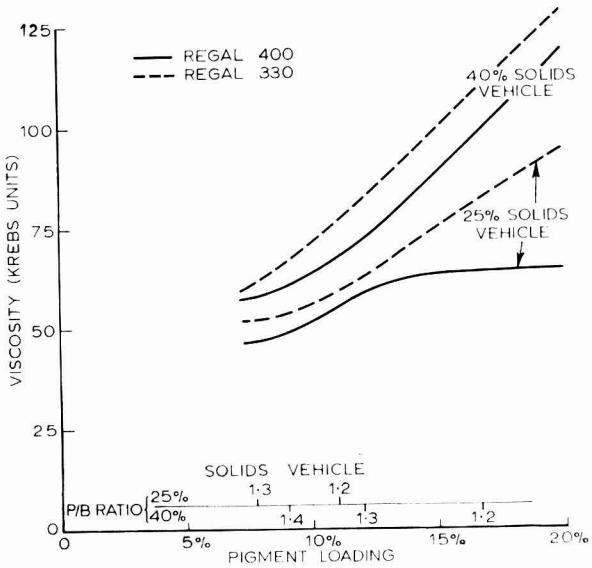


Fig. 4. Rheology of ball mill pastes of low colour blacks

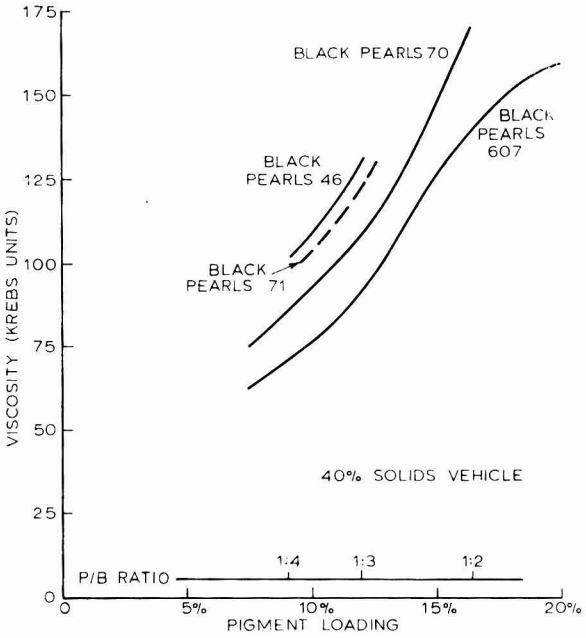


Fig. 5. Effect of surface chemistry on rheology of mill pastes

Regal 400 should be milled at a higher black loading in a more concentrated resin solution in order to increase the viscosity nearer to 90 KU without increasing the p/b ratio to more than 1 : 1.

At the higher colour end we have compared the rheology of mill pastes based on Black Pearls 71, Black Pearls 70, Black Pearls 607 and Black Pearls 46. This is seen in Fig. 5.

The relationship between black loading and viscosity in a 40 per cent solids vehicle for the low volatile, medium colour black, Black Pearls 71, is plotted for comparison of the effects produced by the after-treatment of the other three blacks. Black Pearls 46 is a much finer particle black and would be expected to give higher viscosities at equal loading, but its thermal oxidation has resulted in a higher volatile concentration which has offset the purely physical effect. At the same particle size as Black Pearls 71, the progressive increase in volatile concentration of Black Pearls 70 and Black Pearls 607 respectively, result in the lower viscosity curves for these two blacks.

At the surface area of Black Pearls 46, a pigment/binder ratio of 1 : $4\frac{1}{2}$ has been indicated in earlier work for this particular resin system. Combining this requirement of the black with the optimum viscosity requirement of our laboratory ball mill (of the order of 90 Krebs Units), we derived a mill paste formulation of 7.5 per cent of the high colour black in 92.5 per cent of a 40 per cent resin solution.

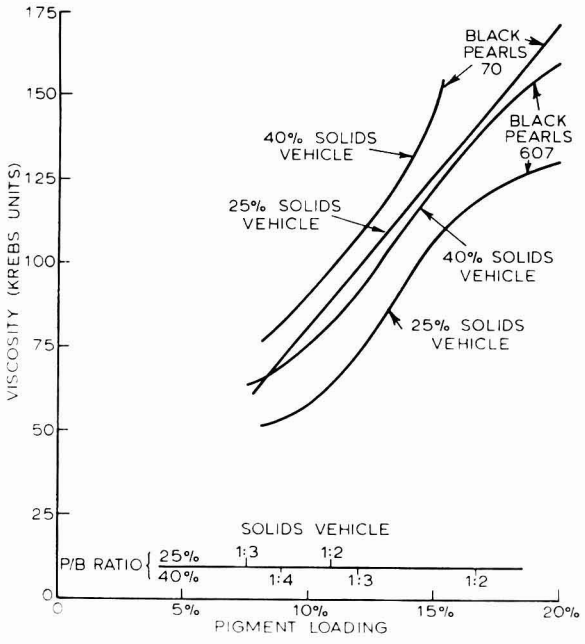


Fig. 6. Rheology of ball mill pastes of high colour blacks

The pigment/binder ratio required to stabilise the surface of Black Pearls 70 and Black Pearls 607 appears to be approximately 1 : 3½. In the 25 per cent solids vehicle this condition is satisfied at 6.6 per cent black loading, but the viscosity is too low for efficient ball milling. On the other hand, in the 40 per cent solids vehicle Black Pearls 70 at 10 per cent loading gives a suitable mill paste viscosity, whereas Black Pearls 607 is still low and a mill paste formulation of 11 per cent black in a 44 per cent solids vehicle is indicated.

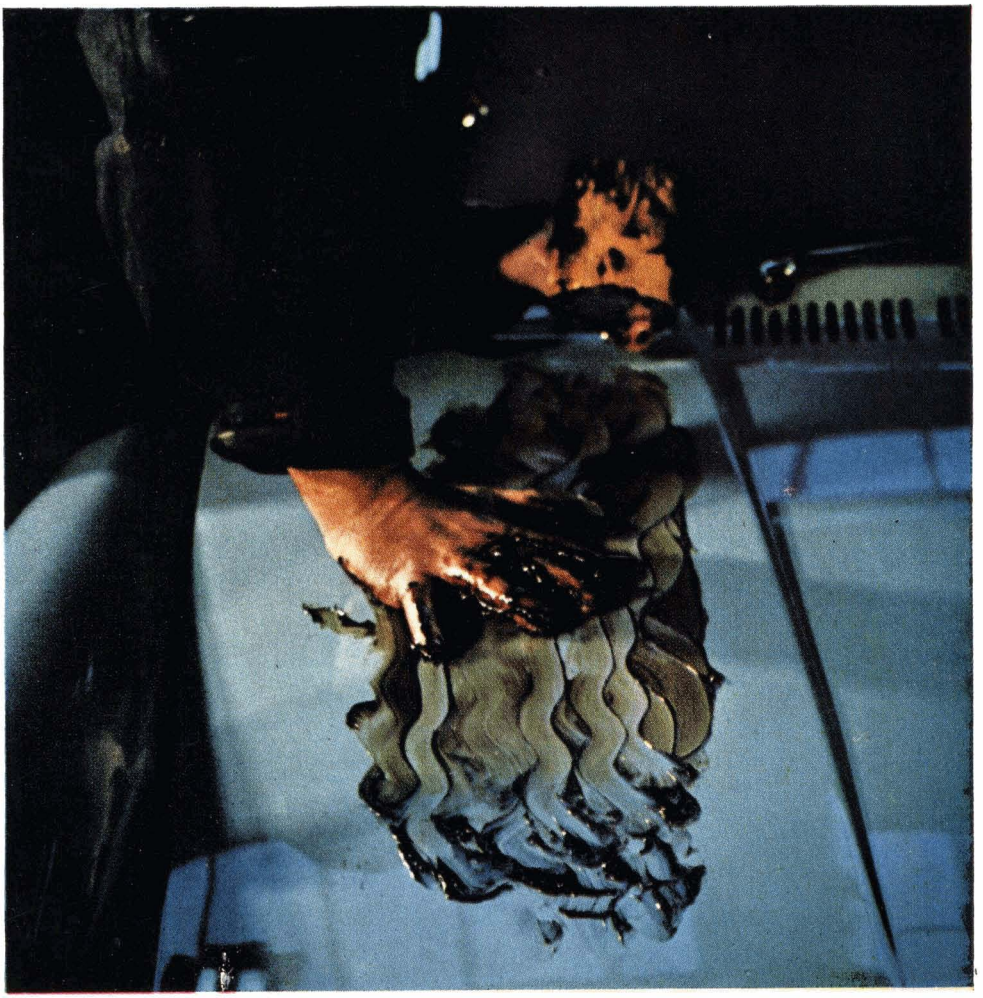
It must be pointed out that the above mill paste formulations are specific to the particular resins and dispersing equipment used in the study, but the general indications are that they may be adapted to individual conditions. Based on these principles, satisfactory enamels have been prepared in styrenated alkyds and thermosetting acrylics which are considered comparatively poor dispersing media for carbon blacks. The simplified procedure we used to derive the mill paste formulation in these systems was to fix the resin solids in relation to the black surface area (using the pigment/binder ratios indicated above as a first approximation) and to adjust the solvent to give a viscosity sufficiently high for maximum shear consistent with efficient cascading of the balls. In production mills this is achieved at higher viscosities than in the case of a small laboratory mill.

