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OIL AND COLOUR CHEMISTS' ASSOCIATION

Vol. 51 No. 3 **March 1968**

OCCA-20 PREVIEW

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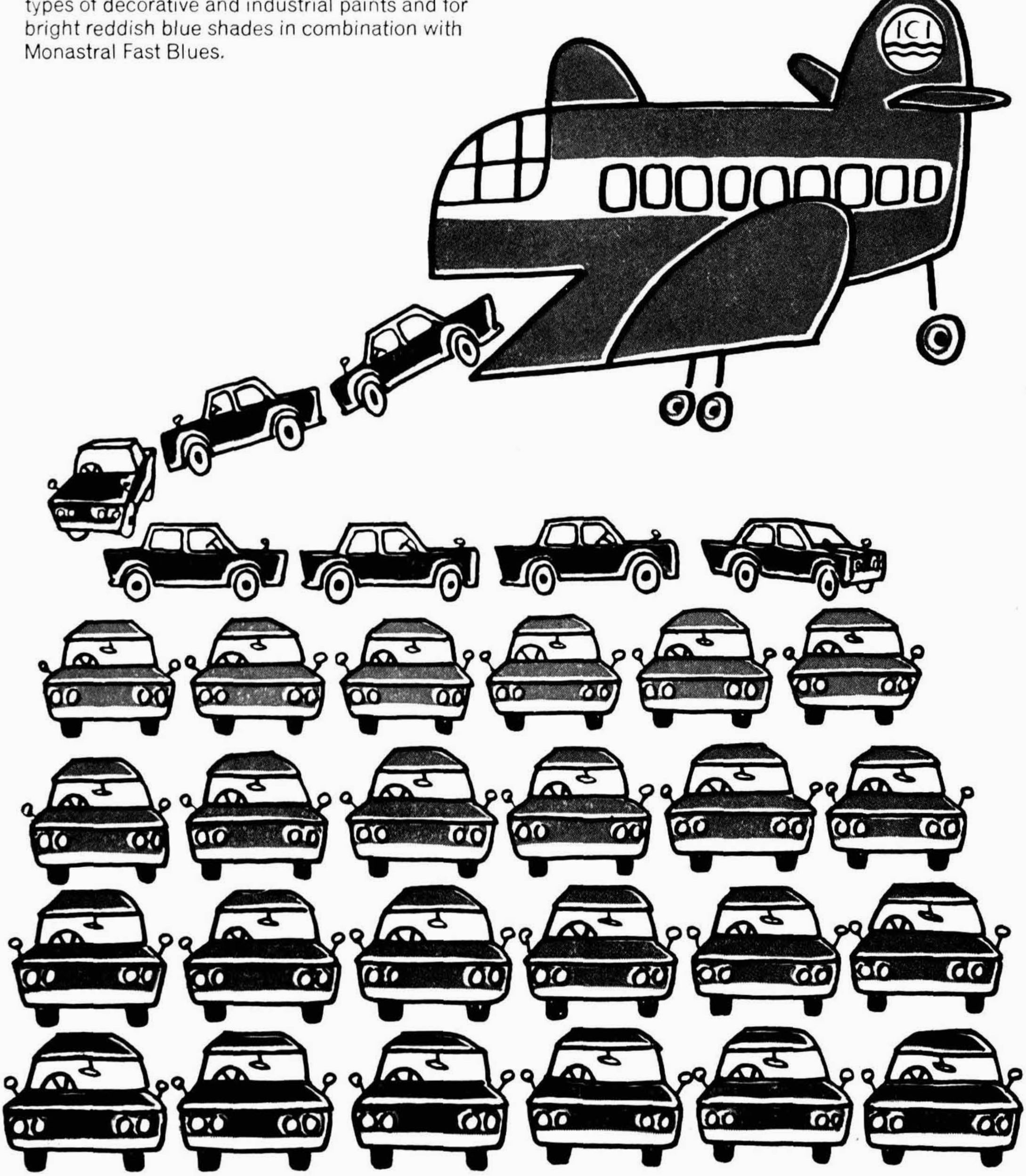
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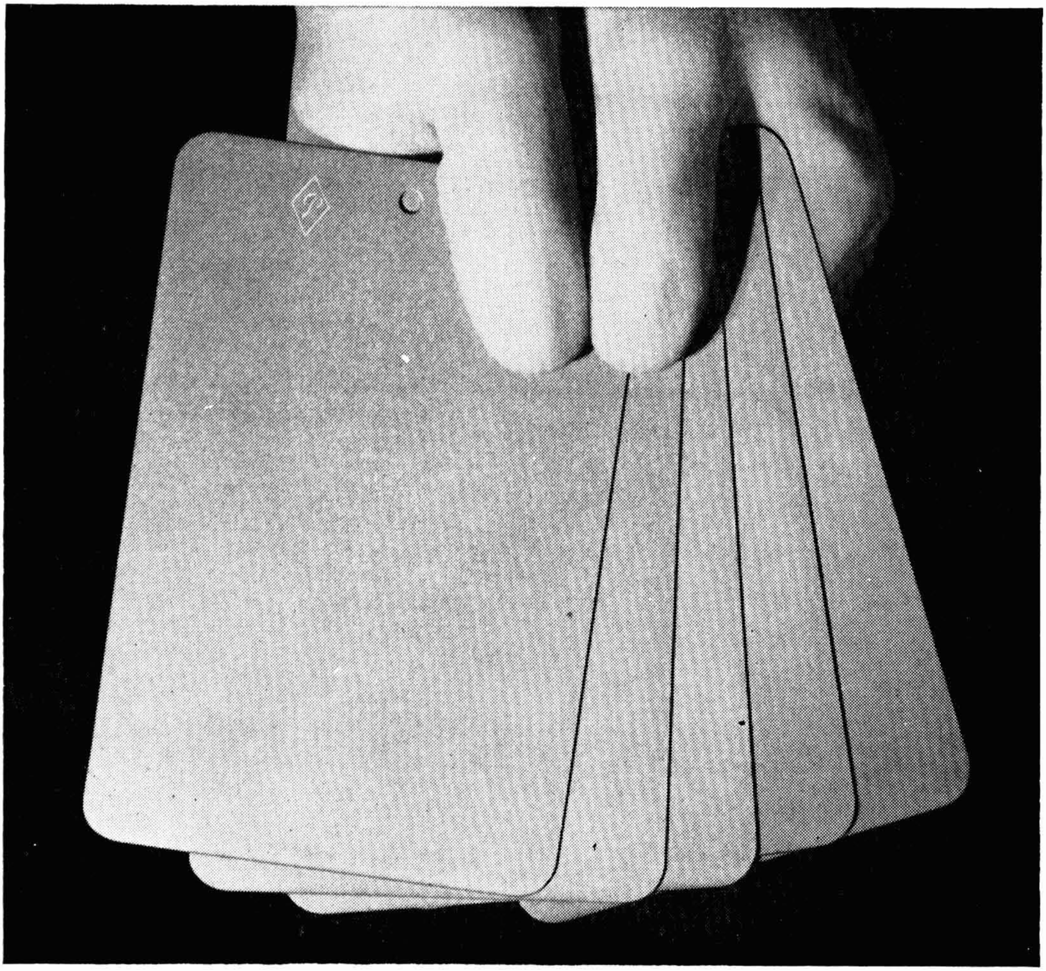

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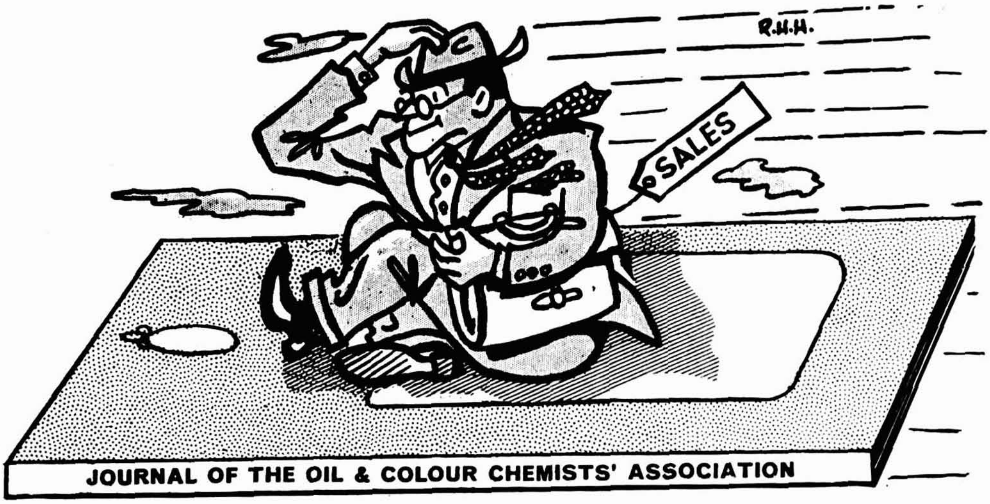

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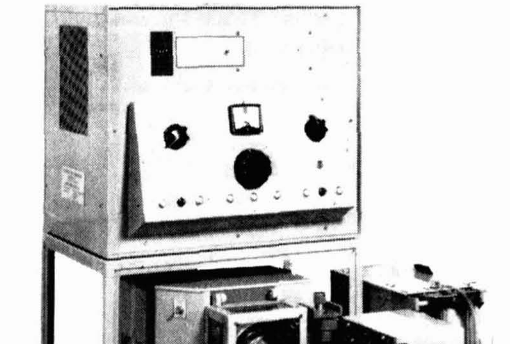

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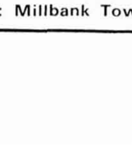

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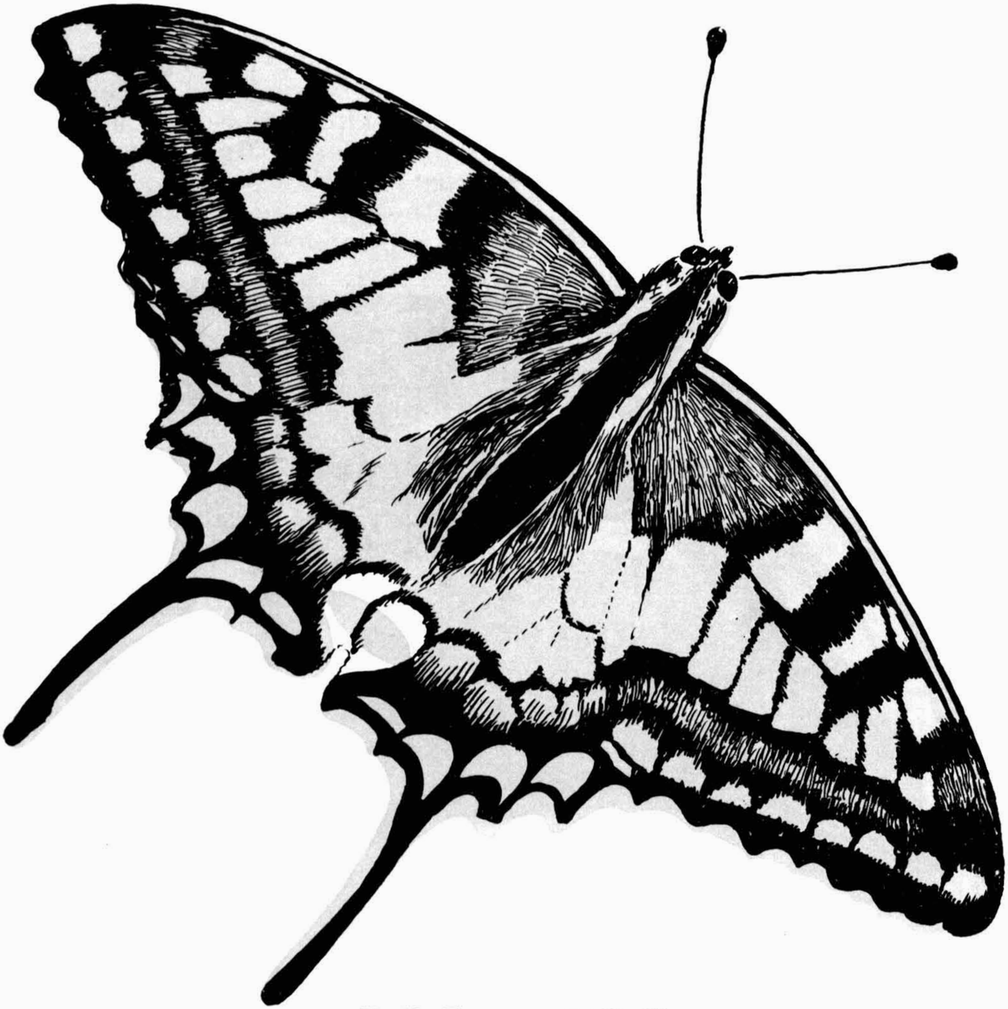

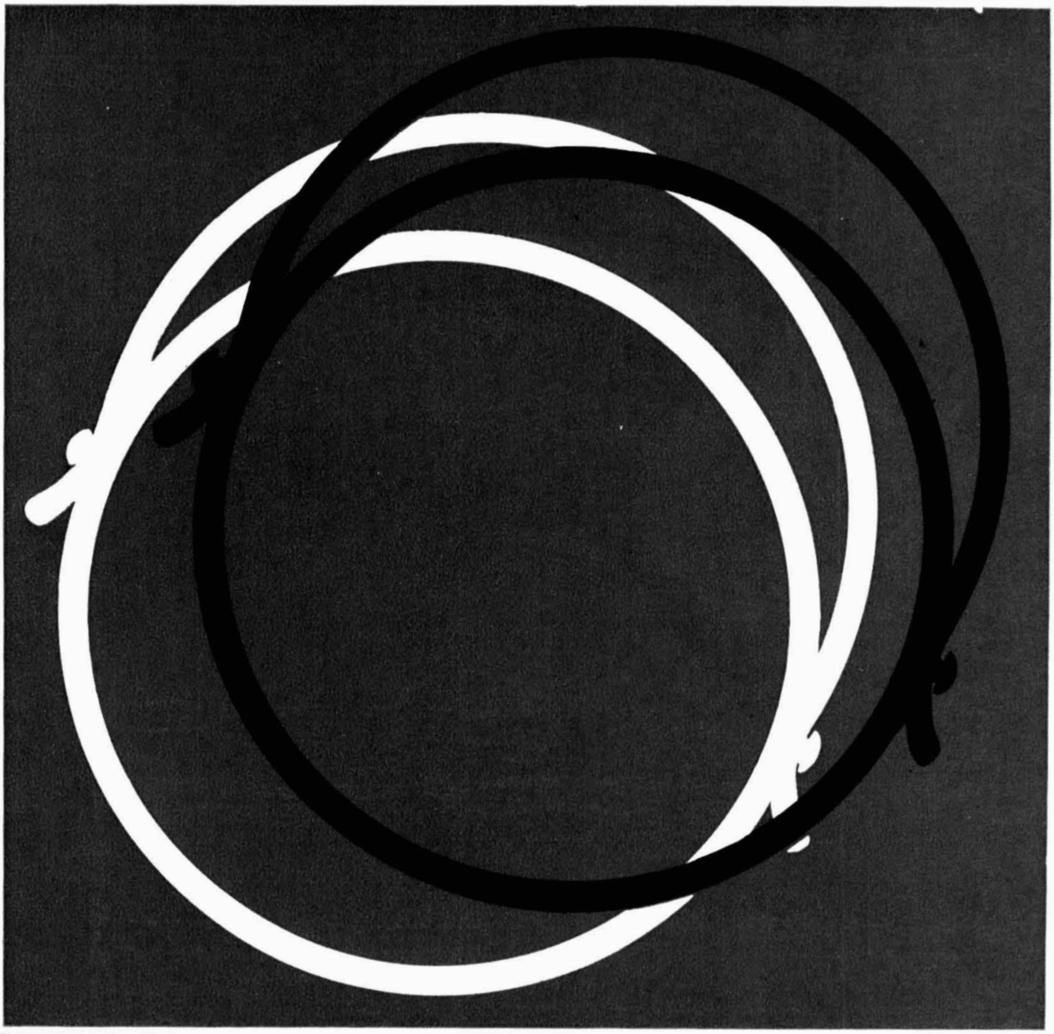

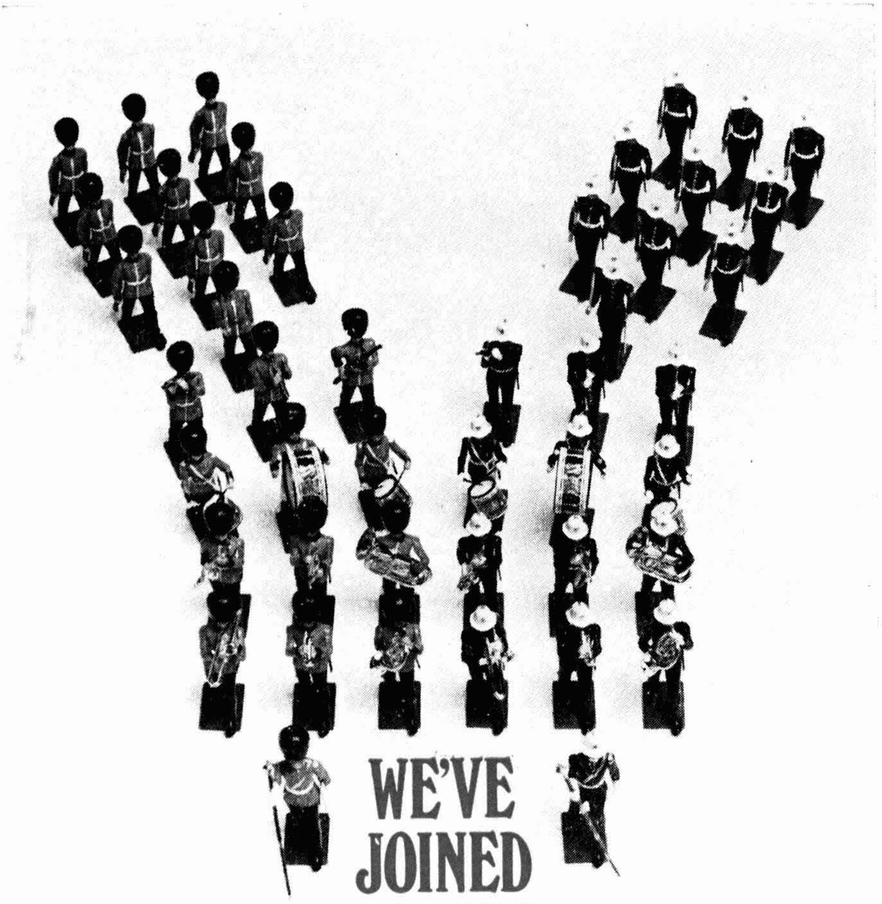
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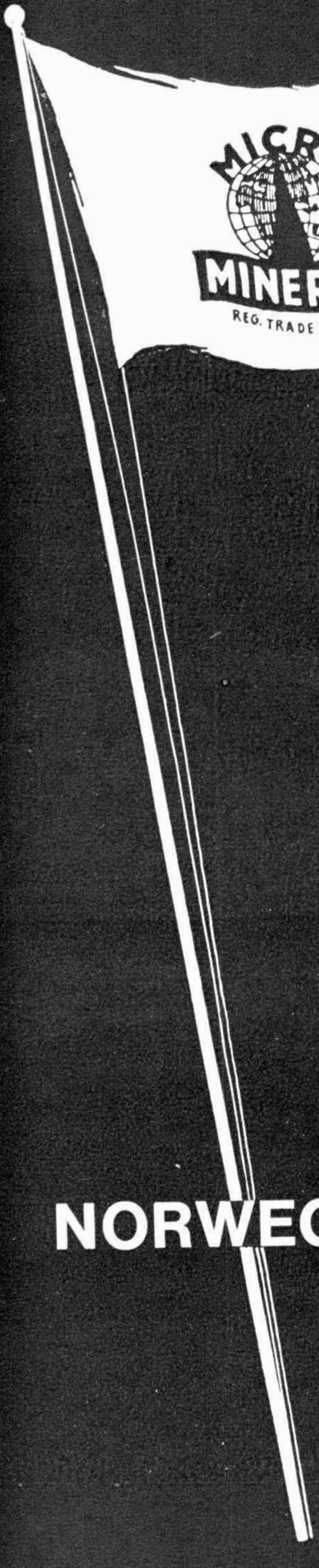
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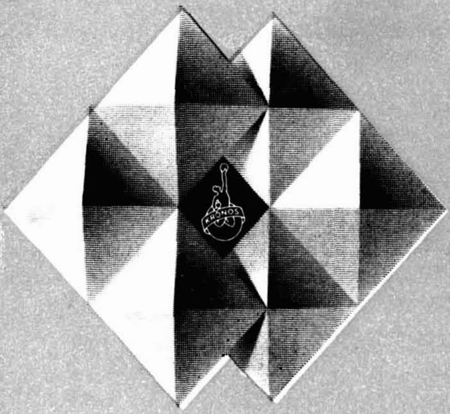
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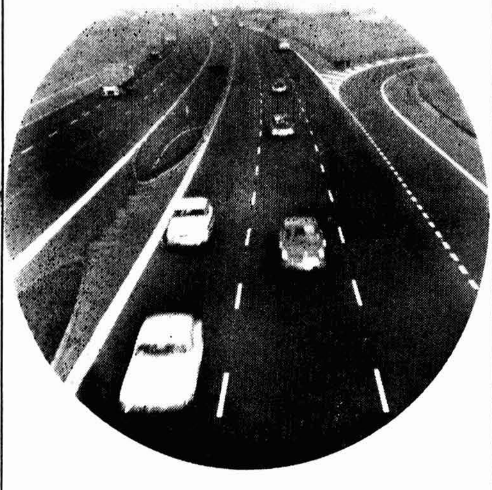

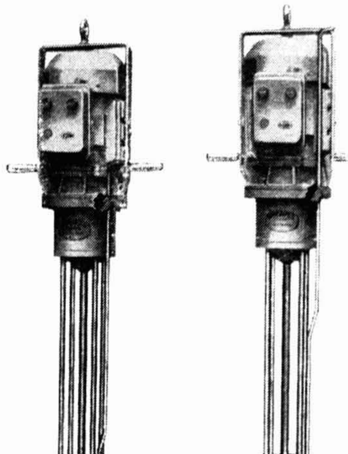

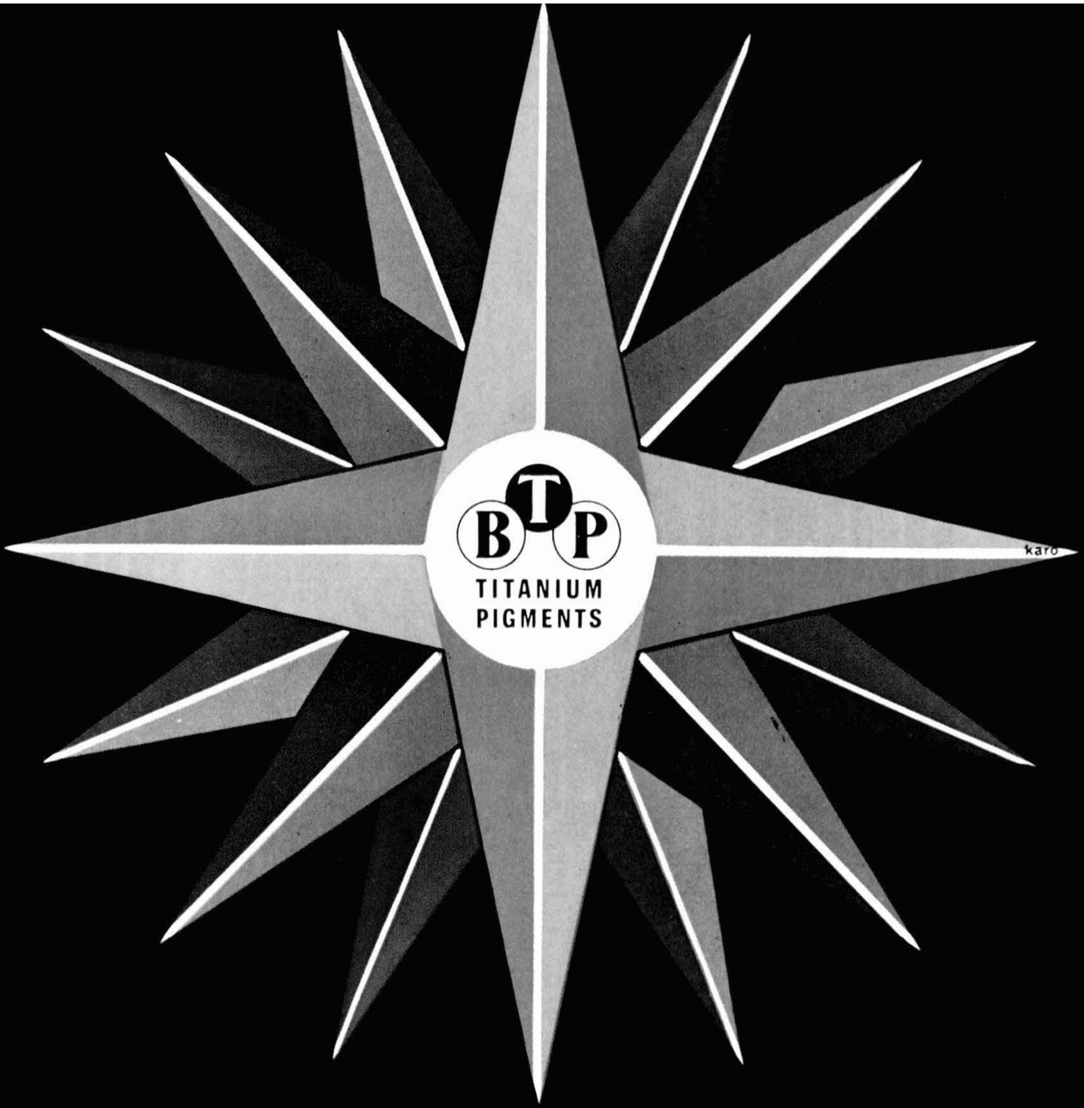
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Published by

OIL AND COLOUR CHEMISTS' ASSOCIATION
Wax Chandlers' Hall, Gresham Street, London E.C.2

J. Oil Col. Chem. Assoc. 1968, 51, 191-197

Transactions and Communications

An opacity gauge for the surface coating industries

By I. H. Day

Research and Development Department, Polymark Limited, Jeddo Road, London, W.12

Summary

This article describes a new type of opacity gauge which can be used by all sections of the surface coating industry and which is a very simple tool to provide information on :—

(1) the fineness of grind.

(2) the opacity of a film of ink or paint, both on application and after drying.

(3) the colour strength of transparent formulations.

(4) the spreading rate of the dry paint, in terms of the wet thickness applied.

One of the main advantages of the opacity gauge is that it can be used with the wet film. It should therefore find a place both in the laboratory and the mill room.

Arrangements are being made for the gauge to be produced commercially.

Une jauge d'opacité destinée aux industries de peintures et de revêtements assimilés

Résumé

Cet article décrit un type nouvel de jauge d'opacité dont tous les secteurs de l'industrie de revêtements superficiels peuvent s'en servir. Ce dispositif, très facile à l'emploi, peut fourni les informations suivantes :

(1) Finesse de broyage.

(2) Opacité d'un feuil d'encre d'imprimerie ou de peinture, soit frais, soit après séchage.

(3) Pouvoir colorant de revêtements transparents.

(4) Rendement en surface en fonction de l'épaisseur du feuil frais appliqué.

Un des avantages principaux de la jauge d'opacité est la possibilité de son utilisation sur le feuil frais. Ainsi elle doit trouver sa place dans la laboratoire et dans l'atelier.

On est en train de faire des dispositions pour la fabrication commerciale de la jauge.

Ein Opazitäts-Messgerät für die Lackindustrie

Zusammenfassung

Ein neuartiges Opazitäts-Messgerät wird in dieser Abhandlung beschrieben, das von allen Zweigen der Anstrichmittelindustrie benutzt werden, und trotz seiner Unkompliziertheit über folgendes Auskunft geben kann :

(1) Mahlfeinheit.

(2) Deckfähigkeit von sowohl nassen, als auch getrockneten Druckfarben- oder Anstrichfilmen.

(3) Farbkraft transparenter Vorschriften.

(4) Ausgibigkeit, wobei aus der Dicke des nassen auf die des trockenen Anstrichfilms gefolgert wird.

Einer der wesentlichen Vorteile des Opazitäts-Messgerätes besteht darin, dass es mit nassen Filmen angewandt werden kann. Daher dürfte es sowohl im Laboratorium, als auch im Mühlensaal Verwendung finden.

Es sind Schritte im Gange, das Messgerät industriemässig zu erzeugen.

Показатель непрозрачности для применения в красочной промышленности

Резюме

Описывается новый тип показателя непрозрачности который может применяться во всех отраслях красочной промышленности и который является очень простым прибором для получения сведений о следующих:-

(1) тонине помола

(2) непрозрачности чернильной или красочной пленки, как при применении так и после сушки

(3) прочности цвета прозрачных формуляций

(4) скорости распространения сухой краски в зависимости от приложенной мокрой толщины.

Одним из главных преимуществ показателя является тот факт что он может применяться с мокрой пленкой и потому может потребляться как в лаборатории так и в заводской мастерской. Принимаются меры для промышленного производства показателя.

Introduction

In order to carry out tests on coating films, it is usually necessary to produce the film in the dried form to check its covering power. Methods of application vary, and test results are ultimately dependent on the operator producing a uniform film.

During some experimental work, the writer found himself in a position of having to evaluate the opacity of ink films of many different formulations and found that there was no simple piece of equipment which would do this readily.

Description of opacity gauge

The opacity gauge is reminiscent of the well-known fineness-of-grind gauge, as the measuring area is an accurately sloping channel between two shoulders. The construction is, however, quite different.

The channel consists of two " vitrolite " glass strips, 12.7 mm wide × 15 cm long, one black, one white, cemented together and ground optically flat. The channel, and the shoulders, also of glass, are cemented into a metal body. The shoulders are ground at a slight angle to the channel so that the depth of the channel varies uniformly from 0-50 microns or 0-100 microns along its length. The instrument is calibrated in microns at regular intervals along its length.

A steel scraper blade and a leather case complete the instrument.

Experimental

The following experiments illustrate the usefulness of this opacity gauge.

For this work, an early prototype gauge calibrated in British units was used.

(A) In a development project where absolutely fast pigments were required for paints with high opacity, a cadmium red pigment was in use. Paints made

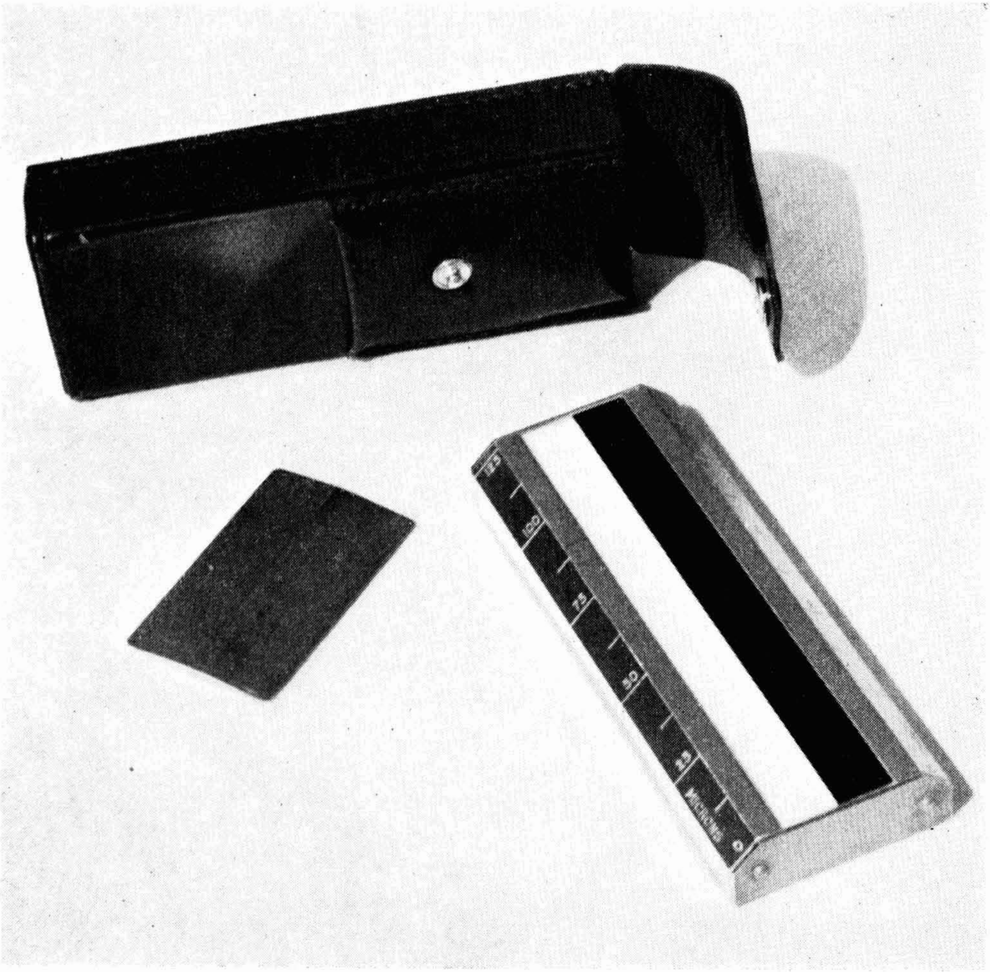


Fig. 1. The opacity gauge, complete with scraper blade and leather case

from this pigment, however, cost twice as much as others in the colour range. A replacement (not necessarily an exact match) was sought and a number of fast organic pigments were obtained for trial.

The pigments were first rubbed out with titanium dioxide and medium to find the maximum amount of dilution which could be tolerated while retaining a suitable colour.

Paints were then made up to the same pigment volume concentration and the opacities measured.

The spreading rate and the cost of pigment per 1,000 sq. ft. were calculated.

Considerable savings in cost were readily achieved.

Fig. 2. The opacity gauge in use, showing an opacity reading of 90 microns

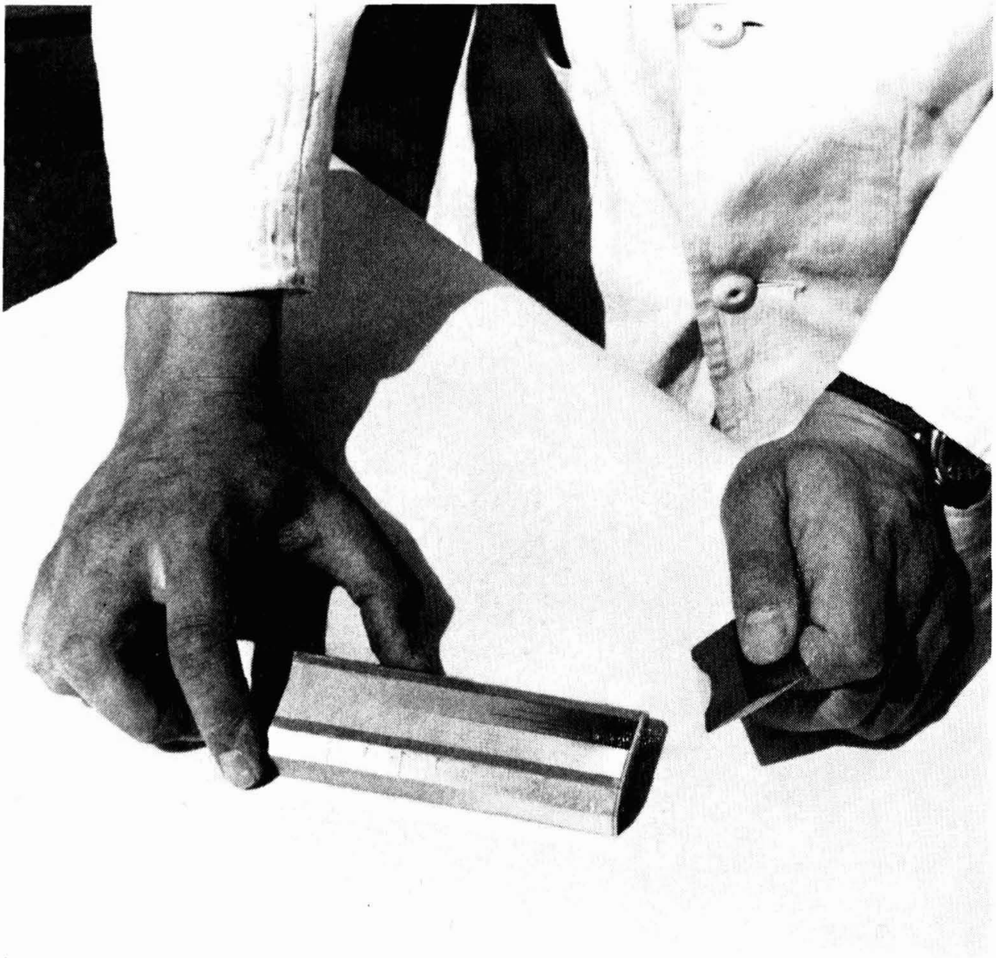


Fig. 3. The opacity gauge in use, during a milling operation, showing the scratch lines caused by oversize particles. Here the opacity reading is 90 microns, and the fineness of grind is 40 microns

Table 1

Pigment		Cost per lb	Opacity in. × 10^3	Spreading rate (sq. ft/gal)	Cost of pigment per 1,000 sq. ft
A	Organic	68s.	2.0	960	36s.
B	Organic	48s.	1.8	1,070	21s.
C	Organic	28s.	1.2	1,600	8s.
D	Cadmium	34s.	2.0	960	49s.

(B) A further problem was the formulation of a flexographic ink to give a bright blue colour at a wet thickness of about 0.0007″.

A laboratory high speed stirrer and a 300 ml jar charged with 50 ml of 20-40 mesh sand were used for the dispersion. 100 ml of medium were measured into the jar and the sand added. 5 g of phthalocyanine blue were added and milling commenced. After ten minutes, milling was stopped and the sand allowed to settle. A small quantity of ink was removed and tested on the opacity gauge for fineness of grind and colour strength.

One g portions of pigment were added, followed by milling and testing until the required colour was obtained.

As less than 1 ml of ink was removed for each test, the change in composition was negligible—see Table 2.

Table 2

Wt. added (Total)	Fineness of grind	Colour strength	Theoretical % pigment w/v	Calculated % pigment assuming 1 ml removed for test
5 g	off scale	0.0018″	4.84	4.84
1 (6)	,, ,,	0.0015″	5.77	5.79
1 (7)	,, ,,	0.0011″	6.69	6.72
1 (8)	,, ,,	0.0009″	7.60	7.65
1 (9)	,, ,,	0.0007″	8.49	8.59
1 (10)	,, ,,	0.0005″	9.37	9.52

One small-scale experiment gave the required formulation, thus obviating a considerable amount of proofing press work involving the preparation of larger quantities of several inks of different formulae.

(C) It is well known that the opacity reading of paint films increases on drying, and instruments giving only the wet film opacity can give misleading results when used for spreading rate calculations, etc. The opacity of the dry film, in terms of the wet film thickness, can be measured simply by allowing the paint to dry (but not harden) on the opacity gauge.

In one experiment, the opacity reading of a screen printing ink increased from 0.0035″ to 0.005″ (an increase of 40 per cent).

The spreading rate is calculated as follows :

$$\text{Dry opacity} = t/1{,}000 \text{ in or } t/12{,}000 \text{ ft}$$

$$1 \text{ gal} = 1/6.25 \text{ cu. ft}$$

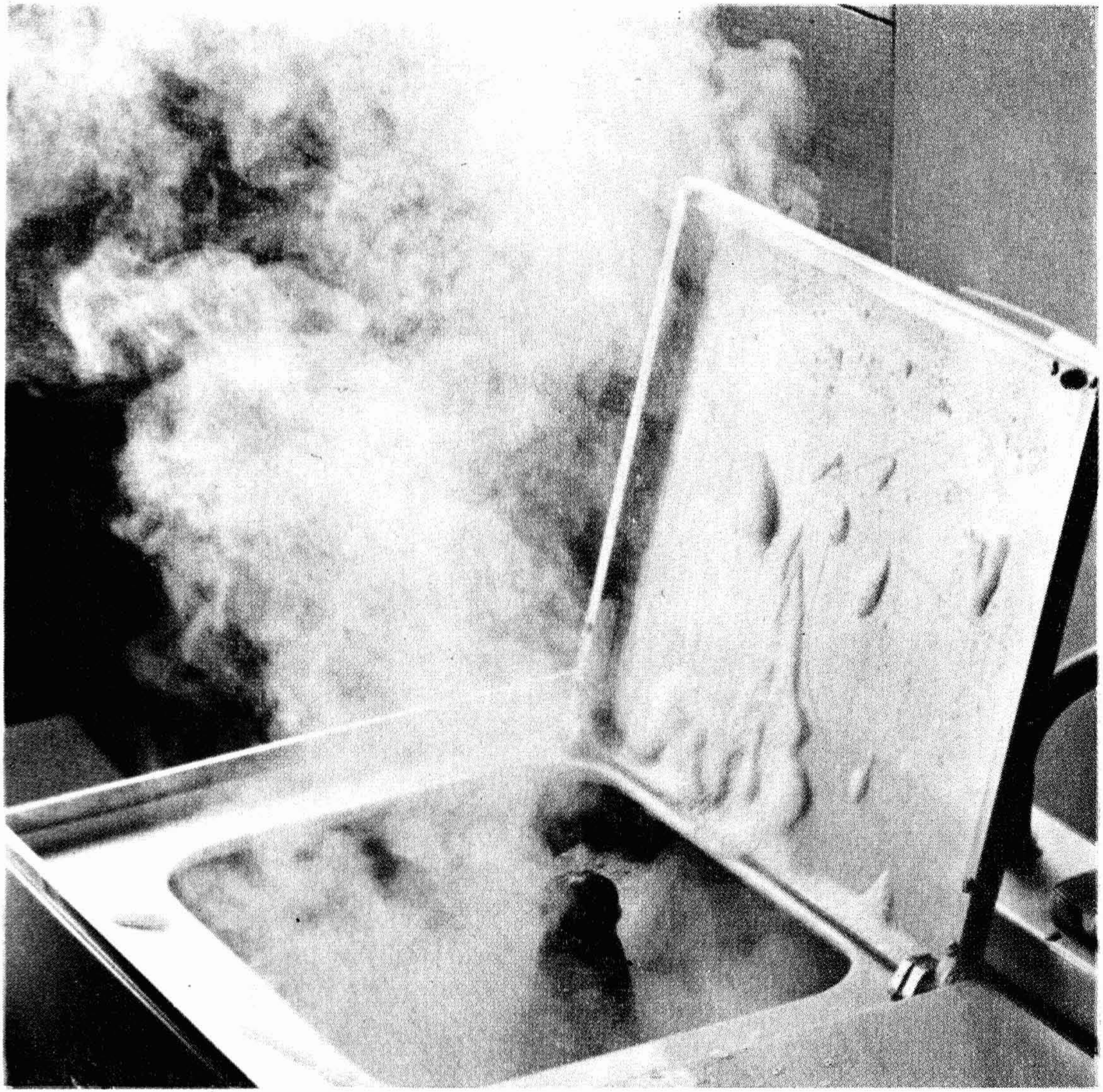
$$\text{Spreading rate} = \frac{12{,}000}{6.25 \text{x} t} \text{ sq. ft per gal}$$

$$\text{In this case } \frac{12{,}000}{6.25 \text{x} 5} = 384 \text{ sq. ft per gal}$$

Where necessary the appropriate factor can be readily determined by experiment, after which in subsequent measurement, the wet film opacity reading can be adjusted to give the dry film opacity. However this will not normally be necessary for quality control and comparative tests.