Excess binder cushions the balls resulting in longer grinding times than necessary. On the other hand, even at correct pigment/binder ratios, low vehicle solids, whilst giving faster grinds as judged by fineness gauge, do not allow the ultimate colour development that can be achieved at higher vehicle solids and often result in a brown undertone.

Contribution of the Vehicle

Table 5

Property	Significance
Particle size distribution	1. Weighted mean particle diameter determines jetness. 2. Skew to coarser particles imparts blue tone. 3. Indicative of structure.
Surface area	1. Determines energy required to achieve dispersion. 2. Ratio of nitrogen area to that calculated from EM diameter indicates porosity. 3. Fixes p/b ratio for optimum mill paste formulation. 4. Governs minimum requirement of protective colloid type dispersant.
Oil absorption	1. Proportional to superficial surface area. 2. Lower for pelleted blacks. 3. Higher for high structure blacks. 4. Affects viscosity of dispersions.
Surface acidity	1. Contributes to long flow and low viscosity in polar media. 2. Maintains stable, deflocculated dispersion. 3. Surface concentration of carboxylic groups affects requirement of ionic dispersant. 4. Improves colour.



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The main effects arising from the properties of the carbon black are summarised in Table 5. In relation to the chemical groupings on the surface of the black it is remarked that the polarity of the medium determines the influence these will have on the dispersion. Obviously this is an over-simplification of the interaction between pigment and vehicle and the rheology and colour of pigmented systems will be primarily influenced by the properties of the vehicle. At present there are no simple parameters which can be applied to all vehicles to characterise their pigment dispersing potential. Certain resin systems have been found to allow better colour developments with certain blacks, but further study is needed.

A practical indication of the influence of vehicle properties in a closely related series is given by the family of long flow channel blacks in different linseed stand oils as outlined in Table 6.

Table 6

				Nigrometer scale				Flow mm	
Pigment type	AM diameter (Å)	N_2 surface area (m^2/g)	Volatile (%)	8% in 5 poise stand oil	24% in 5 poise stand oil	25% in 35 poise stand oil	22% in 120 poise stand oil	25% in 35 poise stand oil	22% in 120 poise stand oil
Mogul ..	220	340	12	80	80	67	67	0	38
Mogul Plus	230	350	14	81	—	—	—	100	88
Mogul Special	240	360	14.5	82	—	—	—	135	106
Mogul A ..	280	295	12	84	84.5	74	76	31	98

Oil type	Acid value	Refractive index
5 poise stand oil	6.1	1.486
35 poise stand oil	14.9	1.491
120 poise stand oil	21.3	1.492

The Nigrometer Scale test is widely used as an index of potential jetness. Measurements are made on a dispersion prepared at approximately 8 per cent black in a 5 poise linseed stand oil. Under these conditions Mogul black with an arithmetic mean diameter of 220 Å has a Nigrometer Scale of 80 (0.35 per cent diffuse reflectance), whereas Mogul A with a diameter of 280 Å has a scale of 84 (0.40 per cent diffuse reflectance). Increasing the black loading to 24 per cent had no influence on the jetness rating although the higher loading resulted in a bluer toned ink.

The same pair of blacks were also tested in both a 35 poise linseed stand oil and a 120 poise linseed stand oil. Mogul black gave a Nigrometer Scale of 67 (0.23 per cent diffuse reflectance) and Mogul A a scale of 74 (0.28 per cent diffuse reflectance). Slight changes in the black loading were made to give approximately equal consistency. It has been observed previously that high refractive index vehicles tend to produce jetter inks[3], but the order of difference

between the 5 poise stand oil and the 35 poise oil would not in itself be expected to account for the colour difference found.

Comparative flow data illustrate the relative influence of physical and chemical properties of the pigment in different acid value varnishes. Higher volatile blacks gave the best flow properties in the 35 poise vehicle at an acid value of 14.9. It should be noted that this is *total* volatile content and not volatile concentration since Mogul A has a low absolute level, but the same concentration as Mogul Plus and Mogul Special in view of its comparatively low surface area. Thus in vehicles which are themselves slightly deficient in flow, the surface chemistry of the blacks is of major importance.

In the longer 120 poise varnish (acid value 21.3) the physical effect of the coarse particle size of Mogul A offset the influence of the higher volatile of Mogul Special to give the same approximate order of flow.

Other vehicles similarly will give different levels of jetness and rheological properties according to their characteristics. Thus varnishes based on isophthalic alkyds have sufficient length and good wettability to enable the formulation of good flowing inks with adequate strength from mildly after-treated oil furnace blacks similar to Regal 400 in comparison with conventional high volatile channel blacks.

Conclusions

Increased understanding of the nature of the carbon black surface enables one to explain and often modify the properties of dispersions since these are ultimately governed by the physical and chemical forces acting at the pigment vehicle interface. Further research is required to characterise adequately vehicles regarding their pigment dispersing characteristics, but within a given system the pigment properties determine the behaviour of the ink or paint.

Carbon blacks are now available with enhanced properties in certain vehicles and areas of application. Specifically, chemical modification of the black surface has produced comparatively low surface area blacks with a high concentration of acidic complexes giving improved dispersibility and stability. In the future it may be envisaged that other methods of chemical treatment will enable the production of carbon blacks tailored for systems which are at present considered difficult.

Utilisation of a carbon black to derive the optimum performance in a given application has been shown to involve the selection of a pigment with a balance of chemical and physical properties appropriate to the vehicle, and its dispersion under the optimum conditions for the dispersing equipment and with at least sufficient vehicle or dispersant for stability. From experience it is clear that in many instances carbon blacks are being wastefully used at the moment due to inattention to these principles. In addition, laboratory evaluation of new carbon blacks is often misleading if based on hand rub-ups, or even dispersions prepared on an automatic muller. Properly milled finishes with pigment loadings adjusted to give good shear are necessary for adequate assessment. Due consideration of these principles will ensure that the carbon black user derives the greatest potential from this pigment.

Acknowledgements

The author wishes to express his appreciation to Messrs. J. Wadelin and L. Doppler for the experimental work and to Messrs. Cabot Carbon Limited for permission to publish this paper.

[*Received 21 March 1966*

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A gel strength tester for thixotropic materials

By C. J. H. Monk and L. S. Hartridge

Imperial Chemical Industries Ltd., Paints Division, Slough, Bucks.

Summary
The paper describes a gel strength tester which is a modification of the standard ICI Rotothinner. With the new instrument a thin steel blade is lowered edgewise into the sample, causing practically no disturbance to the gel structure. The blade is then rotated and a measurement is made of the force required to break the gel. Since the sample can be conditioned and tested in the same standard half-pint can, no time is wasted in conditioning the sample in the apparatus and each complete determination takes only a few minutes.

Un dispositif pour mesurer la rigidité de gélification des matériaux thixotropiques

Résumé
Cet article décrit une modification au modèle normal du " Rotothinner " de l'I.C.I. Au moyen de l'instrument nouveau on peut éviter, au commencement de l'essai, tout dérangement de la structure du gel, grâce à une lame mince en acier que l'on fait descendre sur le bord dans l'échantillon. Ensuite on fait tourner la lame afin de mesurer la force nécessaire à rompre le gel. Puisque l'on peut également conditionner et essayer l'échantillon dans le même bidon normal de demie-pinte, il n'y a pas de perte de temps en conditionnant l'échantillon dans l'appareil et chaque essai n'occupe que quelques minutes.