Discussion of results

These three experiments illustrate the use of the gauge for experimental work and show how economies can be made in both time and materials.



As a quality control tool, the opacity gauge can with advantage replace the Hegman gauge, as the operator can check not only fineness of grind, but also opacity and colour development.

Paint users can use the opacity gauge both to check the quality of their paint supplies, and to determine the spreading rate.

This opacity gauge is covered by British Patent Specification No. 1,085,595 and corresponding Foreign Patents.

Acknowledgments

The author wishes to thank Cook and Perkins Ltd. for their assistance in the design and production of the prototypes. Thanks are also due to Polymark Limited for permission to publish this paper and to the Paint Research Station and members of the industry who helped in the evaluation.

[*Received 13 September 1967*

J. Oil Col. Chem. Assoc. 1968, **51**, 198-212

Relationship between the adsorption of divalent metal soaps on inorganic pigments and their dispersion*

By L. Brandts Buys and E. Dysseleer

Trimetal Paint Co Belgium S.A., Machelen Bt.

Summary

A relationship has been established between the adsorption of divalent metal soaps, in particular, driers, and the dispersion of pigments in a non-polar solvent. Deflocculation was followed by the Daniel flow point method. It has been found that the dispersion of pigments is independent of the nature of the divalent metal but dependent on the nature of the hydrocarbon chain, increasing in the order octoates—naphthenates—oleates. The adsorption experiments show that there is very good correlation between the critical adsorption and the optimum dispersion from Daniel curves.

Le rapport entre l'adsorption de savons de métaux divalents sur des pigments inorganiques et leurs dispersions résultantes

Résumé

Un rapport a été établi entre l'adsorption de savons de métaux divalents, en particulier, des siccatifs, et l'état de dispersion de pigments, dans un solvant non-polaire. Le progrès de défloculation était remarqué par la méthode Daniel de point d'écoulement. On a trouvé que l'état de dispersion de pigments est indépendant de la nature du métal divalent mais il se dépend de la nature de la chaîne hydrocarbure et il s'accroît selon l'ordre octoates—naphténates—oléates. Les expériences sur l'adsorption démontrent qu'il y a de bonne corrélation entre l'adsorption critique et l'état de dispersion optimale derivé des courbes Daniel.

Beziehungen zwischen der Adsorption zweiwertiger Metallseifen Durch Anorganische Pigmente und deren Dispersion

Zusammenfassung

Es wurde festgestellt, dass zwischen der Adsorption zweiwertiger Metallseifen, besonders Trockenstoffen, und der Dispersion von Pigmenten in einem nichtpolaren Lösungsmittel eine Beziehung besteht. Mit Hilfe der Fliesspunktmethode von Daniel wurde der Entflockungsvorgang verfolgt. Dabei wurde gefunden, dass die Pigmentdispersion von der Art des zweiwertigen Metalls unabhängig ist, aber von der Natur der Kohlenwasserstoffkette und zwar in verstärktem Masse in der Reihenfolge Oktansäure-, Naphthensäure-, Ölsäure-Verbindungen. Wie die Adsorptionsversuche zeigen, besteht gemäss der Daniel-Kurven gute Übereinstimmung zwischen der kritischen Adsorption und der optimalen Dispersion.

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*Previously published in *Chimie des Peintures* 1966, **29**, 10, 363.

Соотношение между адсорбцией двухвалентных металлических мыл на неорганических пигментах и их дисперсией

Резюме

Установлено соотношение между адсорбцией двухвалентных металлических мыл, в частности сушителей, и дисперсией пигментов в неполярном растворителе. Дефлоккуляция наблюдалась методом точки текучести Даниеля. Найдено что дисперсия пигментов не зависит от характера двухвалентного металла а зависит от характера углеводородной цепи, увеличиваясь в следующем порядке: октоаты-нефтенаты-олеаты. Адсорбционные испытания показывают что существует хорошее соотномение между критической адсорбцией и оптимальной дисперсией по кривым Даниеля.

Introduction

Most of the major properties of paints are linked in one way or another with the physical and chemical conditions prevailing at the pigment-medium interface. However, in a paint the complexity of the solid/liquid interface is such that experimental study is very difficult and it is necessary to consider much simpler systems, i.e. more ideal systems, if fundamental knowledge is to be gained about surface phenomena. Recent years have seen numerous papers devoted to adsorption in the liquid phase[1] and the paint technologist has available basic data on the adsorption by solid surfaces of particulate materials of substances such as polymers, organic electrolytes, etc. Now adsorption plays an important part in the dispersion of solid particles. Consequently there is obvious interest in any attempt to find a quantitative relationship between the surface adsorption of the pigment and its dispersion in a medium.

The effect of surfactant concentration on various properties of pigment suspensions or of the films obtained from them has been under constant study in these laboratories in recent years and one part of this work formed the basis of a paper given to the VIIth FATIPEC Congress.

The present work was designed to establish a relationship between dispersion and adsorption on a pigment in a system comprising simply a solvent, a pigment and divalent metal soap, e.g. cobalt and lead soaps which are employed industrially as driers, as well as the calcium and zinc soaps used as milling additives.

Experimental

Measurement of dispersion

The wetting and dispersing effect of the divalent metal soaps examined was estimated by the Daniel curve method[2]. This method consists of mixing the pigment or extender with a solution of dispersing agent and then measuring the volume of solution required to produce two characteristic and reproducible consistencies : the wet point and the flow point. The method also involves the observation of the rheological properties of the mixture. Three types of rheological behaviour may be seen :

1. active dilatancy, indicating good dispersion.
2. passive dilatancy, indicating fairly good dispersion.
3. plasticity, indicating a flocculated system.

These three types of behaviour may occur in the same pigment system with the same dispersing agent solution, depending on the concentration of the latter. For a given pigment and solvent, and for dispersing agents belonging to the same group of chemical substances, the absolute amount of solution to be mixed with the pigment to reach the wet point and the flow point is a measure of the wetting and dispersing action of the substances under investigation. In other cases it is also necessary to consider the rheological behaviour of the mixture in addition to the difference between the volume of solution added to reach the wet point and that added to reach the flow point. A small difference between these two volumes is a sign of good dispersion. The wet point curves generally have a minimum. According to Daniel, this minimum corresponds to the optimum concentration of the dispersing agent solution. At this concentration, a minimum of solution is required to reach the wet point or flow point consistencies. The composition of the paste at the minimum of the curve enables the best dispersion to be reached in a ball mill. In certain cases this minimum is extended over a wide range of concentrations. Thus the wet point of Fig. 1 has a flat, horizontal section. The point where the curve becomes horizontal is called here the " critical wet point."

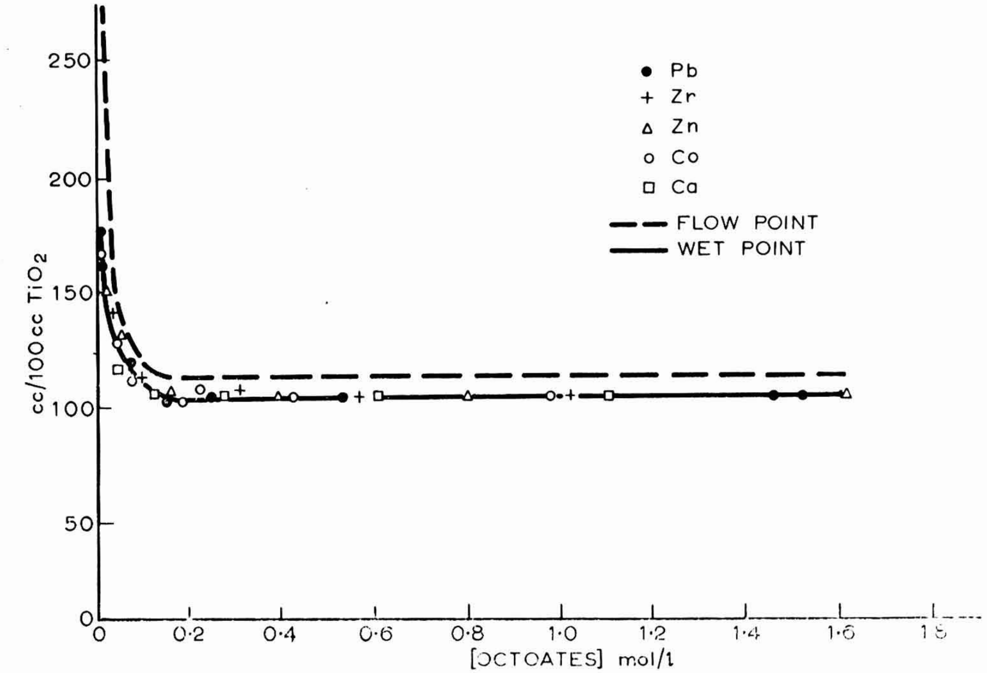

Fig. 1. Wet point (–) and flow point (—) of TiO_2 RNC and octoates in white spirit solution

In the systems studied, the horizontal part of the curves corresponds to a rheological behaviour of the passive dilatancy type, and the vertical section corresponds to plastic behaviour. The reproducibility of the wet point is generally better than that of the flow point. The error is 2 per cent in dilatant systems and 5 per cent in plastic systems.

Measurement of adsorption

The adsorption of metal soaps on pigments is determined in the following way : 20 g of pigment are mixed in a wide-necked 150 ml flask with the amount of metal soap solution required to reach the wet point of the system.

After the addition of 100 ml benzene, the flask is shaken for one hour on a " Red Devil " paint shaker before being allowed to stand overnight. In general an equilibrium is reached after 15 min agitation. The excess of metal soap is determined after sedimentation or, where necessary, by centrifuging the pigment and decanting the supernatant liquid. The titration of lead, cobalt, calcium and magnesium is carried out with EDTA in ethanol/benzene solution after Luchesi and Hirn[3].

Products used

Pigments : Kronos RNC and Hombitan R 505 titanium dioxides.

Extender : Socal E2 precipitated calcium carbonate.

Divalent metal soaps : Nuodex Servo octoates and naphthenates, Siccatol octoates, Valirex naphthenates, Barlocher oleates.

Solvent reagents : AR grade : White spirit with 15 per cent aromatics.

Results and discussion

Dispersing effect of divalent metal soaps.

Effect of the nature of the metal : The dispersing effect on rutile titanium dioxide of the Pb, Zr, Zn, Co, Ca, and Mg naphthenates, octoates and oleates in solution in white spirit is shown in Figs. 1, 2 and 3. The amount of solution

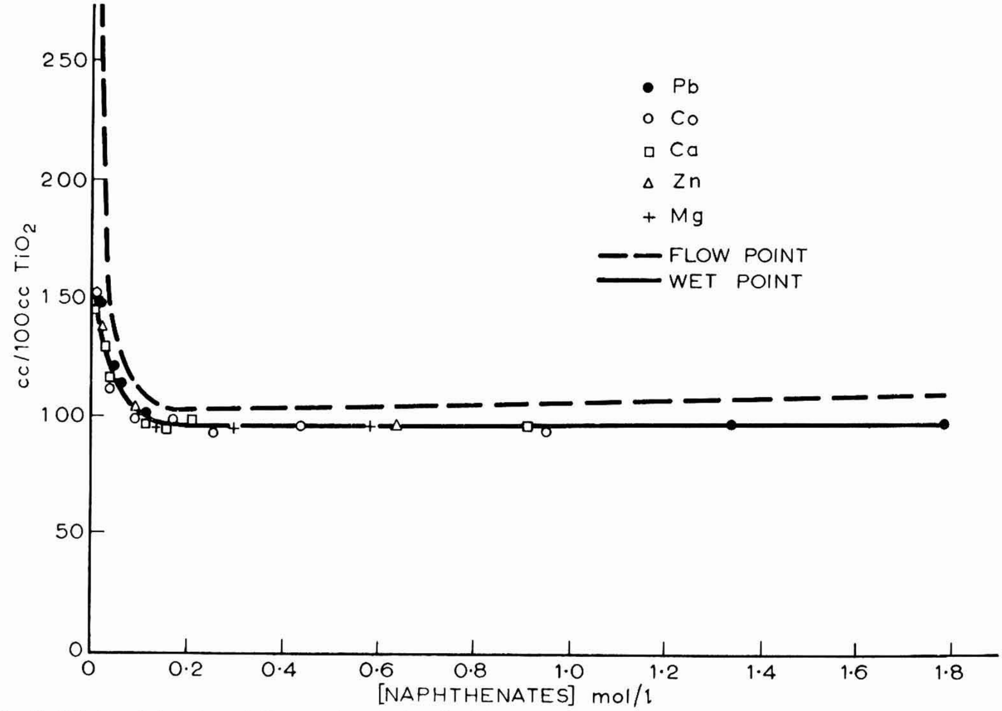

Fig. 2. Wet point (–) and flow point (—) of TiO_2 RNC and naphthenates in white spirit solution

required to reach the wet point and the flow point (cc solution 100 cc pigment) is expressed as a function of the molar concentration of the metal soap in solution.

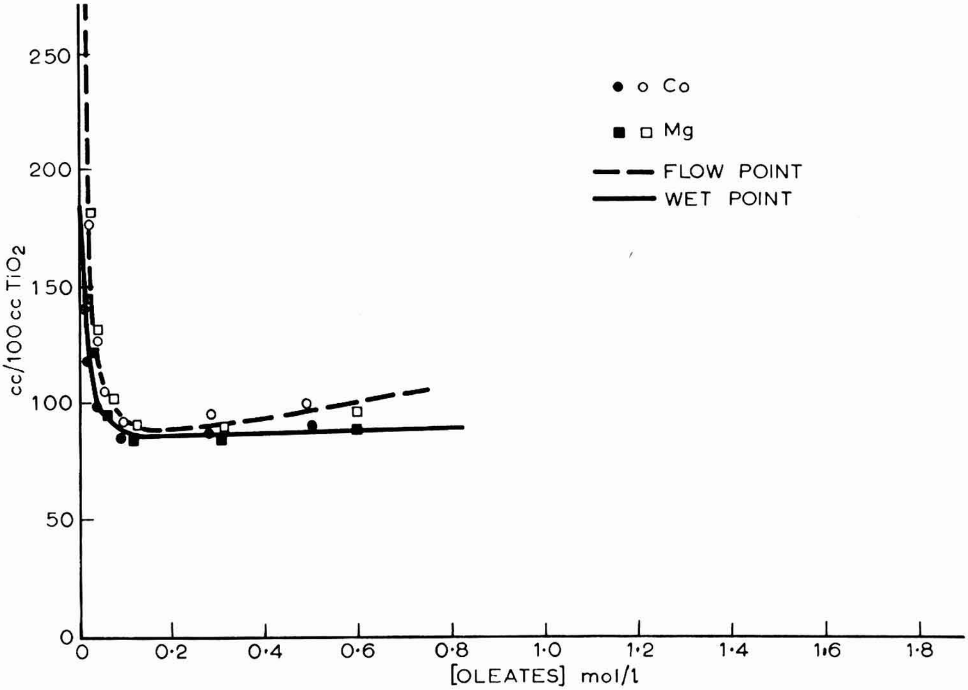

Fig. 3. Wet point (–) and flow point (—) of TiO_2 RNC and oleates in white spirit solution

In each case within one group of divalent metal soaps, the points lie on the same curve, indicating that the nature of the metal has no effect on the dispersing action of divalent metal soaps. This confirms the results found by Kindervater[4] in the case of naphthenates.

From these results it would appear that the dispersing effects attributed in particular to the zinc and calcium naphthenates and octoates, used as grinding agents, are solely due to the fact that in practice these products are used on a weight basis. It is obvious that a unit weight of a light-metal octoate contains a greater number of moles, and consequently equivalent solutions on a weight basis of octoates will show a dispersing effect increasing from lead to calcium (Fig. 4). Identical results have been obtained with precipitated calcium carbonate.

Consequently the divalent metal soaps exhibit the same behaviour as the alkali metal soaps since their replacement by Na^+, K^+ or NH_4^+ does not modify the surface activity to any degree[5].

Effect of the hydrocarbon chain : From Fig. 5 it is seen that the dispersing effect of metal soaps, amongst those studied, increases in the order octoates <naphthenates <oleates.


GOOD HOPE CASTLE
GOOD HOPE CASTLE

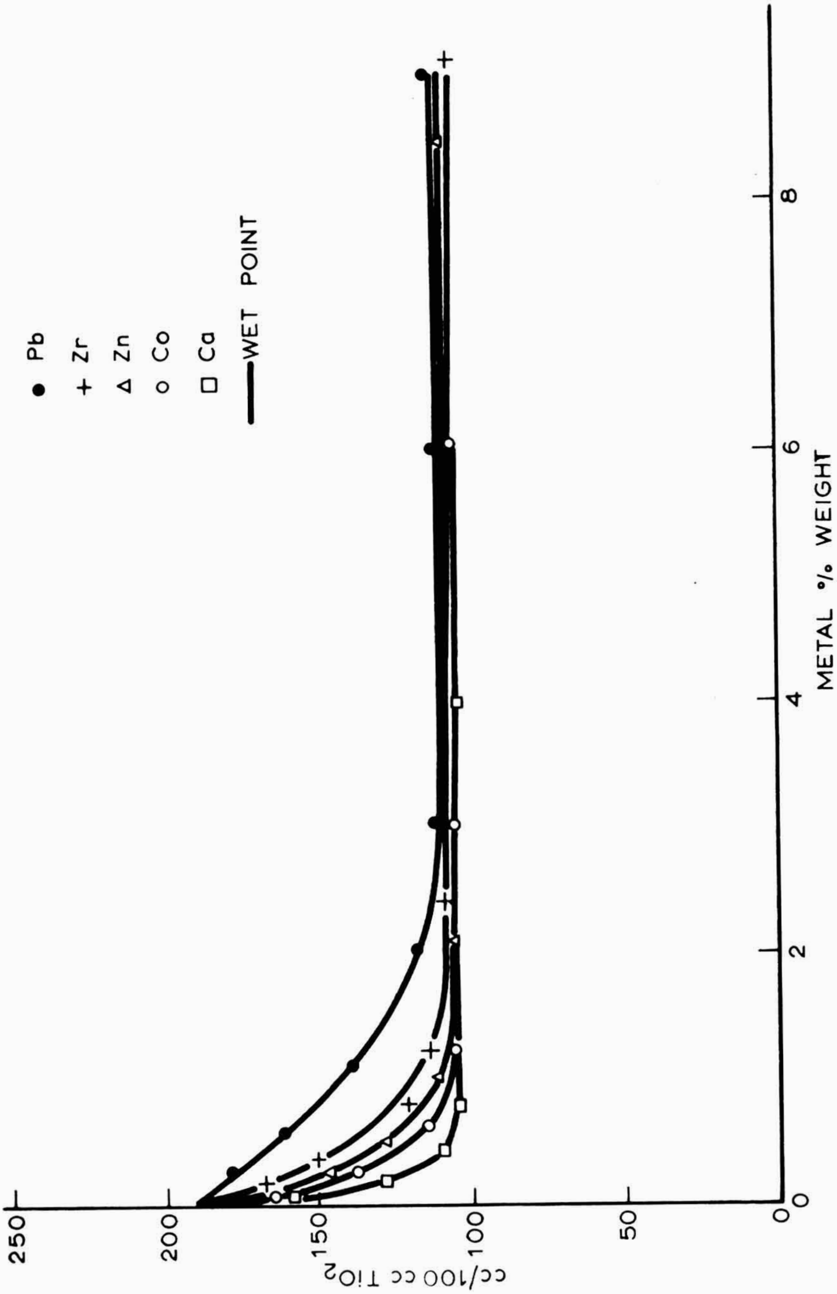

Fig. 4. Wet point of TiO_2 RNC and octoates in white spirit (concentration by weight)

Adsorption

Several authors[4, 6-12] have studied the adsorption of metal soaps, in particular, driers, on pigments. From these papers it is found that :

1. A monomolecular adsorption layer is formed. The metal occupies a site on the surface of the pigment and the hydrocarbon chains are orientated at

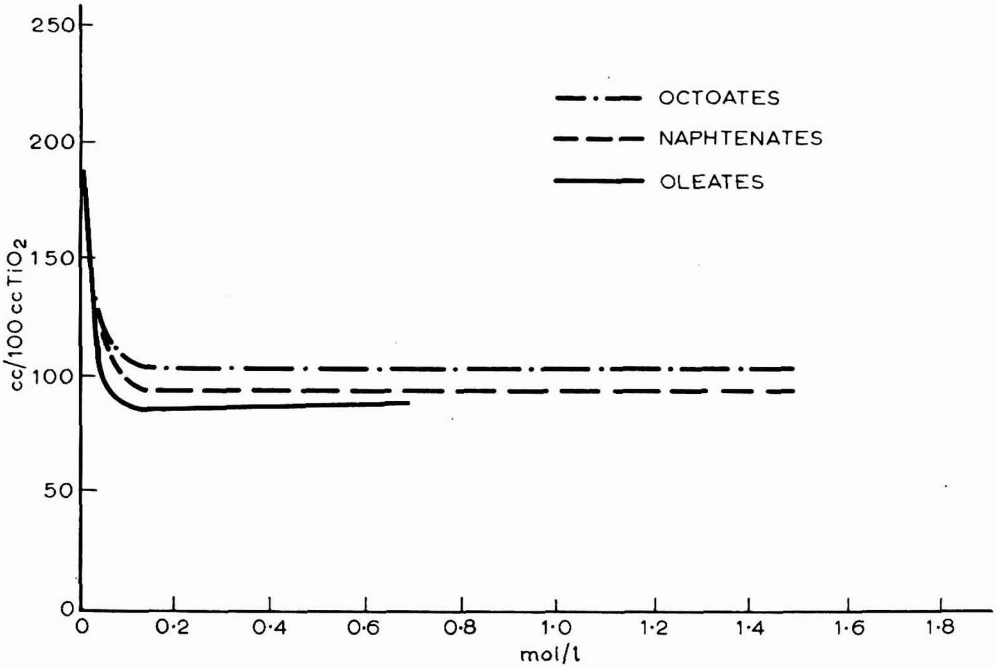

Fig. 5. Wet point of TiO_2 and divalent metal soaps in white spirit solution. Octoates — naphthenates — oleates

right angles to the surface. In this way divalent metal soaps improve the dispersion of hydrophilic pigments in a non-polar medium by giving a hydrophobic character to the pigment surface.