Ein Prüfgerät zur Bestimmung des Gel-Grades thixotroper Stoffe

Zusammenfassung
In dieser Arbeit wird ein Prüfgerät zur Bestimmung des Gel- Grades beschrieben, das aus einem modifizierten Standard ICI Rotothinner besteht. Bei dem neuen Instrument wird eine dünne Stahlklinge hochkant in das Muster eingeführt, wodurch die Gelstruktur in keiner Weise gestört wird. Die Klinge wird daraufhin rotiert, und die Kraft gemessen, die für die Zerstörung des Gels erforderlich ist. Da die Probe im gleichen, 284 ccm fassenden Gebinde sowohl in den Ruhezustand gebracht, als auch gemessen werden kann, wird keinerlei Zeit mit der Konditionierung derselben im Prüfgerät verschwendet, sodass jede Bestimmung lediglich einige wenige Minuten in Anspruch nimmt.

Прибор для испытания устойчивости гели тиксотропных материалов

Резюме
В статье дано описание прибора для испытания устойчивости гели, который представляет из себя видоизмененный стандартный Рото-разбавитель типа I.C.I. В этом новом инструменте тонкое стальное лезвие погружается острым краем в образец, без заметного возмущения структуры гели. Затем лезвие поворачивается и измеряется сила затраченная на разрушение гели. Так как можно приготовить и испытать образцы в одном и том же стандартном полупинтовом сосуде то не теряется времени на приготовление образца в аппарате и каждое полное испытание выполняется в течение нескольких минут.

Introduction

Continuing interest in thixotropic paints has made it desirable to develop a rapid and simple method of measuring the gel strength of these paints and of

the resins from which they are made. Although the gels are normally strong enough to allow fairly easy measurement with simple equipment, they may take up to several days to regain their full strength after having been disturbed. For this reason a number of orthodox viscometers which would otherwise have been suitable were regarded as impracticable since the disturbance caused by filling them would destroy the gel structure and result in the occupation of the viscometer for long periods while the material recovered. This consideration and the possibility that the test would be required in raw material specifications and manufacturing control suggested the modification to the ICI Rotothinner[1] that is described in this paper.

Apparatus for gel strength measurement

Gel strength can be measured by most coaxial cylinder viscometers such as the Ferranti portable, Brookfield and Rheomat 15 but, as mentioned above, the necessity to allow the gel structure to build up in the viscometer cylinder before a reading can be taken, considerably limits the speed with which results can be obtained.

The Gallenkamp torsional viscometer has been used for this purpose but its suitability is again limited by the fact that it is necessary to allow the gel to form in the viscometer container.

The Stormer viscometer[3] is an instrument which can be used for gel strength measurement. This instrument has a flat blade type of rotor that can be lowered into the specimen without destroying the gel structure. The torque on the rotating blade is normally applied by means of a weight and pulley system but a gradually increasing torque could be applied by pouring water or lead shot into a container until the gel structure breaks. The operation of this instrument is not, however, as simple as that of the Rotothinner and the end point is not very well defined.

The Rotovisko viscometer has been used to measure gel strength[2]; here a six bladed rotor has been fitted that can be lowered into the sample without causing much disturbance to the structure.

Another type of instrument, for gel strength measurement, the Bloom Gelometer, is described by Gardner and Sward[3]. Here a flat plate is placed on the surface of the test sample and a gradually increasing downward force is applied by means of an increasing load of lead shot. When sufficient shot has been applied the gel breaks and the plate passes into the sample. The main disadvantages of a procedure that involves penetration through the surface lie in the fact that any skinning or toughness of the surface of the material can considerably affect the results and in many cases the point of rupture of a slowly deforming surface is only approximately defined.

The apparatus that was finally adopted by us was a modification of the Rotothinner (see fig. 1) in which the ordinary rotor was replaced by a two bladed paddle, the normal motor by a 2 rpm motor of the same type, and the scale engraved in g. cm torque. This has the advantage over any of the other instruments described above in that it is very much better suited to routine

measurement. It is robust, relatively inexpensive, easily available, and easy to use and clean. The use of a two-bladed rotor which can be lowered into the sample with negligible disturbance of the gel, and the use of the same standard half-pint can for storing, conditioning and testing each sample, reduces the amount of cleaning necessary and avoids the need for ageing the sample in the instrument. A single complete determination takes only a few minutes.

Description of the instrument

The instrument consists of a standard Rotothinner stand with a low speed motor, special paddle and the rotating table calibrated in g. cm torque (see fig. 1).

The motor used is a Drayton type RQH as normally used on Rotothinners but with an output shaft speed of 2 rpm. The paddle consists of a stainless steel blade 4 cm $\times$ 2 cm $\times$ 0.5 mm fixed to a $\frac{1}{4}$ in spindle in the form of a double flag. The spindle is of such a length that, when the paddle is lowered into the test sample in a half-pint can, the lower edge of the blade is 1 cm from the bottom of the can. The turntable of the Rotothinner is standard with the exception that the scale is engraved in g. cm instead of poises. The full scale covers a range from 0-450 g. cm.

Consideration was given to the possibility of calibrating the instrument in absolute units (dynes/cm^2), but this idea was abandoned in view of the difficulty of defining precisely the surface at which the gel breaks down during test. It was decided that it would be better to express the results as the torque in g. cm required to break down the gel under the arbitrary conditions of the test.

Method of operation of the instrument

The sample for test is stored or conditioned in a standard half-pint can (7.8 cm dia., 8.2 cm deep), which is filled to within 3 cm of the top ; less than this amount of material will give erroneous results. The can is placed directly on the measuring table of the gel tester. Then, with the motor switched off, the paddle is lowered gently into the sample by means of the lever until it is in its lowest position. The motor is switched on and the table rotates slowly until the gel structure breaks. A note is made of the maximum reading in g. cm indicated on the table and this figure is the torque required to break the gel.

Accuracy and reproducibility of measurement

The accuracy of the instrument can be best exemplified by use of data collected during the calibration of three new instruments against the original prototype.

(*i*) Accuracy was checked by applying known loads and comparing applied torque with scale reading. Calculated and observed values agreed to $\pm$ one scale division (5 g. cm).

(*ii*) Gel strength determinations were carried out on five materials varying in gel strength from about 50 g. cm to 300 g. cm. The results obtained were compared with those obtained with the prototype instrument (No. 6317). The results are given in Table 1.

Fig. 1. The gel strength tester

Table I

Test sample		Age of sample	Instrument No.				Range (g. cm)	Mean (g. cm)	Max. divergence (g. cm)
No.	Type	(days)	6317	6345	6346	6347			
			Gel strength (g. cm)						
1	Paint A	2	49.5 50 49.5	50 51 53	51 50 50	58 53 55	8.5	51.7	6.3
2	Paint B	7	83 83	76 75	82 75	80 80	8.0	79.3	4.3
3	Resin A	2	297.5	308	320	314	22.5	309.9	12.4
4	Resin A	7	340	344	355	350	15.0	347.3	7.8
5	Resin B	7	195	173	180	187	22.0	183.8	11.3

The results suggest that with due attention to preparation of sample, temperature of storage and temperature at time of test, agreement within ± 15 g. cm can be expected.

Acknowledgements

Acknowledgement is made to Mr. N. D. P. Smith who first suggested that the Rotothinner could be modified for this purpose and to Mr. G. Lamb who carried out the constructional work ; also acknowledgement is made to Mr. J. F. Cull and Mr. A. Sarkissian for their work in evaluating the first models.

[*Received 21 March 1966*

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Editorial

" Ram thy fruitful tidings in mine ears "

Shakespeare probably had a point—tidings, however fruitful, must often be rammed home before they are perceived. No doubt this explains the rash of recent books on English as applied to science and technology inviting, cajoling and instructing the technical man to write better reports.

The quotation which introduces a recent book* provides an interesting foil for that we have chosen.

" Except ye utter . . . words to be understood, how shall it be known what is spoken ? "—*I Cor., 14 : 9.*

Did one but heed Corinthians, there would be no need of Shakespeare's exhortation —but he was a realist.

No technical effort is of value until its result has been communicated, and the essential features of good communication, which ensure comprehensibility, are simplicity and brevity. There is nothing difficult to grasp in this, so we can only deplore a situation in which the technical man is allowed to qualify in his chosen discipline, or to attain even such mean eminence that his reports are of interest to anyone but himself, without having first mastered the essentials of his mother tongue. It would be no more absurd to allow a doctor several years in general practice before teaching him how to write a prescription.

**Better Report Writing*, by Willis H. Waldo. 2nd Ed., New York : Reinhold Publishing Corporation, 1965. Pp. vii + 276. Price $10.00.

Reviews

CYCLOKAUTSCHUKLACKE

By W. KÖNIG. Colomb, Stuttgart: 1966. Pp. 218. Price DM 45.-.

These publishers have added with the above attractively bound book a further volume to their monographs " Lacktechnische Schriften," which are edited by Hans Kittel. The book appears to be a summary of work done by W. König and others at Chemische Werke Albert, well known for this line of resin. Practically all the relevant literature has also been included.

The main headings of the minutely sub-titled text are :

Chemistry and manufacture.
Chemical and physical properties.
Behaviour in a medium.
Application in surface coatings.
Formulations.
Methods of application.
Testing of paints, generally.
A critical review of anticorrosive protection by paints.
Special tests of their anticorrosive efficacy.
Use in inks and for other purposes.

The author is master of his subject and presents all important aspects well, with 235 footnotes relating to literature and patent references. The reading

of the main sections is profitable and also of what he says about corrosion. For a paint chemist, however, the work contains much too much elementary and many times documented matter, such as general testing, which one would not look for in a book on a particular resin. Printing ink chemists would probably like to find more information on their aspects.

While I cannot agree with every detail W. König has written, I find his treatment of the subject of cyclised rubber informative, excellent and exhaustive, and for this alone his book is worth having.

M. HESS.

POLYURETHANES

By BERNARD A. DOMBROW. Reinhold Publishing Corporation, New York, 1965. Pp. 240. Price $11.50.

During the eight years that have elapsed since the first edition of this book was published there have been many important developments in polyurethane chemicals so that this revision which deals with most of them is well timed and fully justified. The adoption by the using industries of these polyurethane chemicals has been very rapid and data on this aspect is given in the introduction. The author is to be congratulated on a well designed and written book which is easy to read and understand. There are included in the book ten chapters dealing with specific application aspects. Pride of place here is given to the longest chapter which deals with rigid foams and this is indicative of the importance that this type of foam has achieved in recent years.

In this chapter the introduction of fluorocarbons as blowing agents and their value as against water for this purpose is considered as well as the nature of the resin (polyol, polyester etc.), the effect of additives and type of isocyanate used. The move from prepolymers to crude TDI and crude MDI to give one-shot processes is dealt with but there is scant acknowledgement of the work done in the UK with the latter product for this purpose. Neither is there any attempt to discuss the relative merits of these two " crude " isocyanates for the formation of rigid foams save to record, in an excellent section on flammability of such foams, that crude methylene bis (phenyl isocyanates) are superior to tolylenediisocyanates in this respect. The use of the adjective " free " to describe isocyanates which have not been reacted with other chemicals is, perhaps, unfortunate and leads to the following : " Free tolylenediisocyanate has the best cost potential . . . " This would seem to be self evident even when left in its context.

The chapter on Flexible Foams deals with both polyester and polyether foams and their respective uses. It is now clear that both types are needed ; the polyester foams particularly for foam backing of fabrics etc., and polyether foams in comfort cushioning etc. This chapter contains an excellent table on " Molding Defects " giving possible causes and suggested remedies. It also deals at some length with a description of a block-making machine using the high pressure system but no reference is made to the alternate low pressure system which now seems to be finding favour even in the USA. A criticism of this book is that not enough is said about foaming equipment generally and particularly for rigid foams. References are given where such details can be found but it is certainly

true that polyurethane technology depends almost as much on engineering as on chemistry and one would have thought that the present edition would have been more useful if this aspect had been given more attention.

The chapters on rubbers and adhesives are very helpful and it is interesting to note that in both MDI is proving increasingly useful. It is somewhat surprising so much attention is given to "Vulcaprene A" when elsewhere so little is said of UK developments. The chapter on " Coatings " is satisfactory so far as it goes but in this revision one would have expected some mention of the very light-fast and very durable effects that can be obtained with the Desmodur N type of isocyanate as well as the rapid drying properties and improved light-fastness obtainable from products of the polyisocyanurate type (exemplified by Suprasec 3240) and the extreme chemical, solvent and water resistance obtainable by the use of specially designed polyethers (such as Daltolac 2190) cured with MDI and deliberately added water.

The chapter on textile applications deals entirely with the development of polyurethanes as textile fibres, the original target for the development of urethane chemicals. Success in the hard fibre field has not been outstanding but development in elastic fibres (spandex) is much more promising and such products are making significant inroads in the foundation garment and allied industries. The Miscellaneous chapter deals with encapsulation techniques and leather tanning.

This book should prove valuable to any technologist concerned with polyurethanes.

H. A. HAMPTON.

Introduction to Colloid and Surface Chemistry

By D. J. Shaw. London : Butterworths. 1966. Pp. ix + 186. Limp. Price £1 12s. 6d.

This book is intended to bridge the gap between the treatments accorded the subject in text books of physical chemistry and in the specialised treatises. It is aimed at a wide readership comprising undergraduate students in universities and colleges of technology, those commencing post-graduate research, industrial scientists and others desiring a broad background understanding of colloid and surface phenomena. The author suggests that academic courses tend to neglect colloid chemistry because it cannot be treated with the rigour of some other branches of science. He will not find universal support for this contention, nor is it likely that many undergraduate students of pure science will want to spend time and money in concentrating on a subject which, however important it may be, constitutes but a small part of the degree course.

The treatment purports to be fundamental though only a few theoretical derivations of equations are given. On the other hand, descriptive matter is treated very fully and many practical procedures are described, so that the subject comes to life and is seen as one of widespread interest and appeal. This is in marked contrast to the rather arid chapter to be found in many text books. The book will be especially valuable to those studying for qualifications with a technological bias. Few paint technologists could fail to benefit from it.

M. J. ZISSELL

The Separation of Biological Materials

British Medical Bulletin, Vol. 22, No. 2. May 1966. Pp. 103-194. Price £1 10s.

The September 1954 issue of the *British Medical Bulletin* was devoted entirely to chromatography and this can almost be said of the present issue. It is not surprising that, with such a main theme, more than half the 15 review articles on non-destructive methods of separation should be concerned essentially with chromatography of various kinds. Three papers describe ultracentrifuge, electrophoretic and liquid-liquid extraction procedures, whilst the remainder are devoted to reviews of the newer techniques applicable to specific classes of materials such as lipids, proteins and sub-cellular particles.

The papers of particular interest to readers of this *Journal* will be those of Ogston on the physical chemistry of porous systems, Andrews on molecular sieve chromatography, Scott on gas-liquid chromatography, and Nichols *et al.* on the separation of lipids. The other papers make none the less fascinating reading and much of value is to be found in them, though some readers would no doubt have preferred a more rigorous or mathematical approach in one or two cases.

It is disappointing to find clearly emphasised in this collection of papers the lack of agreement on nomenclature which still lends confusion to the literature of chromatography. Some years have elapsed since chromatographers agreed to abandon the term " vapour phase chromatography " in favour of " gas chromatography " but the proliferation of near-synonyms which they have since been at some pains to avoid is again to be found in "gel filtration," "gel permeation," " gel chromatography," " molecular sieve chromatography " and " exclusion chromatography."

The authors are to be commended for their inclusion of numerous references to very recent work ; indeed a futuristic touch is to be found in a reference dated 1967 (in the press). This is a clearly printed, well produced issue, but it is a paper-backed journal and the cost of binding in stiff covers would be amply justified by the heavy usage it undoubtedly deserves.

D. GRIME

The Chemistry of Polymerisation Processes

Monograph No. 20. London : Society of Chemical Industry 1966. Pp. v + 360 Price £4 5s.

This is a bound volume of the papers presented at the April 1965 symposium held by the Plastics and Polymer Group of the SCI. The list of contributors reads like a polymer scientist's " Who's Who " and, since all the papers describe original work, the general impression is of a scientific journal rather than a text book.

The papers, 23 in number, may for the most part be classified according to the nature of the polymerisation process. It is impossible to review them all and difficult to select those of particular general interest. The coatings chemist may be less attracted to the section on stereospecific polymerisation because of the low solubility associated with stereoregularity though A. J. Burgess and R. O. Colclough provoke speculation in their paper on the stereospecific

polymerisation of epoxides. Few companies in the coatings business actually conduct ionic polymerisation processes and readers of this *Journal* are likely to be attracted mainly by the sections on free-radical and condensation polymerisation.