2. There is constant exchange between the adsorbed drier and that in solution, whether of the same metal or of a different metal.

3. A non-polar solvent cannot desorb the drier from the surface of the pigment.

The adsorption values given vary between 0.02 and 0.05 mmols of metal naphthenate or octoate per gram of rutile titanium dioxide. The variation in these results is probably due to the use by the various authors of titanium dioxides whose specific surfaces differed.

The variation of adsorption with the nature of the divalent metal remains a controversial question. According to some[6, 7] it is independent, while according to others[4, 8, 9] it varies with the nature of the metal. Certain authors have carried out experiments with cobalt and calcium radioisotopes[10].

The curves for the adsorption of cobalt, lead and calcium octoates on the surface of titanium dioxide RNC and precipitated calcium carbonate Socal E2 are given in Figs. 6 and 7 respectively. The adsorption of cobalt octoate on titanium dioxide R.505 and magnesium oleate on titanium dioxide RNC are shown in Fig. 8. At initial concentrations lower than those of the critical adsorption, the drier is wholly adsorbed, showing by this its very great affinity for the hydrophilic surfaces of titanium dioxide and calcium carbonate. On TiO_2 it is found that the adsorption of the driers increases with the molecular weight of the metal. On precipitated calcium carbonate the differences in adsorption are minimal and, in fact, close to the experimental error ($\pm$0.001 mmol/g pigment).

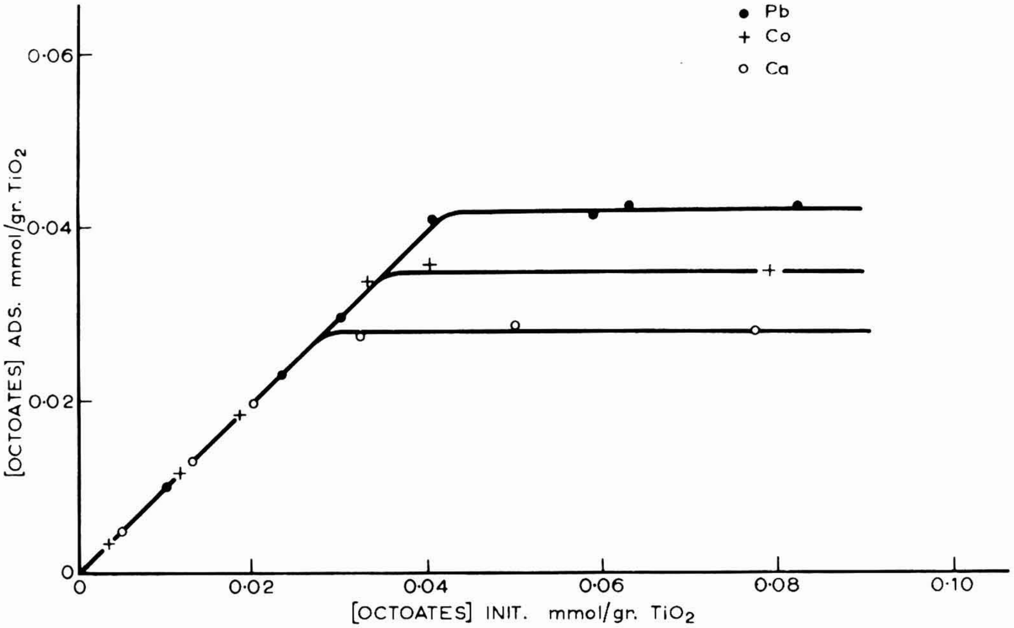

Fig. 6. Adsorption of octoates on TiO_2 RNC (solvent : benzene)

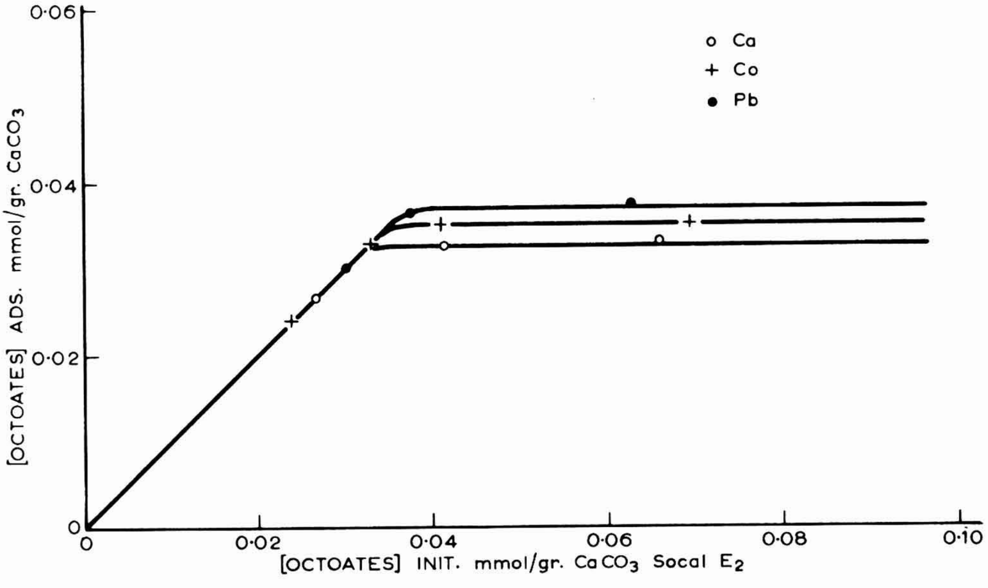

Fig. 7. Adsorption of octoates on $CaCO_3$ Socal E2 (solvent : benzene)

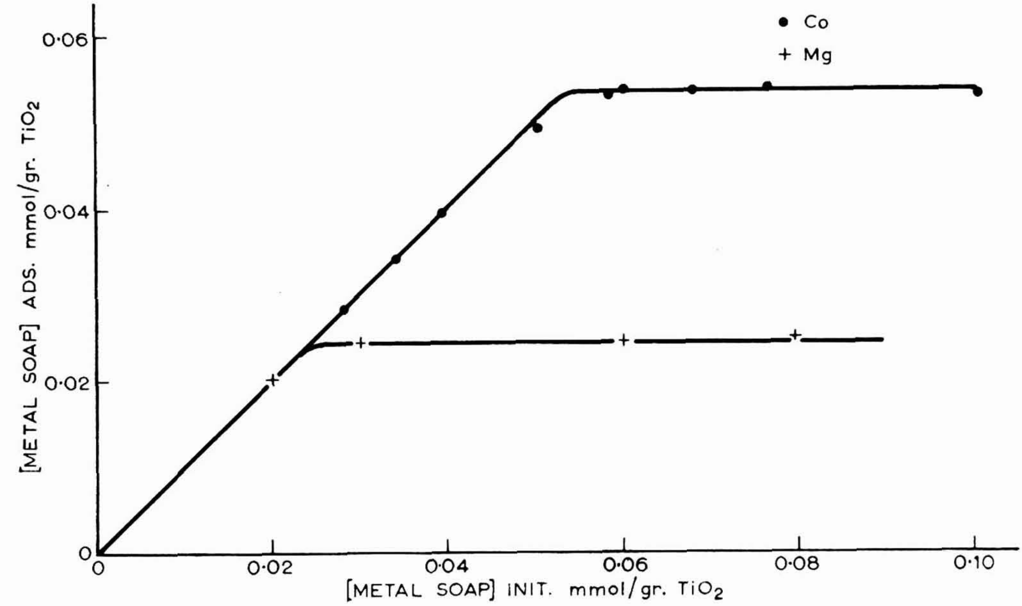

Fig. 8. Adsorption of cobalt octoate (–●–) on TiO_2 R.505: of magnesium oleate (–+–) on TiO_2 RNC (solvent : toluene)

Adsorption and the dispersion of pigments

Various authors have tried to connect the dispersing effect of a substance, with respect to a pigment, to the adsorption of this substance on the surface of the given pigment. Thus the dispersing action of fatty acids, alcohols, amines, metal soaps[13–18], alkyd resins [19–21] and acrylic copolymers [22] has been studied in relation to adsorption on various pigments. A one-to-one correspondence was found by Dintenfass[14] between minimum sedimentation volume and maximum adsorption of octoic and stearic acids on rutile TiO_2. Shütte[19] found a relationship between fineness of grind and degree of adsorption of an alkyd on ZnO. Crowl [23] showed that the minimum amount of surfactant necessary in a certain latex paint corresponded to a monomolecular layer. More often, optimum dispersion was found to correspond only imperfectly, or not at all, to the maximum adsorption of dispersing agent on the pigment.

The systems examined in the course of this study have, however, shown an interesting relationship between adsorption and dispersion.

The comparison of the adsorption curves and the Daniel curves for systems comprising divalent metal soaps in solution in a non-polar solvent and either rutile titanium dioxide or a precipitated calcium carbonate shows a precise relationship between the adsorption and the dispersion. In Figs. 9-12 the amount of the metal soap solution required to reach the wet point consistency is plotted on the ordinate (cc solution/100 cc pigment). The concentration of this solution is plotted on the abscissa (mol/l). In addition the ordinate carries the amount of metal soap adsorbed by the pigment (mmol/100 cc pigment) as a function of the concentration of metal soap solution with which the pigment is mixed to reach the wet point.

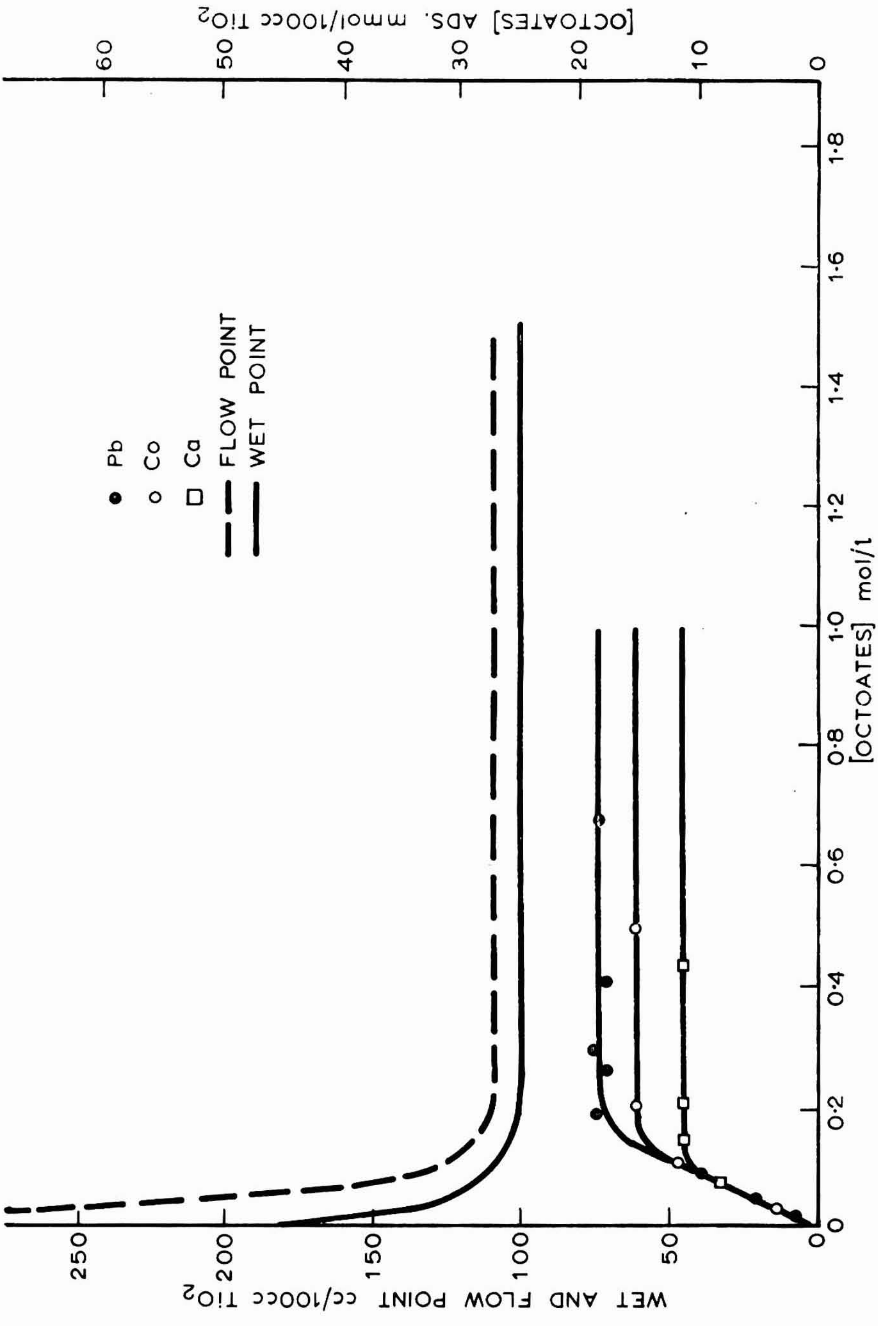

Fig. 9. Wet point (-) and flow point (—) curves of TiO_2 RNC and octoates. Adsorption curves of Pb (●) Co (o) and Ca (□) octoates

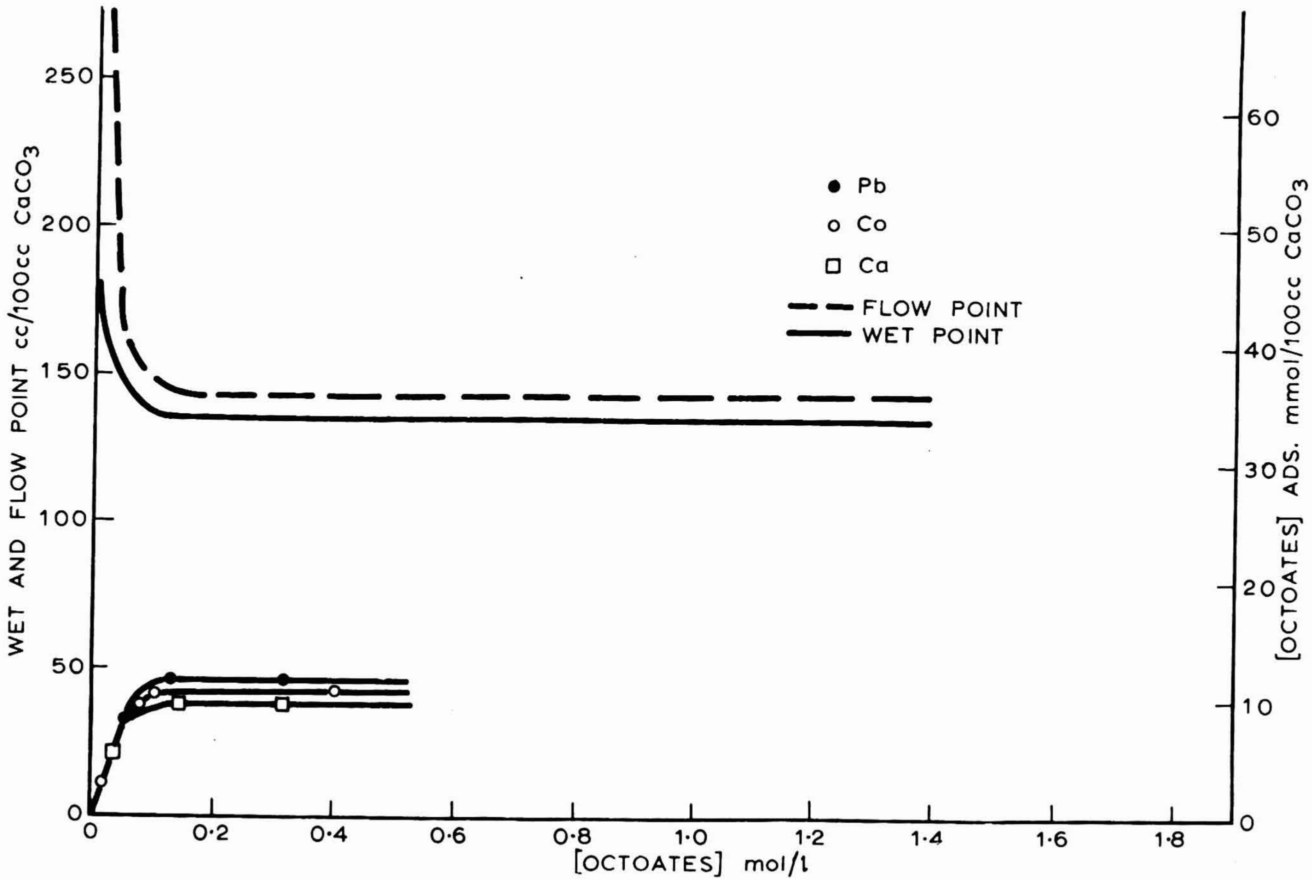

Fig. 10. Wet point (–) and flow point (—) curves of $CaCO_3$ Socal E2 and octoates. Adsorption curves of Pb (●) Co (o) and Ca (□) octoates

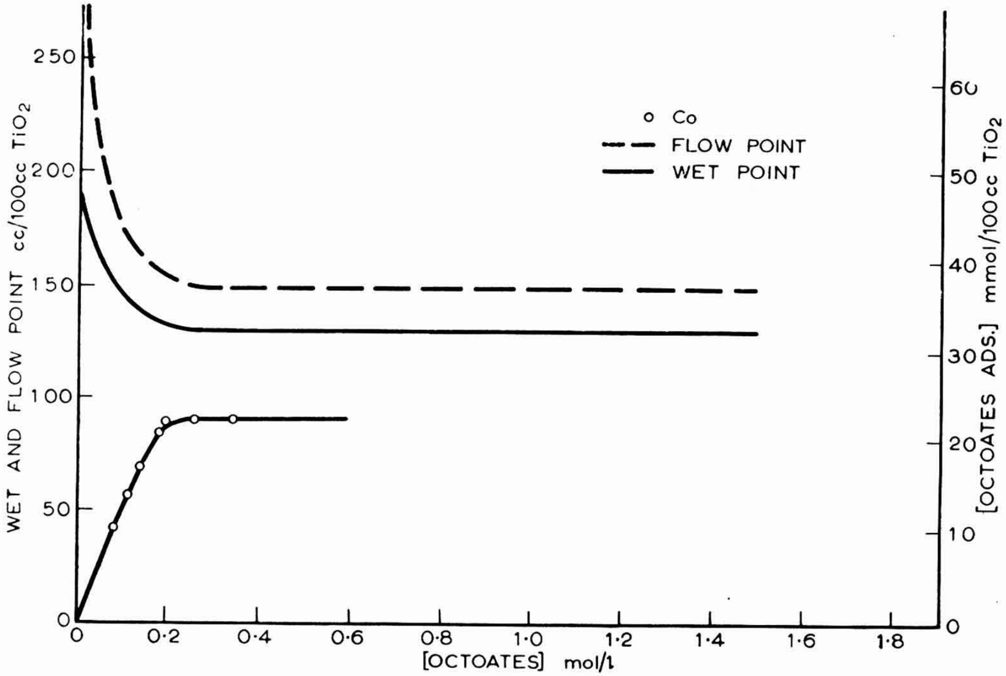

Fig. 11. Wet point (–) and flow point (—) curves of TiO_2 R.505 and Co octoates. Adsorption curves of cobalt octoate (o)