M. Gordon *et al.* present an interesting picture of the melamine-formaldehyde reaction, showing clearly the separability of the methylolation and resinification stages. Some unusual routes to polyurethane foams are described by C. R. Thomas, whilst W. Gerrard adds another chapter to the chemistry of semi-organic polymers. Several papers cover the kinetics of free-radical polymerisation and copolymerisation. R. J. Ceresa demonstrates the ease with which graft copolymers can be produced by using low molecular weight resins such as coumarone-indene, shellac and poly(vinyl acetals) as efficient transfer agents.

This book is part of the original literature and must find a place in any polymer science library. It is less likely to be purchased, though it may profitably be perused, by those whose interests are less specialised.

A. R. H. TAWN

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.)*

Amongst this year's winners under the BRITISH SAFETY COUNCIL's Award Scheme are Sandoz Products Ltd., John E. Sturge Ltd. and Winstones Ltd.

ICI DYESTUFFS DIVISION has announced the addition of two new pigments to their range. These are Monolite Fast Yellow 6GS, a high strength green-yellow organic pigment which the company says will be of interest to all manufacturers of industrial finishes, including vehicle finishes, and Rubine Toner BOS, which provides yellowish-red shades, especially suitable for paints for toys, meeting the recommendations for BS 3443, 1961.

ICI NOBEL DIVISION is to build a major extension to its pentaerythritol plant at Dumfries. The extension, which will be completed early in 1968, will make the Dumfries unit the largest pentaerythritol plant in Europe and the third largest in the world. Existing capacity will be increased by 5,000 tons a year.

BERK LTD. has assumed full responsibility for all sales of "Upol" urethane prepolymers manufactured by RDT Chemicals Ltd., Boston Spa, Yorkshire.

CIBA CLAYTON LTD. has announced the availability of a new quality of their product Microlith White R-K for use in vinyl media. They claim that this new quality gives improved whites, appreciably superior gloss and somewhat superior dispersion characteristics when compared with the previously marketed manufacture.

PINCHIN JOHNSON AND ASSOCIATES LTD., the Courtaulds paints subsidiary, has announced that it has concluded an agreement with Denofa og Lilleborg Fabriker for the manufacture in Norway of Red Hand compositions and marine paints.

HOECHST UK LTD. has established a purchasing and export department to centralise buying in this country of UK-produced materials by Farbwerke Hoechst AG and its subsidiaries in Germany.

LAPORTE CHEMICALS LTD. has recently developed an MEKP of reduced fire hazard rating, but still retaining the very high activity associated with the RGS grade.

In collaboration with the North of Scotland College of Agriculture, FARQUHAR AND GILL LTD. has developed a new plastic type paint which they claim prevents corrosion to silos caused by acids in silage.

BADISCHE ANILIN- UND SODA-FABRIK AG has issued coloured folders describing their Heliogen, Fanal and Lithol pigments. These folders give information concerning the properties and application of these products. The issue of further folders is planned.

Scottish

Eastern Branch

The fourth meeting of this session was a lecture on " Micronised pigments in paint manufacture " given by Mr. W. G. Wade and held on 26 January 1966 in the North British Hotel, Edinburgh.

The first part of the lecture described the principles, and the operation of the process involved in the micronising of pigments. Mr. Wade gave an account of the uses of the resulting product in the paint industry, with particular reference to the very high outputs of decorative paints possible when micronised pigments were used along with modern high-speed impeller machines.

In the second part of the lecture the limitations of the micronising process as applied to pigments were detailed and some of the anomalous properties which resulted were explained. The possible uses in printing inks were mentioned and finally, the trend of future developments in this field was indicated.

There followed a full discussion amongst Mr. Wade and those attending which completed this interesting evening.

P. A. G.

West Riding

Final Technical Meeting

The final technical meeting of the present session was held on Tuesday 8 March 1966, at the Great Northern Hotel, Leeds, when Mr. F. G. Dunkley of the Protective Coatings Laboratory, British Railways Board, Research Dept., Derby, presented a paper with the imposing title " The exploitation of new methods of paint application and the examination of their potential advantages and the development of paints designed to exploit them."

The speaker began by stating that with the development of newer types of polymer coatings the old limitations of film thickness were being overcome and gave rise to a wide range of methods of application. Brief descriptions were given of the following methods : electrostatic, ultrasonic, curtain-coating, fluidised-bed, dip coating and strip coating, electrostatic powder spray and flame spray techniques. The speaker then came to the nub of his paper—airless spray as applied to the finishing and refinishing of railway rolling stock.

The old method involved the application of seven coats of paint, the last three being varnish, over ten days and took 63 man-hours per carriage.

Using airless spray this system is reduced to three coats, three man-hours per coat per carriage, and the whole job is finished in one day. The system is also inherently able to overcome " fogging ".

To apply the system in an open shop there are two prime considerations—the fire risk and the toxicity. These limit the choice of resin as the preferred thinner is white spirit, the paint must go wet on wet to maintain speed of throughput, and the finish must have good gloss to maintain the finish reached with three varnish coats in the old specification. The base resin chosen was therefore a vinyl toluenated alkyd.

To conclude the lecture numerous slides were projected showing airless spray equipment, views of finished carriages, and a variety of illustrations of techniques and equipment in use in Switzerland, Germany and the UK.

Many questions were asked by the members, including Mr. N. Cochrane, Mr. D. Morris, Mr. H. Young, Mr. D. Gray, Mr. C. Butler and Mr. M. Cochrane.

There was an extremely good turn-out for the lecture—40 members and guests being present—and Mr. Dennis Gray spoke for all present when he proposed the vote of thanks which was heartily endorsed.

J. N. MCK.

Mr. N. Cochrane, Chairman West Riding Section, is shown with Mr. Quealy Walker (centre) and Mr. L. H. Silver, Vice-Chairman West Riding Section

Visit of Mr. W. Quealy Walker

Mr. W. Quealy Walker, president of the Dixie Paint and Varnish Co. Inc. of Brunswick, Georgia USA spoke at a luncheon meeting of the West Riding OCCA recently on " Problems of the Smaller American Paint Company."

Mr. Walker who was in England on a visit to his daughter, flew to Leeds from London for the meeting, returning the following day.

He spoke at some length on the highly competitive nature of present trading conditions in the USA.

He quoted examples of stunts the American paintmakers have to employ, to encourage the order from the customer. It was emphasised that the 2,000 American paint companies (in the opinion of the speaker) worked a lot harder selling to their established users and buyers than do their British counterparts. They subsequently delivered smaller quantities to retailers more frequently then did the UK paint maker.

Mr. Walker brought with him a comprehensive display of " give-away " material, currently in use in America.

Mr. Walker, who founded the Dixie Paint and Varnish Co. Inc. in 1940, and who is a trained architect by profession, produced his first batch of paint in a copper clothes boiler, borrowed from his mother-in-law—a point much emphasised in his company's " hand-outs." His company today is considered to have one of the most attractively designed buildings and modern plant lay-out in the United States, producing over 1½ million US gallons per year.

OCCA Nineteenth Technical Exhibition 1967

As announced in the June issue of the *Journal*, OCCA 19 (the Association's Nineteenth Technical Exhibition) will take place at Alexandra Palace, London, on the following dates and times :

Monday 13 March
3.00 p.m.—**6.30** p.m.

Tuesday 14 March
10.00 a.m.—6.00 p.m.

Wednesday 15 March
10.00 a.m.—6.00 p.m.

Thursday 16 March
10.00 a.m.—6.00 p.m.

Friday 17 March
10.00 a.m.—**4.00** p.m.

Forms for applications for stand space were despatched to companies at the end of May, and a large number of completed forms has already been returned. Those companies intending to exhibit must send in their completed forms of application not later than **Friday 2 September 1966.**

There will be an Exhibition Luncheon at the Savoy Hotel, London, on Monday 13 March. The forms of application for tickets for the Luncheon will be included in each copy of the *Official Guide*, which will be sent to all members of the Association early in 1967. The *Official Guide* will also be sent, as far as possible, to all consuming firms in Britain and individually to chemists and technologists in the paint and allied industries in Western Europe. They can also be obtained by visitors to the Exhibition without charge; admission to the Exhibition will also be free.

It is believed that the Exhibition is unique in that it is entirely a technical one, aimed at ensuring that technical advances are passed on as quickly as possible to the technical personnel within the paint, printing ink, linoleum and allied industries. The technical advances may relate to new products, new knowledge of existing products, and their uses, or, in suitable cases, existing knowledge which has not been available to the consuming industries.

The standard shell scheme for the 1967 Exhibition will include felt covering to the facias, which will be trimmed with British Columbian pine ; the colour of the felt on the facias in the north-south corridors will be light green and, in the east-west corridors, Minorca blue. The standard shell scheme wall and floor covering, both of which may be altered by exhibitors, will be in grey. The standard facia name plaques will be painted in black letters on a white background.

The Exhibition is not confined solely to British firms, and Continental companies wishing to be considered for stand space, and any companies in the United Kingdom who have not previously exhibited and who would like to have their names submitted to the Committee for consideration should write to R. H. Hamblin, General Secretary, Oil and Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London E.C.2. (MONarch 1439, Ext. 3).

Conference on advances in polymer science and technology

The second Conference to be organised jointly by the Plastics and Polymer Group of the Society of Chemical Industry, the Plastics Institute and the Institution of the Rubber Industry will be held at the Institution of Electrical Engineers, Victoria Embankment, London, W.C.2, on 20, 21 and 22 September 1966. The Oil and Colour Chemists' Association and the Society of Dyers and Colourists will also participate on this occasion. The purpose of the Conference is to provide a forum for discussion between scientists and technologists whose work is concerned with polymers. Papers will be preprinted and presented in summary form at the Conference. Ample time will be given for questions and discussion.

The papers will cover the following fields :

The Structure of Polymeric Systems.

Polymerisation Reactions, particularly polymerisation and cross-linking reactions in films—interaction with substrates and adhesion.

Physical and Chemical Changes in Polymers with particular reference to long-term ageing and load-bearing properties.

Practical Implications of Rheological Measurements.

Fuller details and application forms to attend can be obtained from the Assistant Secretary, Society of Chemical Industry, 14 Belgrave Square, London, S.W.1. Please mark your envelope " Joint Plastics Conference."

Hull Section

Visit of Members to Laporte Titanium Limited

The final ordinary meeting of the Hull section of OCCA took the form of a joint visit, with the Hull Chemical and Engineering Society, to the Stallingborough factory of Laporte Titanium Ltd.

After a short voyage by paddle-steamer across the Humber, the party of 26 was transported by coach the 20 miles or so to Stallingborough where they were welcomed to the plant by Dr. A. R. Pinnington who outlined the scope of the Laporte Group's interests and by Mr. S. Beaver who described the organisation of their Research sections. After being neatly divided into groups, the party visited six laboratory sections which combined to deal with everything from pigment manufacturing techniques and basic research to customer service work. Instruments and methods seen being used included electron microscopes, X-ray fluorescent spectroscopy, spectrophotometers, gas/liquid chromatography, a cathode ray polarograph and the ingenious mercury displacement technique for measuring the density of paint films for use in scatter measurement calculations. Equipment used for customer service work included high speed dispersion mills of both impeller and sand milling types, electrodeposition apparatus, machinery for processing plastics and paper, and accelerated exposure testing machines of both the xenon discharge and carbon arc types. The staff in these laboratories outlined the objectives of various projects currently being carried out and the results of their work relating to the paint and ink industries.

Following an excellent lunch, the party was again subdivided and, suitably

The party from Hull Section is shown outside Laporte Titanium Ltd.'s factory

attired, set forth on a tour of the manufacturing process. Starting from the " black end " where the ilmenite arrives by steamer, the processes of milling, sulphation, purification, hydrolysis, filtering, washing and further purification, calcination, surface treatment, final milling and packaging were observed. The party duly emerged from the " white end " of the plant into glorious sunshine, convinced that the superficially simple task of extracting the Titanium Oxide from the ore engaged the application of many specialised skills.

Over refreshments, Mr. L. W. Wynn (Chairman, Hull OCCA) and Mr. Bellamy (on behalf of the Hull Chemical and Engineering Society) expressed the thanks of the visitors for the hospitality extended to them and the very interesting and well organised tour of the laboratories and factory.

London Section

Thames Valley Branch

Visit to factory of Metal Box Company

On the afternoon of Tuesday 10 May a party of 12 members of the Branch visited the Palmers Green, London, factory of the Metal Box Company. This factory produces a large variety of tinplate containers for solid and liquid products, all decorated by printing.

The sequence of operations involved in translating a customer's description or sketch into sets of copper on stainless steel lithographing plates was described by Mr. Melling (Factory Superintendent, Printing). The principles of lithography were briefly explained with special reference to its suitability for continuous

high speed printing of tinplate in individual sheets.

The party was then conducted round the printing department, seeing the application of white background coatings, followed by the colour printing stages and finally varnish coating. All the applications were followed by stoving. The machines which were shaping tins, soldering or folding seams and attaching bases and tops were then inspected.

During tea time, technical questions were put to Mr. Melling and Mr. Lott (of the company's printing research department). The party was finally shown the quality control sample room which contained a specimen of every type of tin recently produced in the factory. The display of tins numbered hundreds, and provided an interesting survey of current ideas on packaging design for sales promotion.

Mr. W. J. Arnot expressed the party's thanks for an interesting and instructive afternoon.

Scottish Section

The Annual Golf Outing of the Scottish Section took place at Aberdour on 20th April, when 23 members and visitors took part, members competing for the Whittaker Cup.

In brilliant sunshine some excellent scoring resulted. The prize winners were as follows :

MEMBERS		*Net Score*
1st (Whittaker Cup)	D. Rowley	59
2nd	J. H. Stewart	61
3rd	R. Harvie	62
VISITORS		
1st	J. Robertson	62
2nd	O. Anderson	67
3rd	A. Pullar	72
Best scratch score	R. A. Sim	72
Best outward half	A. MacGuire	37
Best inward half	R. A. Sim	35

West Riding Section

Annual General Meeting

The Section's Annual General Meeting was held on 12 April 1966 at the Great Northern Hotel, Leeds. After the business of the evening, which included the Chairman's Review, the Reports of the Committee, Hon. Treasurer, and Hon. Publications Officer, and of course the election of Officers and Committee Members for the coming year, the wives and lady friends of the members joined the members for a buffet supper followed by a film on the work of the Guide Dogs for the Blind Association.

A spontaneous collection after this very interesting film raised the sum of £3 for this worthwhile cause.

Officers and Committee Members elected for the forthcoming year are as below :—

Chairman : Mr. N. Cochrane

Vice-Chairman and Representative on Council : Mr. L. H. Silver

Hon. Secretary : Mr. D. H. DuRieu

Hon. Treasurer : Mr. T. R. Smith

Hon. Publications Officer : Mr. J. N. McKean

Assistant Publications Officer : Dr. L. J. Watkinson

Social Secretary : Mr. M. J. Cochrane

Hon. Auditor : Mr. P. M. Haigh

Committee : Mrs. K. Driver, Mr. D. T. Young, Mr. C. Allsop, Mr. J. P. M. Denny, Mr. D. Morris and Mr. F. J. Moreham

The decision to replace Mr. Denny as Representative on Council, by Mr. Silver, was taken as it was felt that it would be of assistance to the Vice-Chairman to precede his period of office by being the Representative on the Council.

J. N. MCK.

Obituary

Dr. John Albert Newton-Friend

Dr. Newton-Friend, whose death on 15 April has been noted in the June issue of the *Journal*, was born at Newton Abbot in July 1881. Whilst still a child the family moved to Birmingham, where subsequently he won scholarships to King Edwards' School and the University. He graduated B.Sc. (Hons.) in 1902 and obtained his M.Sc. in 1903. In later life it was a matter of some gratification to him that the costs of his education had been entirely met either by scholarships or his own efforts. For this reason his university studies were interrupted at this point for three years, for which time he was Science Master at Watford Grammar Schools. His savings during this period, together with a Carnegie Scholarship, enabled him to spend two years at the University of Würzburg, where he gained his Ph.D. in 1908. He then returned to the teaching of chemistry, which thereafter was to occupy the whole of his working life. From 1908 to 1912 he was Head of the Chemistry Department, Darlington Technical School, from whence he proceeded to the Headmastership of the Victoria Institute, Science and Technical Schools, Worcester. Whilst there he obtained his D.Sc. (Birm.) in 1912, and the following year was awarded the Carnegie Gold Medal for his research work on corrosion. From 1920 until his retirement in 1946 he was Head of the Chemistry Department, Birmingham Technical College—now the University of Aston in Birmingham.