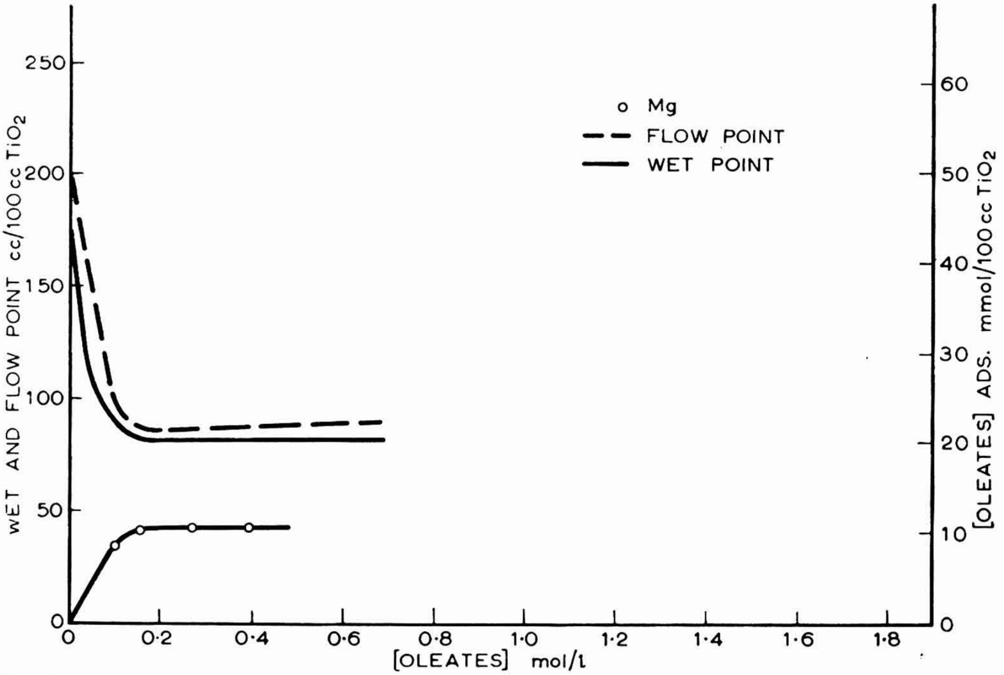

Fig. 12. Wet point (–) and flow point (—) curves of TiO_2 RNC and Mg oleate. Adsorption curve of magnesium oleate (o)

It is necessary to point out that the point of inflexion of the adsorption curves coincides with the point of inflexion of the Daniel curves. Now when the Daniel curves reach the " critical wet point," the point at which the curves become horizontal, the systems under investigation pass from a plastic state to a dilatant state and reach optimum dispersion. The adsorption curves evolve in a parallel manner, the point of critical adsorption corresponding exactly with the " critical wet point."

In the light of this relationship between adsorption and dispersion for a pigment, the independence of the dispersion effect of the nature of the metal is explained by the very close values of the critical adsorption for various metal driers. It is probable that the differences in adsorption between the various metal soaps are too small to influence the dispersion of titanium dioxide and calcium carbonate measured by the wet point and flow point methods. On the other hand the adsorbed molecule, the hydrocarbon chains of which are immersed in the non-polar solvent, establishes the pigment-solvent bond. The nature of these hydrocarbon chains consequently must influence the dispersion, as has been observed.

The relationship obtained between the critical adsorption and the optimum dispersion measured by the Daniel curves seems to be capable of extension to other systems, e.g. to fatty acids and titanium dioxide.

The wet point and flow point curves of an RNC titanium dioxide-octanoic acid system in white spirit (Fig. 13) have the same form as those obtained with

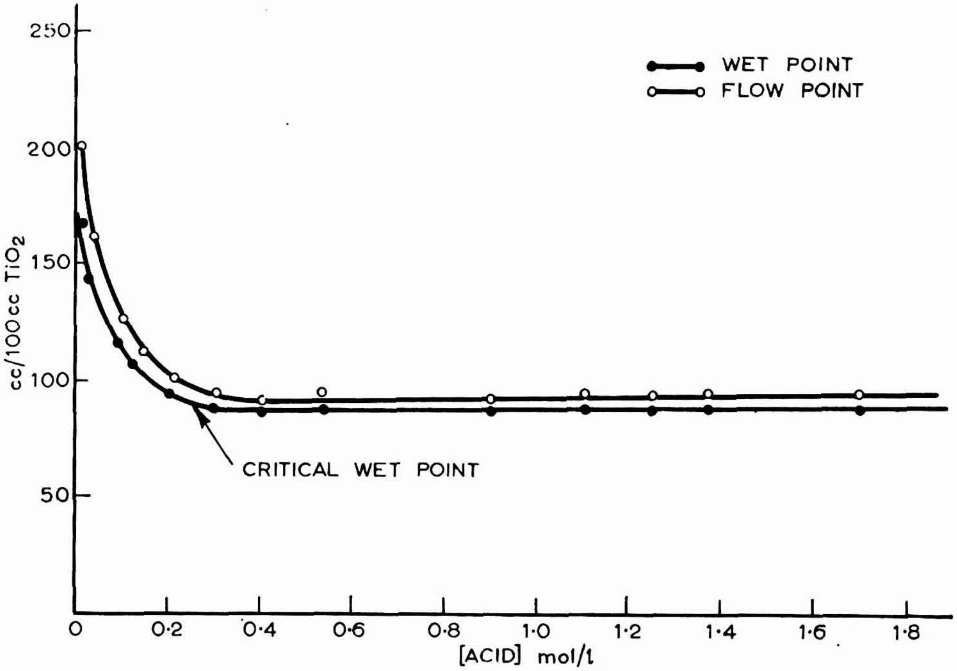

Fig. 13. Wet point (●) and flow point (o) of TiO_2 RNC and octoic acid in white spirit

the drier. Calculation of the amount of acid employed at the " critical wet point " gives $8.5 \pm 1.0 \times 10^{-5}$ mols/g TiO_2. Knowing that the titanium dioxide used has a BET specific surface of 10.5 m^2/g, and assuming that there is a monomolecular adsorption layer, it is possible to calculate the area A_m occupied by a molecule of *n*-octanoic acid on the surface of the titanium dioxide by the formula

$$A_m = \frac{S}{X_m . N}$$

where S is the specific surface in m^2/g.

X_m is the critical adsorption in mol/g pigment.

N is the Avogadro number.

A_m is found to be equal to $20.5 \pm 2Å^2$ which is an area of occupation in excellent agreement with the values given in the literature for linear fatty acids[24, 25].

Since the replacement of X_m by the amount of acid used at the "critical wet point " gives a correct value for the area occupied by this amount of acid, it is proved that the amount of acid necessary to reach the optimum dispersion by the wet point and flow point curves is equal to the amount of acid adsorbed. If these findings are verified by others, with different dispersing agents and pigments, then it becomes possible from the point of inflexion of the wet point curve to estimate the amount of dispersing agent required to satisfy the adsorption by the pigment. The authors are continuing their research in this direction.

Conclusions

In systems comprising rutile titanium dioxide and calcium carbonate, metal soaps and a non-polar solvent, the dispersion as measured by means of the wet point and flow point curves is independent of the nature of the metal but varies with the nature of the hydrocarbon chain of the metal soaps.

Optimum dispersion is achieved when the adsorption of the divalent metal soaps by the pigment or extender reaches the critical value determined by the adsorption curve.

[*Received 21 August 1967*

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The reliability of outdoor exposure tests

By R. Epple

Technical Service Department, Titangesellschaft mbH, Leverkusen

Summary

The results of a number of exposure testing programmes carried out at various sites throughout the world have been used in an attempt to derive estimates of the anticipated durability at one site from those at another and to relate these to practical performance. The inadequacy of a *time factor* relating solely the periods of exposure to a given degree of failure is shown, while more satisfactory predictions can sometimes be made using a linear regression equation. It is thus shown that the performance of paints outdoors depends not only on the overall climatic conditions but on local seasonal and annual variations and the time at which exposure commenced. Rank correlation is used to show that differing climatic conditions affect not only the life of a paint film but also the order of performance relative to other paints.

La sûreté des essais de résistance aux intempéries

Résumé

Les résultats de plusieurs programmes d'essais de résistance aux intempéries, effectués aux divers emplacements autour du monde, ont été utilisés pour tenter de dériver les estimations de durabilité prévue dans un emplacement à partir des résultats en provenance d'un autre, et en même temps de les rapporter au rendement de la peinture dans le pratique. On démontre l'insuffissance d'un *facteur de temps* qui met seulement la durée d'exposition en rapport à un certain degré d'affaiblissement et également que l'on peut faire parfois des prédictions plus satisfaisantes en se servant d'une équation linéaire de régression. Ainsi on démontre que le rendement au dehors de peintures se dépend non seulement du régime total climatérique, mais aussi des variations locales, annuelles et de saison, et à la fois du moment du début de l'exposition. On utilise une corrélation par ordre de rendement pour démontrer que des différences dans les régimes climatériques influencent non seulement la résistance aux intempéries d'une peinture mais aussi son ordre de rendement relatif aux autres peintures.

Die Verlässlichkeit von Bewitterungsversuchen

Zusammenfassung

Es handelt sich darum, die Ergebnisse einer Anzahl von in der ganzen Welt verteilten Bewitterungsstationen zur Prüfung auf Dauerhaftigkeit durchgeführten Programmen zu vergleichen. Dabei war das Ziel aus der voraussichtlichen Wetterbeständigkeit auf einer Station Schlüsse zu ziehen auf die Wetterbeständigkeit einer anderen, um daraus für die Praxis brauchbare Schätzungen zu gewinnen. Die Unzulänglichkeit des sich lediglich auf die Exponierungsperiode beziehenden *Zeitfaktors* hinsichtlich des Grades eines Anstrichschadens wird aufgezeight. Befriedigendere Voraussagen können dagegen manchmal unter Benutzung einer linearen Regressionsgleichung gemacht werden. Auf diese Weise wird demonstriert, dass die Aussenbeständigkeit von Anstrichmitteln nicht nur von allgemeinen Klimaverhältnissen, sondern von örtlichen saisonmässigen und jährlichen Schwankungen, sowie der Jahreszeit zu Beginn der Exponierung abhängig ist. Man benutzt die Rank Korrelation um aufzuzeigen, dass unterschiedliche Klimabedingungen nicht nur die Lebensdauer eines Anstrichfilms beeinflussen, sondern auch die Einordnung ihres Verhaltens im Vergleich mit anderen Anstrichmitteln.

Надежность испытаний выветривания на открытом воздухе

Резюме

На основании результатов ряда испытательных программ по выветриванию на открытом воздухе проведенных в различных местах, были сделаны попытки произвести оценку ожидаемой надежности в том или ином месте и связать зти оценки с рабочими характеристиками. Показаны недостатки временного коэффициента связывающего лишь периоды обнажения с известной степенью повреждения, в то время как более удовлетворительный прогноз может иногда быть сделан при помощи уравнения линейной регрессии. Таким образом показано что рабочие характеристики красок на открытом воздухе зависят не только от общих климатических условий но и от местных сезонных и годичных вариаций и от момента начала выветривания. Применялись классифицированные соотношения чтобы показать что изменения в климатических условиях влияют не только на долговечность красочной пленки но и на порядок рабочих характеристик по сравнению с другими красками.

Introduction

Pigmented systems should ideally be examined for durability in relation to the atmospheric conditions at weathering stations situated within the vicinity of the site of their future practical use. The reliability of the results so obtained is greater in terms of the actual performance of the systems under the weather conditions which will be obtained in use, the closer the conditions at the test site agree with those at the site of application. The use of a *time factor* should render it possible to calculate the life of the system at any given site from the life at the test site.

The examples in the following paper show how far it is possible to use this time factor for such calculations and the errors which may occur. The measures of agreement are based on the linear correlation coefficient and Spearman's rank correlation coefficient. It will be shown that a statistical approach must be adopted.

Observations

When heavy chalking occurs after 3 months with an exterior quality paint, it may fairly be considered grounds for complaint. When this was brought to attention, it was first presumed that the paint manufacturer had used an anatase titanium dioxide in error for a stabilised rutile pigment. After consultation with the paint manufacturer, it was found that this could not be the case. Analysis of the paint in question proved that the correct grade of pigment had been used and both vehicle and pigment/binder ratio were in order.

In order to ascertain what had happened, panels were prepared with some of the rejected material and exposed at the central exposure site at Engstenberg. At the same time, paints prepared in the laboratory to the manufacturer's formulation and using his vehicle were also exposed. Rutile and anatase pigments were included.

None of the paints containing a rutile grade displayed any chalking before the expiration of 9 months. With the reject material and its laboratory-made counterpart, chalking occurred after 11 months. After 18 months when the tests were terminated, chalking had not reached the intensity that the rejected material had shown on site after 3 months.

The difference between the test and site results could be due to the following factors.

1. The complaint referred to paints applied in a residential area in Austria while the test panels were exposed in the Rhineland.

2. The rejected paint was applied in December just before a period of frost while the test paints were applied on 15 August.

There was therefore no correspondence between the actual climatic and application conditions and those of the tests.

Time factor and time function

It would have been expected that about 10 to 12 months would elapse under the test conditions before the onset of chalking, but patently the reliability of such an estimate is cast in doubt by the practical result. Since such doubts relate to the average expected life of paints under various climatic conditions, a *time factor* is suggested as a possible means of improving the reliability. For example, such a time factor would enable calculations of the life of a paint exposed in Norway to be made from the life of that paint exposed in Florida.

A large number of panels exposed in Florida in July 1961 gave an average period free from chalking M_{C8} (months until a chalk rating of 8 was reached) of 11.0 months. For the same paint exposed concurrently at Fredrikstad, the figure was 17.7 months.

A *time factor* can then be calculated as follows :

$$F_t = \frac{17.7}{11.0} = 1.6$$

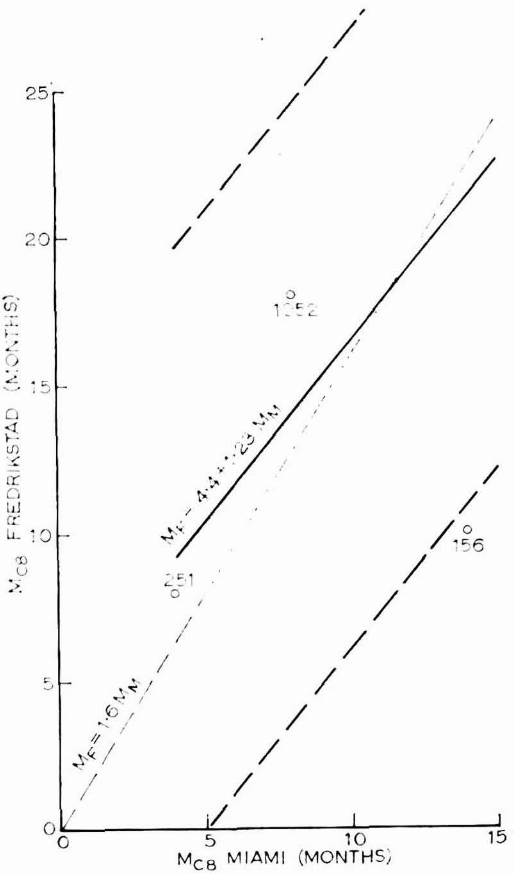


Fig. 1. Chalk-free period at Miami and Fredrikstad

The disadvantage of such a factor is that it is based on the *average* result and not to the performance of *individual* paints having low, medium and high durability. A better relation is possible using a regression equation as a *time function* in which the performance of paints of different durability under two differing climatic conditions may be related. For the example above, the regression equation is (Fig. 1)

$$M_{C8\,(\mathrm{Fred.})} = 4.4 + 1.23\,M_{C8\,(\mathrm{Miami})}$$

This enables the results of Florida exposure to be converted into those for Norway. A paint which has a chalk-free period of 3 months in Florida will on this basis start chalking after 8 months in Norway. If the same conversion be made using only a time factor, F_t, of 1.6, the chalk-free time in

Norway would work out at only 5 months. This is an error of 40 per cent to the paints' disadvantage. The reverse would be true for a paint of high durability which commences to chalk in Florida only after 17 months. The time factor would give in this case 27 months free from chalking in Norway which is too long by 2 months or 7.5 per cent.

It would be even worse if the Florida results were used to calculate those for the Dutch coast by means of the time factor. The average period free from chalking for the paints exposed at Zandvoort was about 12.5 months, giving a time factor, $F_t = \frac{12.5}{11.0} = 1.14$. The chalk-free period for a paint which commenced chalking after three months in Florida would be 3½ months in Holland. If, however, the regression equation (Fig. 2)

$$M_{C8\,(\mathrm{Zand.})} = 7.1 + 0.49\ M_{C8\,(\mathrm{Miami})}$$

be applied, a figure of 8½ months is obtained. The error to this paint's disadvantage would be not 40 per cent but 60 per cent in this case.

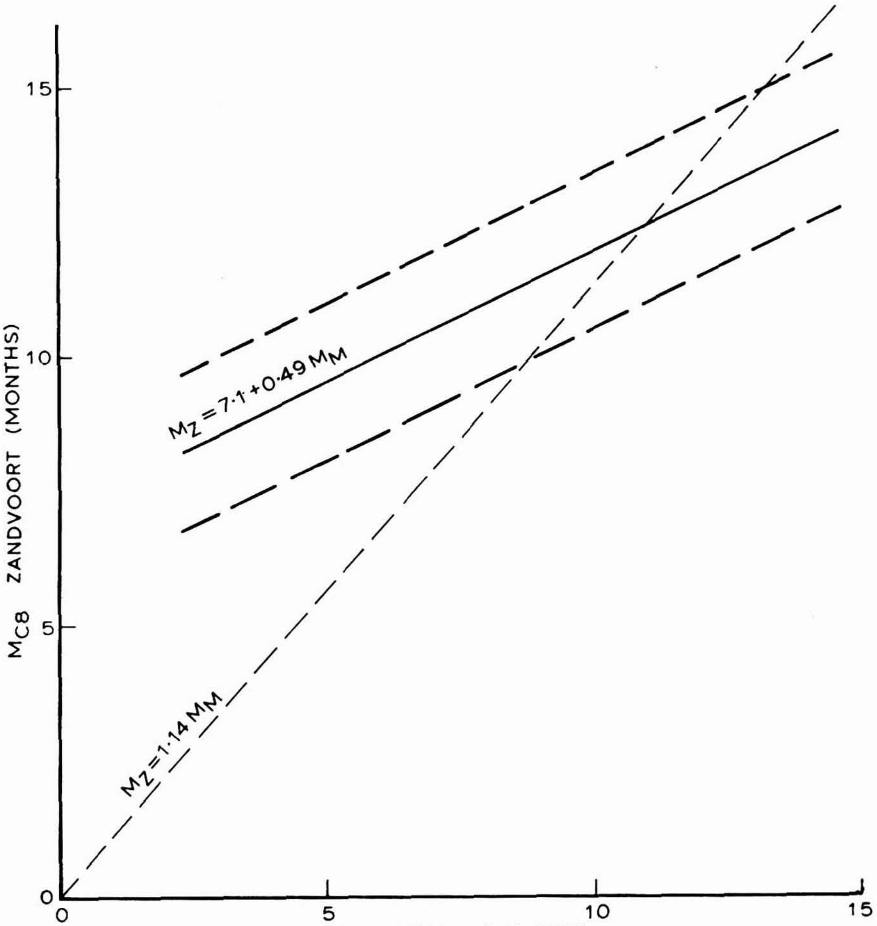


Fig. 2. Chalk-free period at Miami and Zandvoort

Direct comparison of these calculated figures with actual results is not possible for this series of experiments, since the worst panel (long oil soya alkyd, zinc modified rutile pigment of medium stability, 15 per cent PVC) did not start chalking in Florida after 3 months but only after 4 months. Using the time factor for this paint would give 4½ months for the onset of chalking in Holland compared with 9 months using the regression equation. The chalk-free period was 10 months which may be regarded as reasonable agreement.

Figs. 3-7 show that a linear regression function is reasonably justified in all cases as a first approximation.

Fig. 3 shows the times until a chalk rating of 8 was recorded, i.e. until chalking commenced, for Davos against Zandvoort. Theoretically it would be expected that the straight line should pass through the origin, i.e. for the hypothetical case where chalking would start immediately after exposure at both sites, but the fact that the Davos panel started chalking after 4 months does not cause any deviation from linearity.