Dr. J. A. Newton Friend

Dr. Friend's wide interests in chemistry can be best illustrated by a selection of the titles of his numerous books published over a span of 40 years. The first in 1909 was entitled *Theory of Valence*, to be followed a year later by *Introduction to the Chemistry of Paints*. A life-long interest in corrosion is recalled by *Corrosion of Iron and Steel* (1911) A return to paint in 1917 with *Chemistry of Linseed Oil*. From then on the titles reflect his increasing preoccupation with the task for which he will be long remembered—the teaching of inorganic and physical chemistry to generations of students at the Birmingham "Tech." In 1920 appeared the first volume—*Cobalt, Nickel and the Elements of the Platinum Group*—of what was to become the multi-volume *Textbook of Inorganic Chemistry* under Friend's editorship. Finally in 1932 and 1935 appeared volumes one and two respectively of the book with which his former students will always associate him—*A Textbook of Physical Chemistry*.

His connection with OCCA dates from 1919 when he was elected to Honorary Membership. He became President in 1922 and also served as Vice-President in 1925-26 and again in 1962-64. At the

time of his death he was the senior surviving past President and those who have been privileged to attend the annual dinner for members of Council and Officers, past and present, will remember with admiration Dr. Friend's speeches in that capacity. Probably his greatest contribution to OCCA was his willingness to accept the Presidency at a time during its early years when, as a person from the academic world, free from industrial connections, he was able to enhance its status.

He served in the Special Brigade, Royal Engineers, during the 1914-18 War and was again commissioned in the Home Guard during the Second World War. He played a major part in the training of the Home Guard in the Midlands, as well as continuing to administer his department at the Technical College. The writer recalls with pleasure, during the darkest days of the war, a chance encounter with Friend, who seemed remarkably cheerful considering the situation. It transpired that he had just finished marking Home Guard examination papers in which appeared the following question—"What would you do if a gas shell dropped nearby?" The cause of Friend's amusement? The answer of one of the candidates—"I should run like hell."

After his retirement, Dr. Friend was engaged for some years in lecturing on chemistry to members of HM Forces, both at home and overseas. With the ending of National Service he was able finally to devote himself to his other varied interests. Among the list of his publications from 1951, three distinct topics are discernible: numerology—*Numbers, Fun and Fact* (1954), *More Numbers, . . . etc.* (1961), *Still More Numbers, etc.* (1964); the supernatural—*Demonology, Sympathetic Magic and Witchcraft* (1961); the history of Birmingham. The latter subject was especially dear to Friend and he had made himself an authority on it. Alas, his only publications in this field are two slender volumes, *Forgotten Birmingham* and *Forgotten Aston Manor in Birmingham*, which appeared in 1964 and 1965. Members of the Midlands Section will recall the series of lectures on this subject which he gave some years ago, out of which grew the Annual Newton-Friend Lecture, at which Dr. and Mrs. Friend were regular attenders, the last occasion being on 18 March 1966.

His own publications provide the surest epitaph for his scientific achievements; his work on corrosion and linseed oil—so much in advance of his contemporaries—entitle him to a place of honour. However, despite his early promise and undoubted academic brilliance, the ultimate goals always eluded him. This he realised was the price exacted from those who, like himself, feel obliged to voice, without fear or favour, the truth as they see it.

What of the man himself? Friend radiated an old-world courtesy and charm beneath which lay a will of steel—rigid and inflexible when once the issues had been resolved to its own satisfaction. To his students, a stern disciplinarian, ever conscious to maintain the high standards of the College he served so well. In private life a most absorbing conversationalist, whom the writer, despite unceasing mutual antagonism during his student days, came to count as friend in more than name. This experience is by no means unique; countless former students would wish to join with members of OCCA in expressing their admiration of their mentor and their sympathy to Mrs. Friend and her daughter.

S. A. RAY.

Symposium on Wetting

A symposium organised under the auspices of the Bristol Section of the Society of Chemical Industry and the Colloid and Surface Chemistry Group of the Society of Chemical Industry will be held on 12, 13 and 14 September 1966 at the University of Bristol. Registration forms and further details may be obtained from the Society of Chemical Industry, 14 Belgrave Square, London, W.1.

Symposium on Diffusion Coatings

A symposium on diffusion coatings will be held at the Borough Polytechnic on Thursday 30 June 1966. Four papers will be read, and the subjects will be "Fundamental Aspects of Diffusion Coatings," "Chromising," "Aluminium and Zinc Coatings" and "Application of Mixed Diffusion Coatings." Further details and registration forms may be obtained upon application to the Secretary, Borough Polytechnic, Borough Road, London, S.E.1.

XXI International Congress of Pure and Applied Chemistry, Prague, 1967

The XXI International Congress of Pure and Applied Chemistry will be held in Prague from 4 to 10 September 1967. Circulars containing cards of provisional registration may be obtained from Dr. D. C. Martin, The Royal Society, Burlington House, London, W.1.

Symposium on Plastics and Polymer Technology

A symposium on plastics and polymer technology will take place at the Borough Polytechnic on 27 and 28 October 1966. The symposium is designed to cover the production, properties and uses of those addition copolymers for which industrial applications have been realised, and will comprise separate lectures on copolymers of ethylene, propylene, isobutylene, vinyl chloride, acrylonitrile and acrylic esters. Polycondensation products will not be included. There will be a lecture on block and graft copolymers and the uses of copolymers in adhesives, surface coatings, paper treatment and fibre and textile production will be given special treatment.

Further details and registration forms may be obtained on application to the Secretary, Borough Polytechnic, Borough Road, London, S.E.1.

Symposium

Advance notice is given of a symposium to be held by Hull Section at Hull University on 20 April 1967. The subject will be "Organometallic Compounds" and full details will be given in a later issue of the *Journal*.

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

BECALICK, ALAN JAMES, B.SC., 39 Ivy Road, Poynton, Nr. Stockport, Cheshire. (*Manchester*)

BEECHE, CARLOS, c/o Grace y Cia (Peru), Fabrica de Pinturas VENCEDOR, Casilla 1381, Lima, Peru. (*Overseas*)

BERROCAL, ADRIANA, c/o Grace y Cia (Peru), Fabrica de Pinturas VENCEDOR, Casilla 1381, Lima, Peru. (*Overseas*)

BLAIR, EDWARD NOEL, 16 Polson Drive, Johnstone, Scotland. (*Scottish*)

BURAK, NATHAN, M.SC., A.R.I.C., A.M.C.T., 5 Ladythorn Avenue, Prestwich, Manchester. (*Manchester*)

CARGILL, VINCENT PAUL, 240, Mather Avenue, Allerton, Liverpool. (*Manchester*)

CHARNLEY, HARRY FREDERICK JAMES, c/o British Resin Products Ltd., Barry House, Hale, Cheshire. (*Manchester*)

DONKERSLEY, BRIAN, B.SC., 32 Woodhall Park Crescent East, Pudsey, Yorkshire. (*Newcastle*)

ELIAS, SANTIAGO, c/o Grace y Cia (Peru), Fabrica de Pinturas VENCEDOR, Casilla 1381, Lima, Peru. (*Overseas*)

ENNOR, KENNETH STAFFORD, PH.D., A.R.I.C., c/o British Oxygen Chemicals Ltd., Chester-le-Street, Co. Durham. (*Newcastle*)

FENWICK, RAYMOND, 14 Balmoral, Great Lumley, Chester-le-Street, Co. Durham. (*Newcastle*)

FRENCH, G. P., B.SC., c/o Dr. Beck & Co. (England) Ltd., Hayes Lane, Slinfold, Horsham, Sussex. (*London—Thames Valley*)

GHOSH, SUNIL KUMAR, B.SC., A.R.I.C., 51 S. R. Das Road, Ground Floor, Cal. 26, Calcutta 26, India. (*Overseas*)

HAMILL, FRANCIS, A.R.I.C., 3 Fox Hills Crescent, Lanchester, Co. Durham. (*Newcastle*)

HEUZENROEDER, JOHN MORITZ, 24 Comley Street, Brighton, South Australia. (*South Australia*)

HILL, MISS VIRGINIA M., 8355 Camargo Road, Cincinnati, Ohio 45243, U.S.A. (*Overseas*)

HUACO, DANIEL, c/o Grace y Cia (Peru), Fabrica de Pinturas VENCEDOR, Casilla 1381, Lima, Peru. (*Overseas*)

KELLEHER, THOMAS FRANCIS, B.SC., " Suirlee," 34 Louvain, Roebuck Road, Dublin, 14. (*Irish*)

KERCHHOFF, PETER CAMPBELL, B.SC., c/o Alcan Aluminium of South Africa Ltd., P.O. Box 74, Pietermaritzburg, Natal, South Africa. (*South Africa*)

MCDOWALL, JAMES, L.R.I.C., 20 Oakwood Avenue, Paisley, Renfrewshire, Scotland. (*Scottish*)