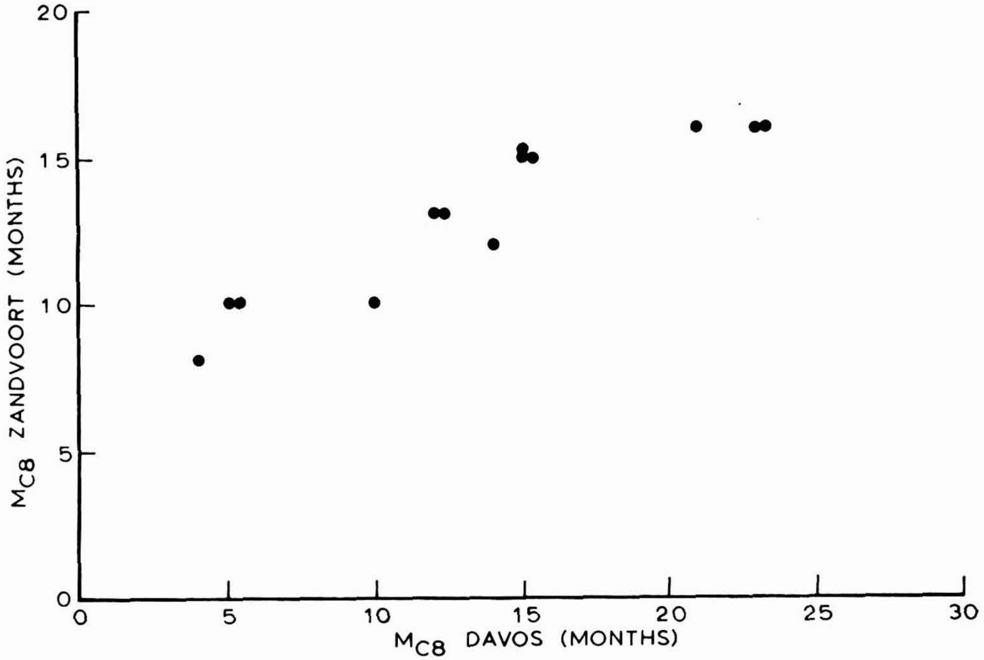


Fig. 3. Chalk-free period at Davos and Zandvoort

Fig. 4 compares the chalking in Florida after 16 months, which was the time when best differentiation between panels was possible, and that found correspondingly at Engstenberg at 20 months. Again, a linear relationship would appear to be justified.

The same is true for the total chalk ratings of another series of panels at Miami and Engstenberg (Fig. 5) and also for gloss after 18 months at London and 24 months at Fredrikstad, at which times best differentiation was possible (Fig. 6).

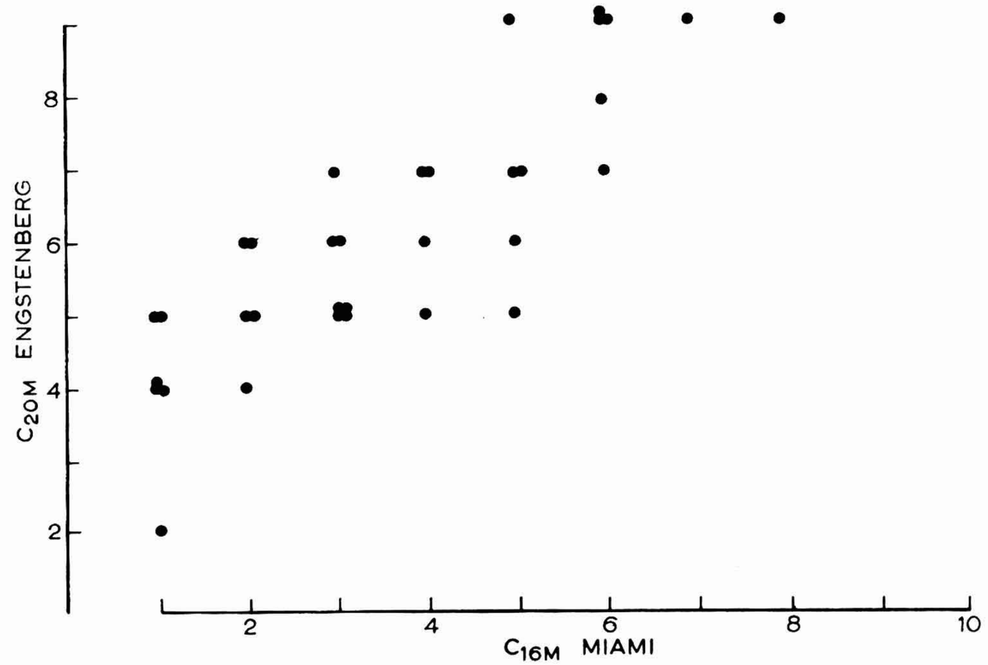


Fig. 4. Chalking at Miami and Engstenberg

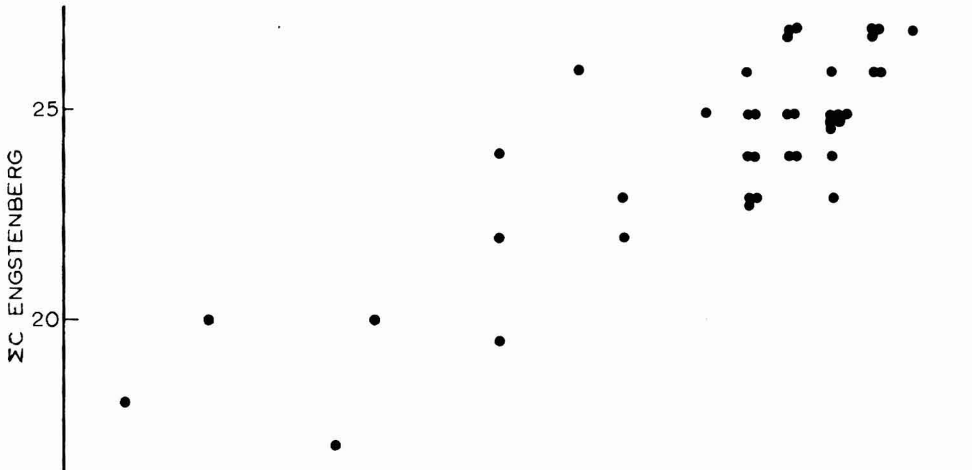
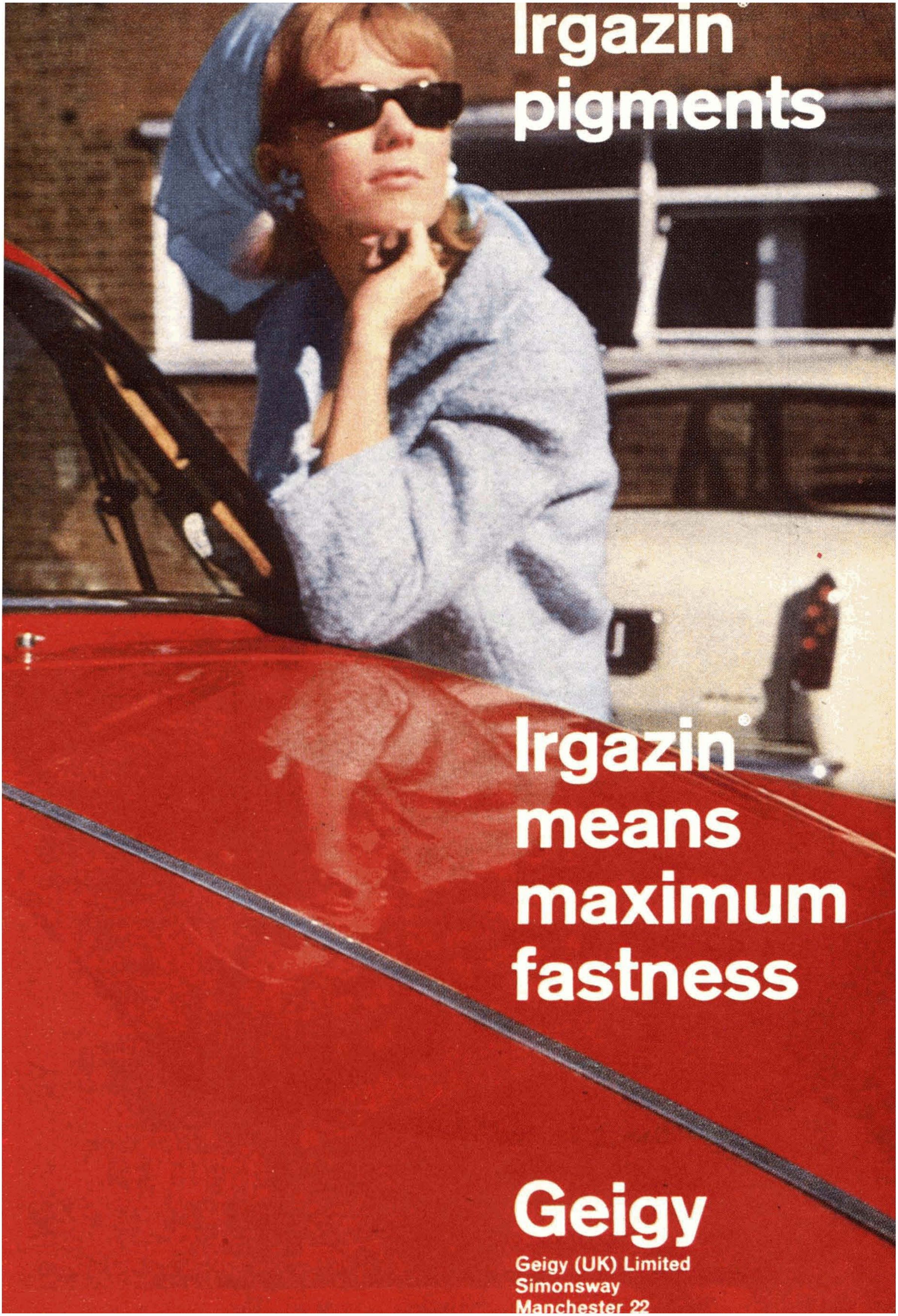


Fig. 5. Accumulated chalking, ΣC, at Miami and Engstenberg



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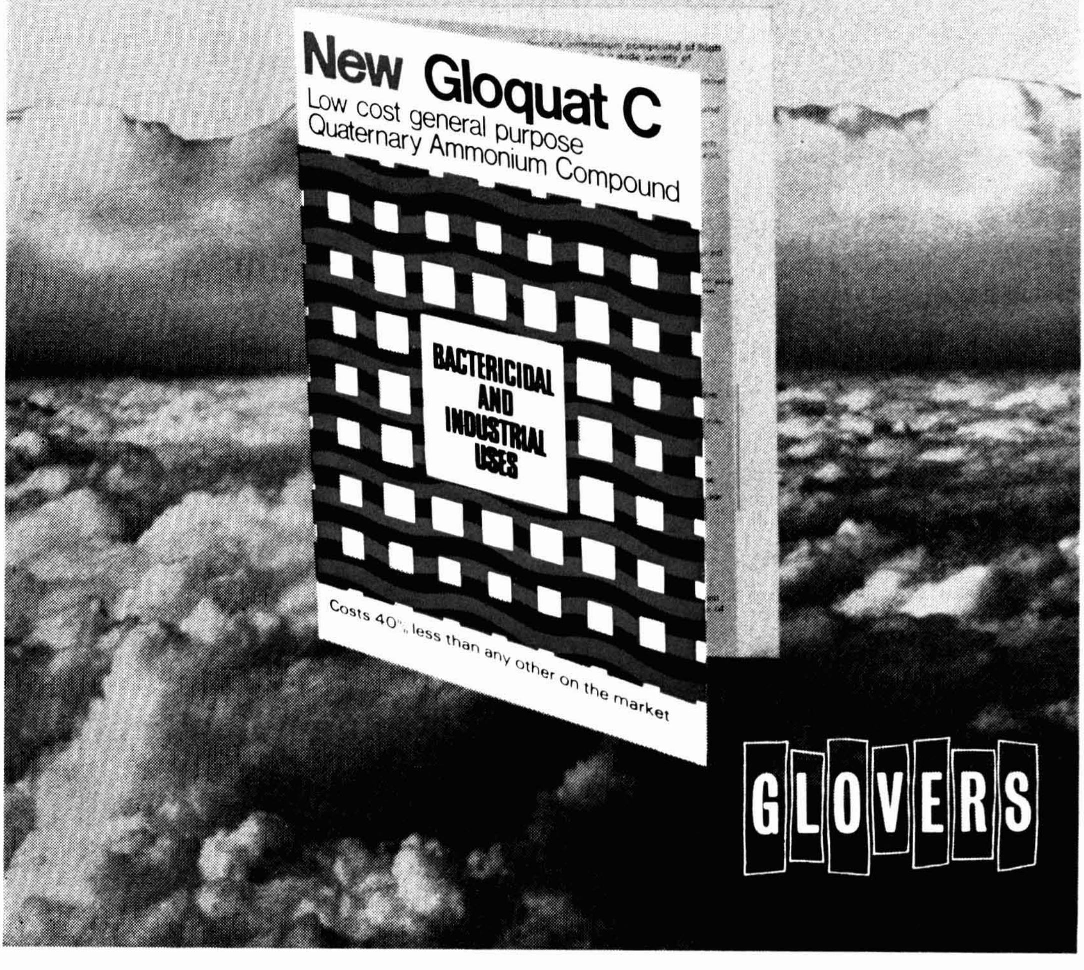
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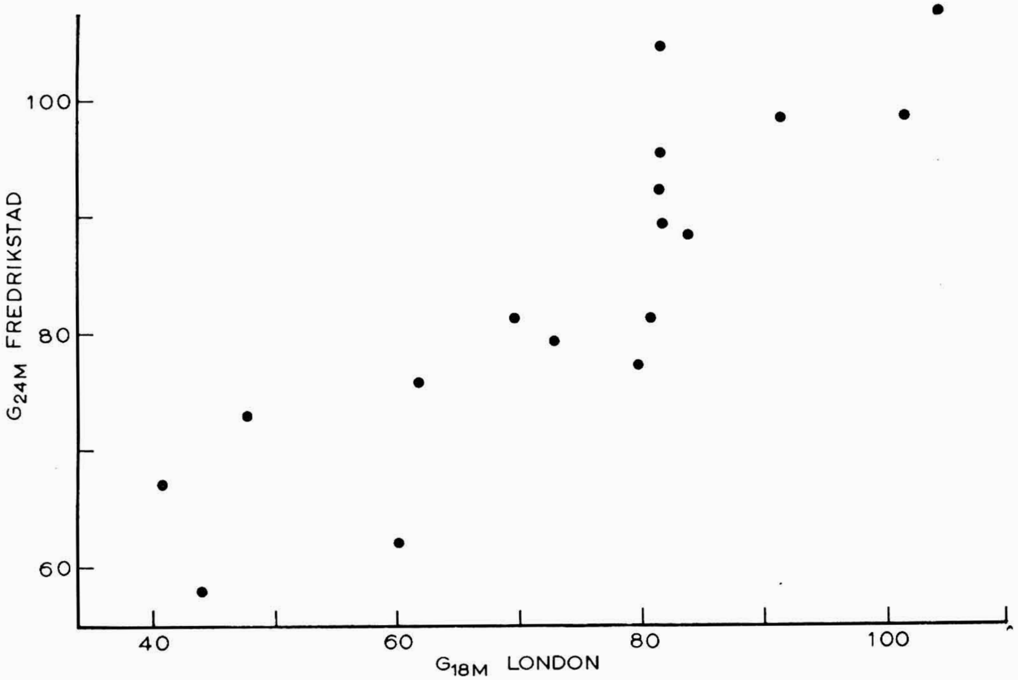


Fig. 6. Gloss at Fredrikstad and London after n months

Fig. 7 gives the retained gloss as a percentage of the original gloss of paints pigmented in different ways for Friedrichshafen against Westerland on the Island of Sylt. Again a linear relationship satisfies the data.

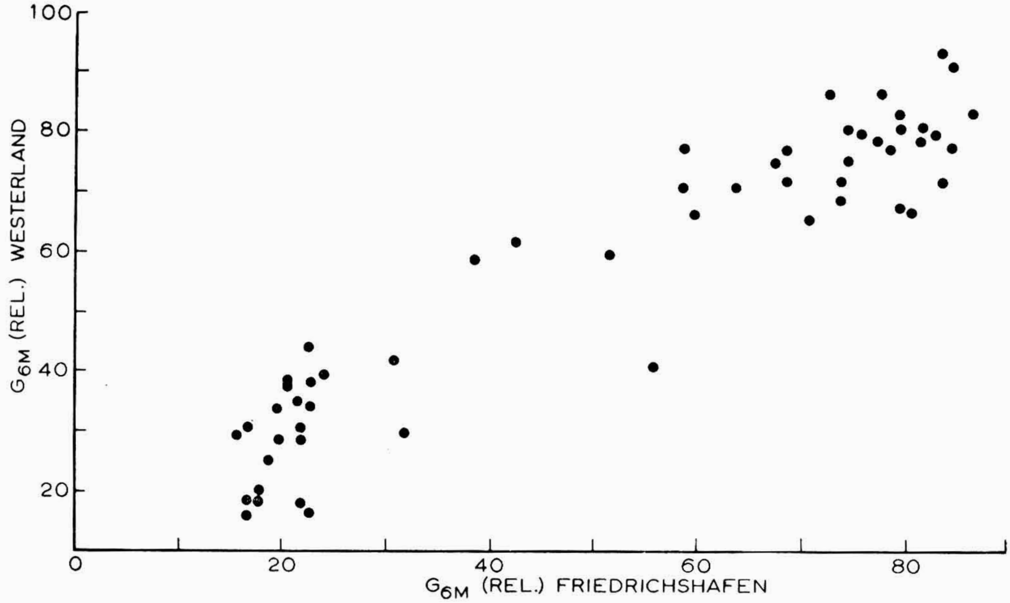


Fig. 7. Retained gloss at Friedrichshafen and Westerland after 6 months

From this, it would seem that it should no longer be difficult to predict the behaviour of paints, say, from exposure data at Manchester, at any place in the world provided that the performance at the test site had been correlated with that of the particular site in question.

When the complaint mentioned earlier was received from Austria, inadequate information was available on the performance of paints under the conditions being obtained there. The nearest possible, but poor, comparison had to be based on exposure results at Engstenberg and at Davos in the Swiss Alps. The regression equation for the time in question was

$$M_{C8\,(\mathrm{Davos})} = 3.95\ M_{C8\,(\mathrm{Engst.})} - 36.8$$

As already recorded, the rejected paint required 11 months to chalk at Engstenberg and this would give a figure for the Swiss Alps as follows :

$$M_{C8\,(\mathrm{Davos})} = 3.95 \times 11 - 36.8$$

$$= 43.4 - 36.8 = 6.6 \text{ months.}$$

Since the climatic conditions of the particular place in Austria are generally less severe than those at Davos which has an elevation of 5,100 feet, it indicated that the conditions during application must be responsible for the poor performance in question. The 95 per cent confidence limits for the estimated chalk-free period are ±4.8 months and this leads to a minimum of 2 months before chalking would have commenced ; a chalk rating of 2 after 3 months would thus never have been anticipated.

Seasonal effects

This then raises the question whether it be sufficient to know where a paint is to be exposed and whether it is also necessary for any reliable prognostication of performance to know when it is to be exposed. Fig. 8 shows the mean values for chalk-free time (M_{C8}) of a series of paints exposed at one site in March, June, September and December. All the paints were produced initially from the same pigment and supply of vehicle. There were 21 paints differing only in the grade of TiO_2 used. Control tests showed no difference between the series. Nevertheless, the diagram shows marked differences in the average chalk-free periods. This ranges from 10 months for the series exposed in December 1958, to 18½ months for those exposed in December 1956. While no seasonal effect is revealed by these data, the differences found must surely be due to the actual weather conditions during exposure.

Loss of gloss (Fig. 9) shows this more clearly. By far the greatest loss of gloss occurred with those panels exposed in March 1957 and 1958. After only 6 months, i.e. spring and summer, the gloss had dropped to 50 per cent of its initial value but the panels exposed in December required 9 months on average to reach this level. For those panels exposed in September, gloss was lost even more slowly, requiring 11-12 months, i.e. autumn, winter and spring to lose 50 per cent gloss, while about the same rate, 10½-11 months, was found for those exposed in June.

Thus any prediction of the possible life of a paint at any one place from data obtained at a given test site must involve not only the relationship between the sites but also the seasonal and actual weather conditions at both places.

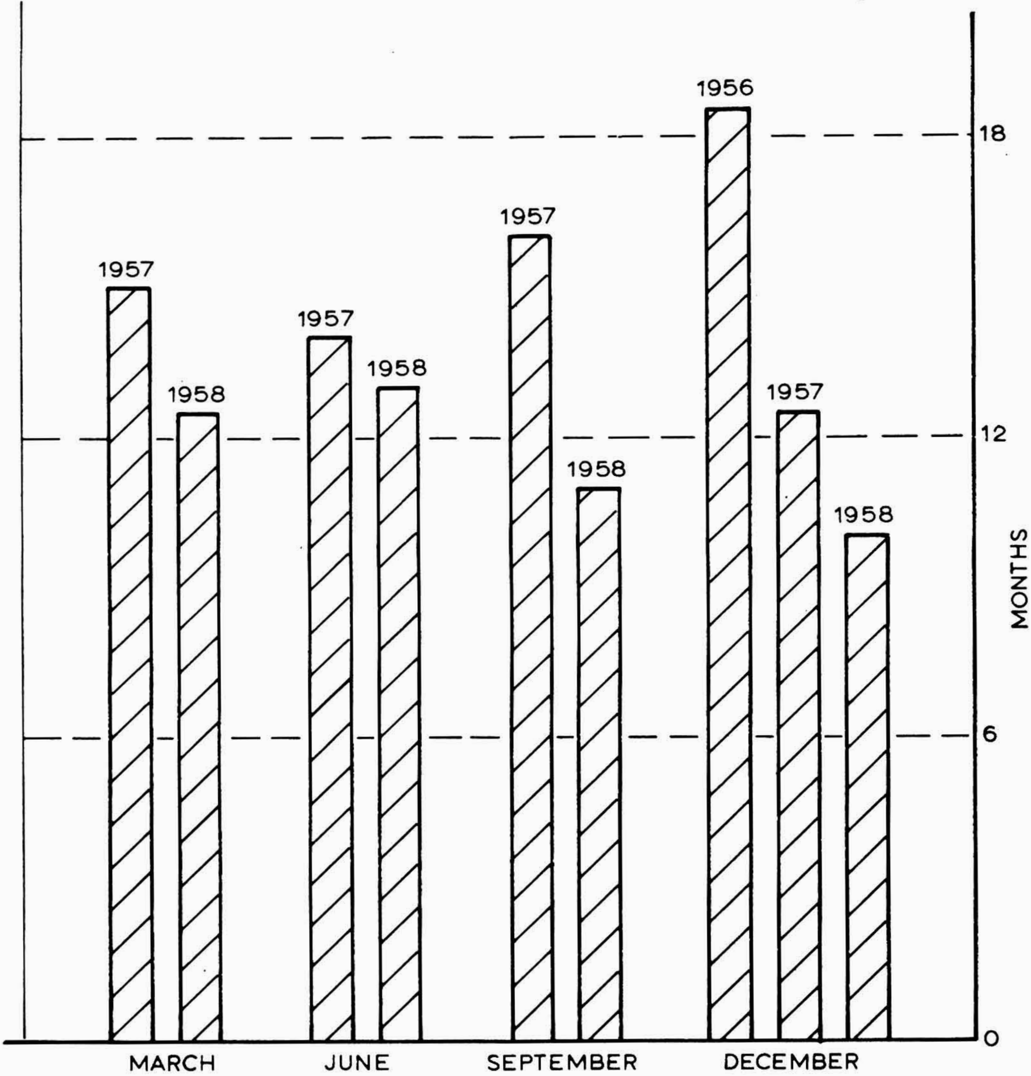


Fig. 8. Chalk-free period and commencement of exposure

Reliability of the time function

So far, only the *average* behaviour of paints taken from evaluations of a wide series of similar paints has been discussed, but consideration will now be given to the performance of *individual* paints. In general, the paint manufacturer is not so much interested in knowing the overall behaviour of finishes, based on long oil soya alkyds for example, as in the performance of a newly developed product against previously available materials. Both the resin and pigment manufacturers are faced with similar problems.

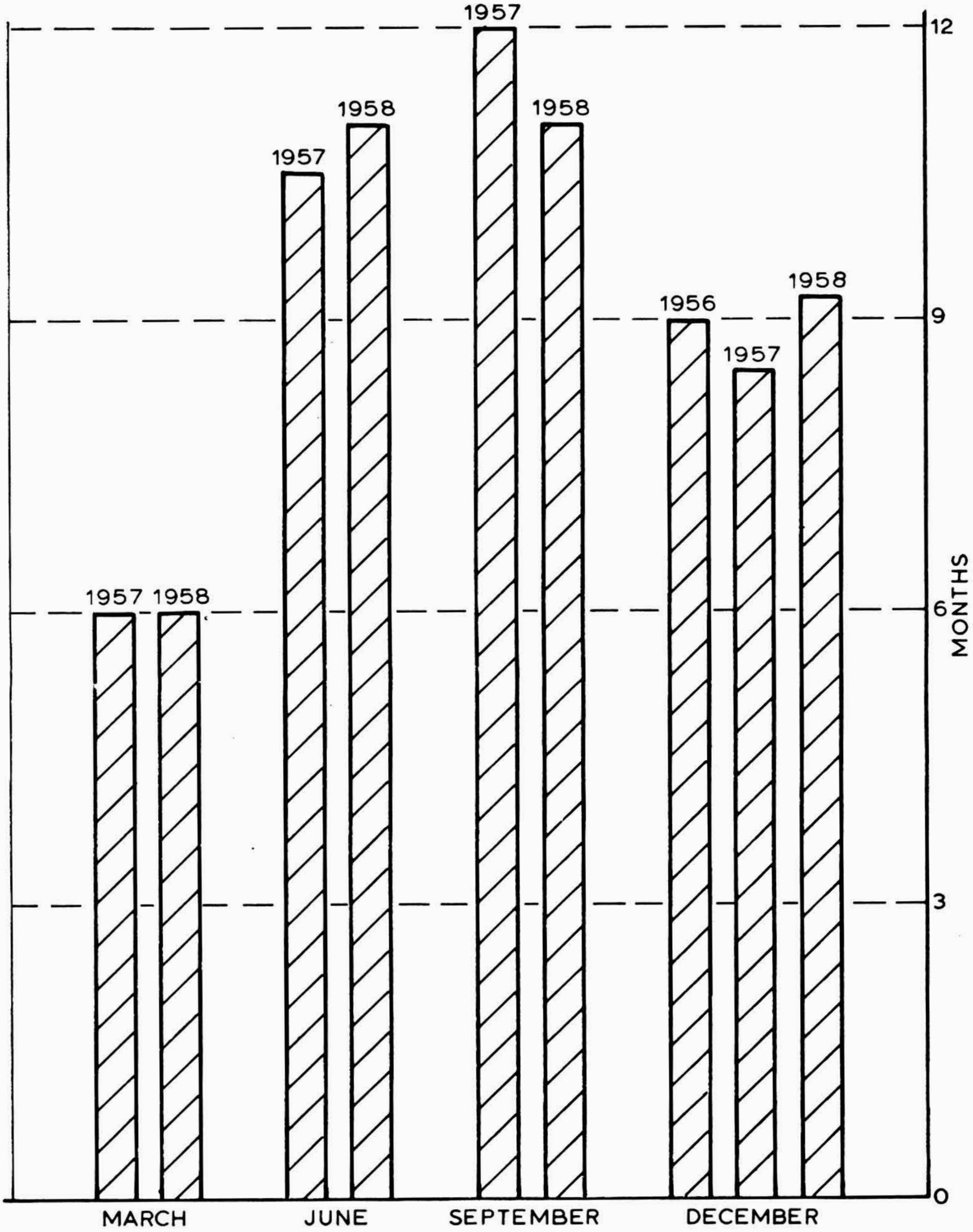


Fig. 9. Time to lose 50 per cent of initial gloss and commencement of exposure

Even if the conditions under which the material had to perform were to be specified, it is not sufficient to decide whether a given material is superior or inferior to another merely from results of the tests and the confidence limits. To compare the behaviour at two different places, the confidence limits for both sites must be taken into account. These are reflected in the confidence limits of the regression equation, and these have been plotted in Figs. 1 and 2. These limits are placed from ±2× standard deviation from the line and should include 95 per cent of all the results.

Fig. 2 shows the chalk-free periods found at Miami and Zandvoort for series No. 308. The confidence limits are ±1.4 months, i.e. a range of 2.8 months. Thus, it may be expected with 95 per cent probability that a paint showing a chalk-free time in Florida of 10 months will not chalk at Zandvoort for 12±1.4 months, i.e. between 10½ and 13½ months. This may, of course, only be predicted with assurance if all the other factors accord with those of series No. 308.

The *correlation coefficient*, r, which is a measure of the extent of the correlation between the two sets of results, is 0.93 and this suggests that the prediction of life at Zandvoort from the Miami data is quite reliable.

This is not so for predictions for Fredrikstad from Miami shown in Fig. 1 where the correlation coefficient is only 0.63 and the confidence limits cover 21.2 months. There is 95 per cent probability therefore that a paint remaining chalk-free for 10 months in Florida will not chalk at Fredrikstad for 16.5±10.6 months, i.e. between 6 and 27 months. Such an estimate is, of course, completely valueless.

Three additional points have been included in Fig. 1 and these refer to three paints pigmented with different grades of TiO_2 designated 156, 251 and 1052. Their durability is given by their German, British and US manufacturers as 156, highest chalking resistance, 251, good chalking resistance, and 1052, high resistance to chalking. This is in agreement with the performance found at Miami, namely :

251	period free from chalking	4 months
1052	period free from chalking	8 months
156	period free from chalking	14 months

The regression equation, $M_{C8(Fred.)} = 4.4 + 1.23M_{C8(Miami)}$ gives the following estimates for Fredrikstad.

251	app. 9½ months
1052	app. 14 months
156	app. 22 months

The times actually found at Fredrikstad were as follows :

251	8 months
1052	18 months
156	10 months

Pigment 156 performs, therefore, rather better in Fredrikstad than 251, but it is markedly inferior to 1052. Hence it is not simply a question of the absolute life of a paint which depends on the place of exposure but also the relationship between paints since different relative orders of performance have been obtained for Fredrikstad and Miami.

The tables indicate some of the deviations which can be obtained. Table 1 refers to paints made with different pigments which all gave a chalk rating of 4, i.e. medium to severe chalking, after exposure for 8 months in Malaysia under tropical conditions. The PVC was 15 per cent and the vehicle a long oil linseed alkyd. Hence, in Malaysia, the chalk resistance of all the pigments was the same.

Table 1

Performance at Miami of paints which gave a rating of 4 after 8 months in Malaysia

Pigment	Chalk rating after 8 months Miami
1156	9
1161	9
VP 649	8
65	7
VP 57	6
951	6
54/1	6
162	5
154	5
152	5
754	5
753	5
54/2	4
VP 52	4
160	4
452	4
453	4
652	4

Performance in Florida, however, was completely different. While six of the paints also reached a chalk rating of 4, the others gave ratings up to 9 where it is impossible to state whether chalking has in fact started or not.

It would, however, be fallacious to conclude from this that the Malaysian climate not only causes more rapid deterioration of paints but also minimises differences between paints, for a similar but contrary situation has also been met. Table 2 shows a series of paints, all of which were rated 4 for chalking

Table 2

Performance in Malaysia of paints which gave a rating of 4 after 8 months in Miami

Pigment	Chalk rating after 8 months Malaysia
1054	1
VP 1052	2
1052/1	2
52/1	2
251	3
162	4
153	4
152	4
754	4
753	4
1052/2	5
52/2	5

after exposure for 8 months in Miami. In Malaysia though the ratings ranged from 1 to 5. In both series, the differences lie outside the deviations of the test method and are thus real.

It is also known that such differences can occur not only between climates such as tropical and sub-tropical but also for any single climatic zone. Table 3 shows a selection of results drawn from a larger series giving the order of gloss after a given period of exposure. Paints based on pigments 1156, 54/1 and 452 have been selected which gave equivalent performance in Malaysia (Table 1). In Miami, 1156 gave the best results, followed by 54/1 and 452.

Table 3

Rankings according to retained gloss

Exposure station	Months	Pigment 1156	 54/1	 452
Engstenberg	12	20	13	17
Friedrichshafen	10	20	15	11
Fredrikstad	24	9	11	19
Manchester	12	6	5	20
Hurst Castle	12	15	10	19
London	14	4	8	16

The same order was obtained in London after 14 months and Fredrikstad after 24 months. At Friedrichshafen, Lake Constance, however, the reverse was the case, 1156 being 20th, 54/1, 15th, and 452, 11th. At Manchester, on the south coast of England and at Engstenberg, pigment 54/1 was the best.

Such diversity of results is discouraging and the more they occur, the less assurance is there in inferring the results to be expected at one site from those obtained at another.

Rank correlation

Spearman's rank correlation cofficient : can be used with data of this type. It is calculated from the sums of squares of deviations of the ranks according to the formula :

$$\rho = 1 - 6 \frac{\Sigma d^2}{n (n^2 - 1)}$$

where n indicates the number of possible ranks and d the difference between corresponding rankings. Fig. 10 shows this function for $n=4$. The connection between the number of inversions in rank order and the rank correlation coefficient, again for $n=4$, is given in Fig. 11. Table 4 shows the dependence of the rank correlation coefficient on the number of differences in rank, the number of inversions and the actual ranks. Comparing the rank correlation coefficients for the comparison between Malaysia and Miami, and those for the various sites in England and on the Continent, it will be seen that they are all of the same order (Table 5). The coefficient for Malaysia and Miami is also included, its value being +0.82, while those for the European sites lay between +0.66 and +0.95.

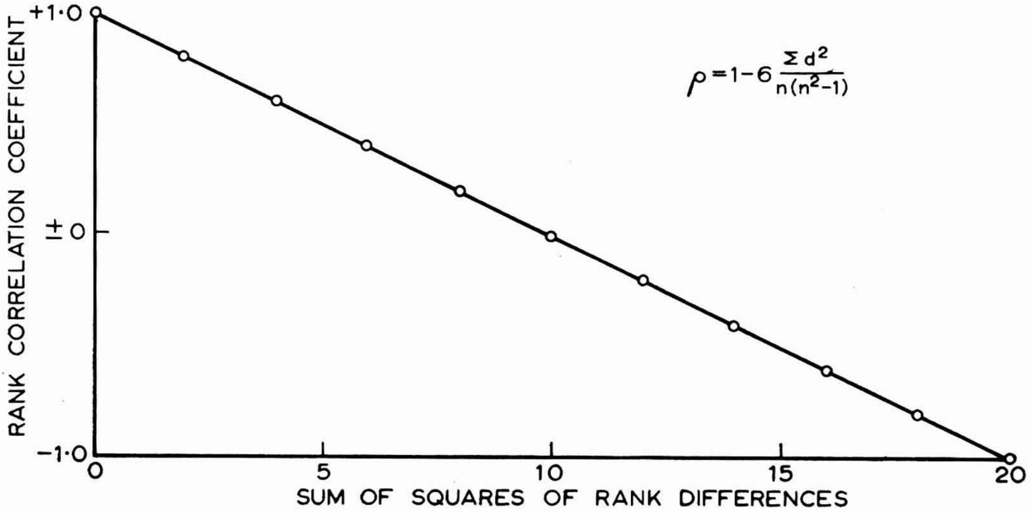


Fig. 10. Rank correlation coefficient ρ (Spearman) against sums of squares of rank differences Σd^2 (n=4)

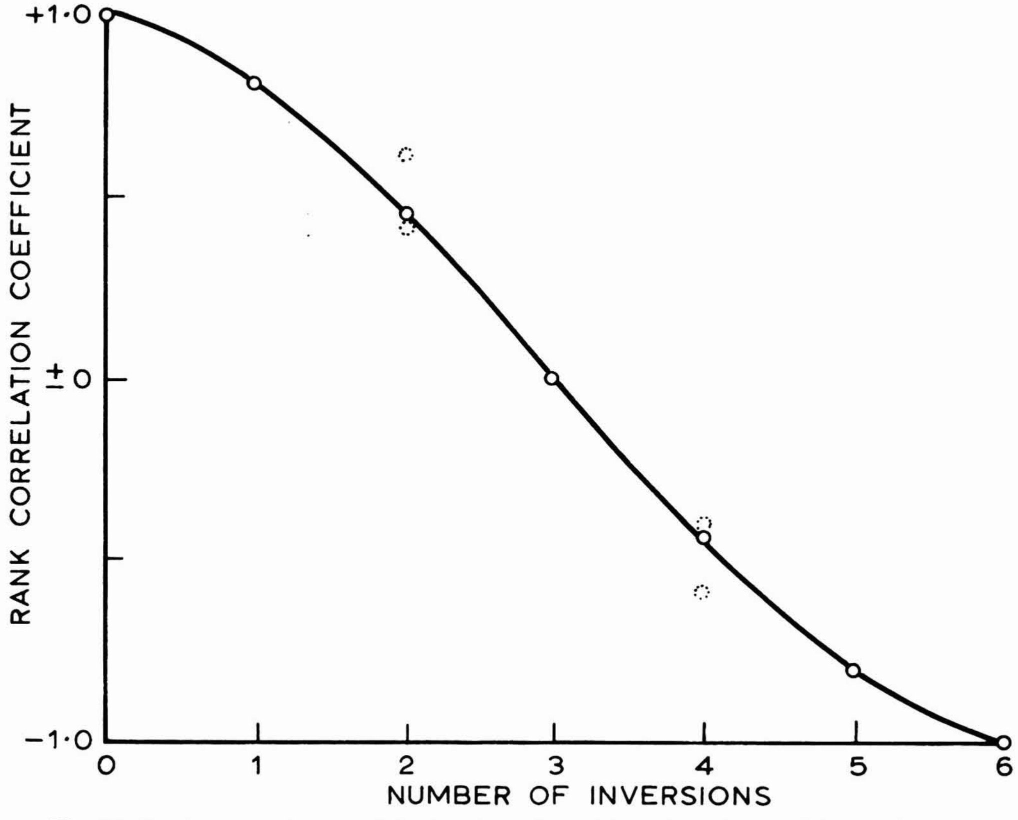


Fig. 11. Rank correlation coefficient and number of inversions for n=4 (see Table 4)

Table 4

Construction of rank correlation coefficient

Comparative sequence*				Number of inversions	Number of rank differences of				Rank correlation coefficient
					0	1	2	3	ρ
1	2	3	4	0	4	0	0	0	+1.0
1	2	4	3	1	2	2	0	0	+0.8
1	3	2	4	1	2	2	0	0	+0.8
1	3	4	2	2	1	2	1	0	+0.4
1	4	2	3	2	1	2	1	0	+0.4
1	4	3	2	3	2	0	2	0	+0.2
2	1	3	4	1	2	2	0	0	+0.8
2	1	4	3	2	0	4	0	0	+0.6
2	3	1	4	2	1	1	1	0	+0.4
2	3	4	1	3	0	3	0	1	−0.2
2	4	1	3	3	0	0	4	0	±0.0
2	4	3	1	4	1	1	1	1	−0.4
3	1	2	4	2	1	2	1	0	+0.4
3	1	4	2	3	0	2	2	0	±0.0
3	2	1	4	3	2	0	2	0	+0.2
3	2	4	1	4	1	1	1	1	−0.4
3	4	1	2	4	0	0	4	0	−0.6
3	4	2	1	5	0	1	2	1	−0.8
4	1	2	3	3	0	3	0	1	−0.2
4	1	3	2	4	1	1	1	1	−0.4
4	2	1	3	4	1	1	1	1	−0.4
4	2	3	1	4	2	0	0	2	−0.8
4	3	1	2	5	0	1	2	1	−0.8
4	3	2	1	6	0	2	0	2	−1.0

* Basic sequence : 1 2 3 4

Table 5

Rank correlation coefficient for chalking

Exposure station		Rank correlation coefficient ρ
Friedrichshafen	Hurst Castle	+0.95
Friedrichshafen	London	+0.89
Friedrichshafen	Engstenberg	+0.88
Engstenberg	London	+0.88
Engstenberg	Hurst Castle	+0.87
Engstenberg	Manchester	+0.86
Friedrichshafen	Fredrikstad	+0.84
Hurst Castle	Fredrikstad	+0.83
Hurst Castle	London	+0.83
Malaysia	**Miami**	**+0.82**
Manchester	London	+0.80
Engstenberg	Fredrikstad	+0.78
Friedrichshafen	Manchester	+0.72
Hurst Castle	Manchester	+0.69
Fredrikstad	London	+0.68
Fredrikstad	Manchester	+0.66

Even larger differences were found between the rank correlation coefficients for gloss retention which varied from +0.04 to 0.81 as shown in Table 6. Although the positive sign at 0.04 suggests that there is no general inversion of the order of performance at London compared with Friedrichshafen, this value is so low that no parallelism between the two sets of results would seem to exist.