MANDELSON, JACK, 34 Lomondside Avenue, Clarkston, Glasgow. (*Scottish*)

MAWSON, JACK FRANKLAND, B.SC., 8 Victoria Road, Saltford, Bristol. (*Bristol*)

ORTIZ, JORGE CHAVEZ, c/o Grace y Cia (Peru), Fabrica de Pinturas VENCEDOR, Casilla 1381, Lima, Peru. (*Overseas*)

PEREZ VELARDE, LUIS, c/o Grace y Cia (Peru), Fabrica de Pinturas VENCEDOR, Casilla 1381, Lima, Peru. (*Overseas*)

RAWLINGS, LEONARD FREDERICK, 3 Colleton Drive, Twyford, Nr. Reading, Berks. (*London—Thames Valley*)

ROBINSON, PETER JOHN, B.SC., M.SC., c/o Durham Chemicals Ltd., Birtley, Co. Durham. (*Newcastle*)

SELDON, MARK WILLIAM, c/o Mander-Kidd (S.A.) Pty. Ltd., P.O. Box 2907, Johannesburg, South Africa. (*South Africa*)

SELLEY, JEFFREY EDGAR, L.R.I.C., 22 Foster Street, Kinver, Staffs. (*Midlands*)

SKEIST, IRVING, Skeist Laboratories Inc., 89 Lincoln Park, Newark, New Jersey 07102, U.S.A. (*Overseas*)

SUGGITT, NEIL WILLIAM STRATHEARN, B.SC., c/o Dow Chemical Africa Pty. Ltd., P.O. Box 3429, Durban, Natal, South Africa. (*South Africa*)

SYKES, ROGER CHRISTOPHER, c/o Ciba Clayton Ltd., Queen Street, Leicester. (*Midlands*)

VAN RENSBURG, MARTIN FRANK JANSEN, c/o Dulux Ltd., 318 Gale Street, Durban, Natal, South Africa. (P.O. Box 1045). (*South Africa*)

Associate Members

BEAN, ALFRED, 225 Braemar Road, Billingham, Co. Durham. (*Newcastle*)

GALANOS, GEORGE FREDERICK, c/o G. J. Wevell (Pty) Ltd., P.O. Box 1240, Durban, Natal, South Africa. (*South Africa*)

HORTOP, MISS MARGARET CECILIA, P.O. Box 1045, Durban, Natal, South Africa. (*South Africa*)

KAVANAGH, JOHN J., 5 Marian Park, Dublin 14. (*Irish*)

LUNDIE, ROBERT, P.O. Box 1366, Johannesburg, South Africa. (*South Africa*)

MEYERKORT, RUDOLF, P.O. Box 1521, Durban, Natal, South Africa. (*South Africa*)

NATION, THOMAS DERRICK, c/o Hoechst Chemicals Ltd., 115/117 Colmore Row, Birmingham 3. (*Midlands*)

PETERS, ANTHONY GEORGE, 55 Fifth Avenue, Edenvale, Transvaal, South Africa. (*South Africa*)

QUIN, DENIS LEO, P.O. Box 829, I.H.B., South Africa. (*South Africa*)

VAN STUYVESANT MEYEN, J. J., N. V. Nederlandse Muurverf Industrie "Nemi," Amsterdamseweg 14, Uithoorn, Holland. (*Overseas*)

Junior Members

COLLIER, RICHARD, c/o British Rail Research Dept., Protective Coatings Laboratory, London Road, Derby. (*Midlands—Trent Valley*)

DICKENS, DAVID MICHAEL FRASER, c/o Dulux Ltd., 318 Gale Street, Durban, Natal, South Africa. (P.O. Box 1045). (*South Africa*)

DOWLING, DAVID GEORGE, c/o John Hall & Sons (Bristol & London) Ltd., Petherton Road, Hengrove, Bristol 4. (*Bristol*)

PHILLIPS, ALAN, 92 Gilbertstone Avenue, South Yardley, Birmingham 26. (*Midlands*)

PHIPPS, PETER CLIFFORD, 66 Heyford Avenue, Eastville, Bristol 5. (*Bristol*)

Forthcoming Events

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

Monday 11 July

Victorian Section. Film Night at Shell Co. of Australia Theatrette. Details from Victorian Section Secretary, G. P. Hartshorn, c/o Australian Titan Products Pty. Ltd., Newson Street, Ascot Vale, Victoria.

Thursday 21 July

Western Australian Section. One day joint Symposium in conjunction with the West Australian Section of the Australian Corrosion Association. Details from Western Australian Section Secretary, G. D. Parsons, c/o Polymer Corporation Interstate Pty. Ltd., 1141 Hay Street, Perth.

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 Western Australian Section Secretary.

Oil and Colour Chemists' Association

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

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

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

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

PUBLICATIONS

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

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

Paint Technology Manuals

Part 1 : " Non-convertible Coatings," Pp. 326, 35s.
Part 2 : " Solvents, Oils, Resins and Driers," Pp. 239, 35s.
Part 3 : " Convertible Coatings," Pp. 318, 35s.
Part 4 : " The Application of Surface Coatings," Pp. 345, 35s.
Part 5 : " The Testing of Paints," Pp. 196, 35s.
Part 6 : " Pigments, Dyestuffs and Lakes," Pp. 340, 35s.

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

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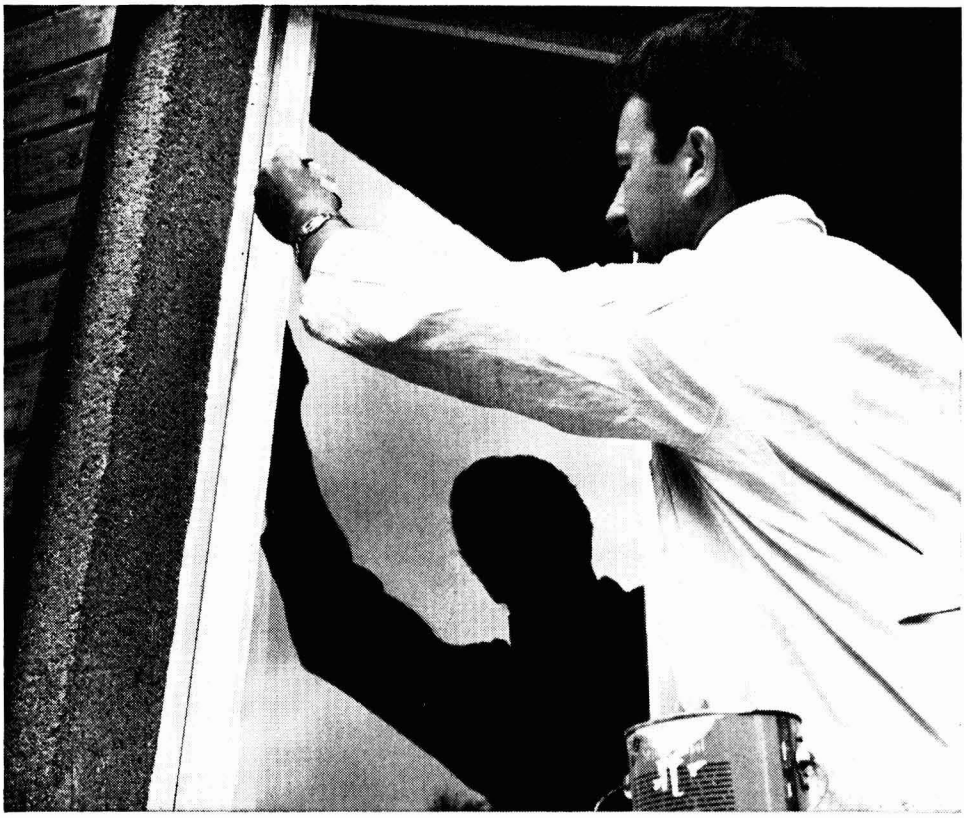

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W. Garmston, Group Personnel Department,
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Hayes, Middlesex.

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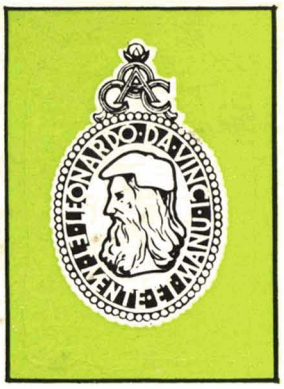

Oil and Colour Chemists' Association

Alexandra Palace
London

March
13 — 17

Closing date for applications for stand space, 2 September 1966

for further details see page 589