Table 6

Rank correlation coefficient for retained gloss

Exposure station		Rank correlation coefficient ρ
Engstenberg	Fredrikstad	+0.81
Manchester	London	+0.81
Manchester	Fredrikstad	+0.77
Engstenberg	Friedrichshafen	+0.76
Engstenberg	Manchester	+0.63
Friedrichshafen	Fredrikstad	+0.62
Friedrichshafen	Hurst Castle	+0.58
Friedrichshafen	Hurst Castle	+0.58
Fredrikstad	Hurst Castle	+0.57
Fredrikstad	London	+0.54
Engstenberg	Hurst Castle	+0.48
Manchester	Hurst Castle	+0.43
Engstenberg	London	+0.39
Friedrichshafen	Manchester	+0.24
Hurst Castle	London	+0.08
Friedrichshafen	London	+0.04

Conclusions

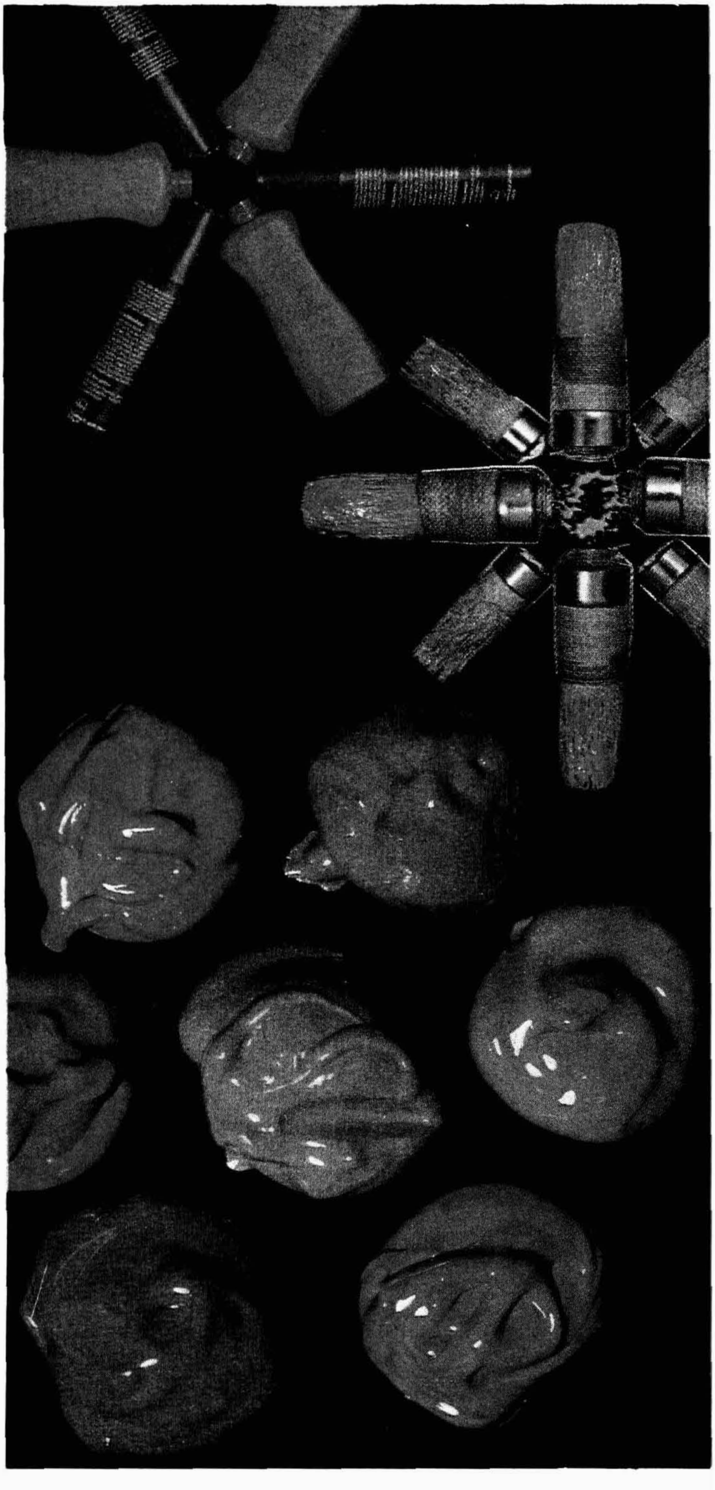
Statements regarding the life of pigmented systems based on test conditions other than those of actual use are questionable in two respects :

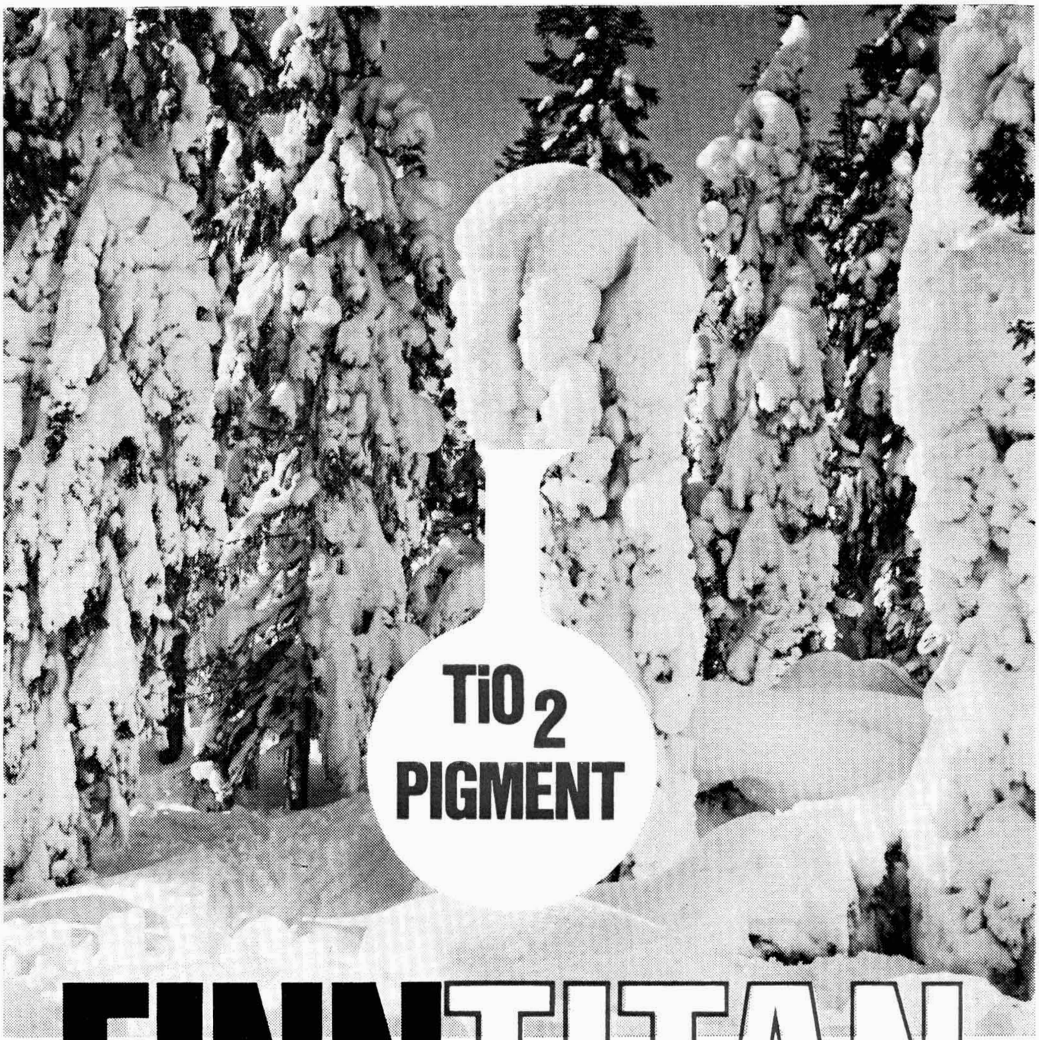
1. *absolutely* in the sense that the anticipated life in practice is derived from test results.
2. *relatively* in that the order of performance need not be the same under different conditions.

It is obvious that to determine the life of a material, agreement between the test and application climates is the first prerequisite for obtaining reliable indications and this reliability decreases as the two climatic conditions depart from one another both in respect of daily and annual variations.

When the test site is situated in an altogether different climatic zone, it becomes necessary to establish the extent of the correlation (i.e. time function) between the two climates for the particular type of material and so to derive estimates for the performance under actual conditions from that of the test site. The reliability to be attached to such estimates depends on the value of the correlation coefficient, the extent to which the material used is representative and, as is generally known, by the number of experiments.

The statistical methods involved require that, for a high degree of reliability in estimating the life of a material, a correspondingly large number of

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experiments must be carried out and if the test programme needs to be curtailed then some qualification of the resulting estimate must be made. The choice of these two alternatives depends on the purpose of any given test and the facilities available.

Appendix

1. *Exposure sites*

Engstenberg.	Central exposure station of Titangesellschaft mbH, on the bight of Cologne, Rhineland.
Fredrikstad.	Exposure station of Titan Co. A.S., on Oslo Fjord, Southern Norway.
Friedrichshafen.	Exposure station of Titangesellschaft mbH, on the Northern Shore, Lake Constance, Southern Germany.
Hurst Castle.	Exposure station of the Paint Research Station, near Bournemouth, South Coast of England.
Johore Bahru.	Tropical exposure station, Malaysia.
Manchester.	Exposure station of the Paint Research Station, North West England.
Miami.	Exposure station of the Subtropical Testing Service, West Coast of Florida.
Teddington.	Exposure station of the Paint Research Station, near London.
Westerland.	Exposure station of Titangesellschaft mbH, on Sylt on the North Sea Coast.
Zandvoort.	Exposure station of Chem. Techn. Adviesbureaus (CTAB), West Coast of the Netherlands.

2. *Terms and definitions*

Fig. 12 shows the normal course of chalking. The broken line is that of the actual readings and the full line represents an ideal curve. The chalk ratings are based on the strength of an impression using the Kempf method[1]. The range between the strength of the impression from a very heavily chalked film and that from one displaying no chalking is divided into ten equal divisions. 10 therefore represents no chalking and 1 very severe chalking[2]. The graph indicates the following definitions.

2.1. *Time until chalk rating 8*

This is denoted by M_{C8} and is a measure of the time until the onset of chalking, i.e. the chalk free period. It was found impracticable to use a rating of 9 for this purpose since the impressions obtained are so weak that accurate distinction between an impression due to chalk and one due to dust is not easily made.

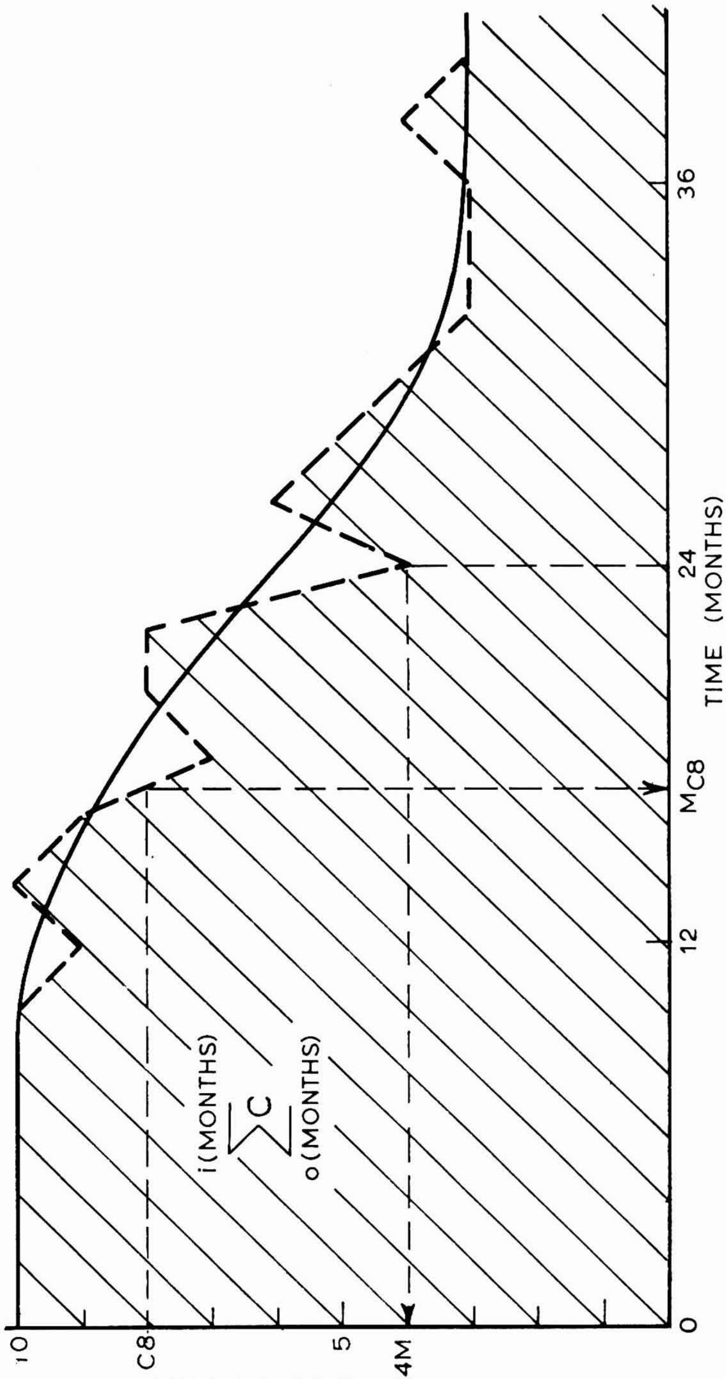

Fig. 12. "Chalking Curve" illustrating chalk-free period (M_{C8}), chalk rating after 24 months (C_{24}) and accumulated chalking (ΣC)

2.2. *Chalk rating after a given time*

In addition to the chalk free period, interest also attaches to the state of the paint after any given time. In Fig. 12 this is illustrated by the extent of chalking after 24 months, designated C_{24M}.

2.3. *Accumulated chalking*

For more accurate distinctions, accumulated chalking, $\Sigma\ C_L$, may be used. This is the sum of all chalk ratings recorded in the course of the exposure trial.

The corresponding characteristics for gloss are, for example, $M_{G25(rel)}$, months until the retained gloss is 25 per cent of the initial gloss, $G_{8M(abs)}$, the gloss after 8 months, and $G_{8M(rel)}$, the gloss after 8 months as a percentage of the initial gloss.

Other aspects of paint deterioration may also be denoted similarly, e.g. the change in brightness (greying) or in tone (yellowing) or the reduction in film thickness.

[*Received 4 March 1967*

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J. Oil Col. Chem. Assoc. 1968, 51, 232-253

The nature of synthetic swelling clays and their use in emulsion paint

By J. Taylor and B. S. Neumann

Laporte Industries Ltd., Stallingborough, Lincolnshire

Summary

The development of a class of synthetic swelling clays is described and an account given of their chemical, physical and rheological properties. The application of these materials as gelling agents for emulsion paints is discussed and the interaction of the clays with other common ingredients of such paints is studied.

Le caractère des argiles synthétiques colloidales et leur utilisation aux peintures-émulsions

Résumé

On décrit la mise au point d'un type d'argiles synthétiques colloidales, et l'on rend compte de leurs caractéristiques chimiques, physiques et rhéologiques. On discute l'utilisation de ces substances en tant qu'agents gélifiants en peintures-émulsions et l'on fait une étude sur l'interaction des argiles avec d'autres constituants ordinaires de telles peintures.

Die Beschaffenheit Synthetischer Quellen der Tonsorten und ihrer Anwendung in Dispersionsfarben

Zusammenfassung

Die Entwicklung einer Gruppe künstlicher quellender Tonsorten wird beschrieben, und es werden Angaben über deren chemische, physikalische und rheologische Eigenschaften gemacht. Der Einsatz dieser Erzeugnisse als Gellierungsmittel für Dispersionsfarben wird besprochen, und die Reaktion derartigen Tons mit anderen üblichen Bestandteilen solcher Anstrichmittel untersucht.

Характер синтетических разбухающих глин и их применение в эмульсионных красках

Резюме

Описывается развитие класса синтетических разбухающих глин и перечисляются их химические, физические и реологические свойства. Обсужлается применение этих материалов в качестве желатинирующих средств для эмульсионных красок и изучается взаимодействие этих глин с другими обычными примесями таких красок.

Introduction

This paper describes a new material, a swelling clay which has been named Laponite*.

At first view, the synthesis of such a very ordinary material as a clay on an industrial scale seems difficult to justify. The synthesis of drugs, dyes and plastics, on the other hand, is easily justified. These are either expensive

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*Laponite is a Registered Trade Mark.

materials used in small quantities, or completely new kinds of materials not existing in nature. Clays are usually considered inexpensive, are used in large quantities and are very abundant in nature in various forms.

There are, however, natural clays which are neither abundant nor cheap, and which have specially useful properties, making them valuable for industrial applications. One such clay is hectorite. It is a good swelling clay, forms thixotropic gels in water which can be used for introducing structure into homogeneous fluids or suspensions ; it disperses almost to colloidal dimensions ; it is heat-stable, and has some other interesting properties. Unfortunately it occurs in a very impure form, requiring a complicated wet purification process. Complete removal of all the fine, gritty particles of the accessory minerals, such as dolomite and quartz, is almost impossible. Hence pure hectorite is a worthwhile target for synthesis.

In general, one can justify the synthesis of a clay mineral if some or all of the following conditions are fulfilled :

(1) the availability of the natural clay suffers from geographical (or political) restrictions.

(2) the natural clay is expensive, while the synthetic clay can be made from readily available materials at a reasonable cost.

(3) the natural clay is impure or non-uniform, while synthesis can produce a pure and homogeneous substance.

(4) the physical or chemical properties of the synthetic clay are superior to those of the natural clay.

All these points are important. The first two, concerned with availability and economy, may sometimes be considered sufficient, but, in the long run, it can safely be assumed that a really sound basis for starting up synthetic clay manufacture exists only when the other two conditions concerned with the quality of the product are satisfied. In fact, superior quality is the most important factor ; so the ideal target was not to make synthetically a copy of an existing natural clay, but a new clay-like material, having valuable, or even specially tailored, properties, which cannot be found in a natural substance.

The first problem was to synthesise a substance which could be identified as structurally conforming to the montmorillonite group of clay minerals to which all the swelling clays, including hectorite, belong, and this had to be done by a practicable method.

Literature search indicated that the synthesis of clay minerals had been studied from the academic point of view by a number of investigators. There were in existence quite active centres working on this subject in France, in Belgium, and in the USA, and earlier pioneering work had been done in Germany. All these centres were apparently working to a large extent on different approaches. Although the general interest was largely academic, there was also, significantly, a project sponsored by the API in the USA.

The methods used for synthesis by the various groups of investigators can be briefly summarised as follows :

(1) *Low temperature, low concentration, very long reaction time*, as practised by the French and Belgian workers[1, 2, 3, 8]. The reason given for the very low concentration is the low solubility of silica in water. Only very small quantities have ever been made by this method.

(2) *High temperature, high pressure, long reaction time*, as practised largely by groups centred round Pennsylvania State University[4]. The pressures are up to 3 kb. As in the case of (1), only very small quantities have been made.

(3) *Fusion, very high temperature, long reaction time*. There is one interesting USA publication on this subject[5]. The products obtained are rather impure.

(4) *Gel synthesis*, also of American origin[6]. Fresh, amorphous, electrolyte-free silica and metal hydroxide are contacted in water at moderate pressure in presence of alkalis or fluoride. This is a very tedious and expensive procedure, but not beyond the possibility of application on the production scale.

(5) *Co-precipitation*. The nuclei of the clay crystals are obtained by direct precipitation from electrolyte solutions which may be further treated to improve their crystallinity. Fundamentally this is the simplest method. One rather complex version of it was briefly tried in Germany in 1941[7], but did not result in much success. Since then, it has been generally considered as an unpromising approach and it has even been stated that " it seems impossible to crystallise co-precipitates of silica and hydroxide."[8]

After this survey, the manufacturing principle was based on co-precipitation, and this has now been applied successfully to produce various well crystallised clay minerals, among them hectorite, and some new materials which are similar to hectorite, but have significantly enhanced swelling and gelling properties.

In addition, a series of other products has been developed. It has been found that from every type of gelling clay one can produce a variant by treatment with a peptising agent during manufacture. In consequence it disperses very readily in water to form a clear colloidal sol of Newtonian flow properties. In this form it can be more readily incorporated into various systems, such as paints, where it develops thixotropic properties.

The surface affinities of the clays can also be modified by treating them with long chain organic cationic compounds, and in this way one obtains organophilic clays. These swell and form gels in organic liquids such as oils or low polarity solvents.

Finally, one can produce organophilic modifications which disperse in organic liquids to form clear sols. These are the counterparts of the hydrophilic sol-forming modifications and are new : no similar natural clay derivatives exist.

The various types of end-products which may all be based on the same fundamental synthetic clay can now be listed.

(1) A hydrophilic gel-forming variety. Forms thixotropic gels at concentrations of 0.5 per cent upwards. At 5 per cent it is usually too stiff to flow even at high rates of shear.

(2) A hydrophilic sol-forming variety. Can be dispersed in water to form almost clear, stable sols of Newtonian flow properties.

(3) An organophilic gel-forming clay.

(4) An organophilic sol-forming clay.

Properties of Laponite

General

The new materials can be described as free-flowing white powders of a moderately high bulk density, around 1 g/cm^{-3}. They are normally ground to 60-300 mesh size for easy handling. In the various liquid media for which they have been designed, the synthetic clays readily disperse to particles of a size substantially smaller than 1μ, and the initial dry particle size is therefore of little importance.

The chemical compositions vary somewhat for different types of synthetic clay and different forms of end-product. Besides the silicon, oxygen and hydroxyl present in all clays, the synthetics contain magnesium and lithium ions in their structure, and those at present being manufactured contain also structural fluoride ion. Some grades will be made in the future without fluoride. Sodium is present as exchangeable cation. The variety which forms sols in aqueous systems also contains some phosphate, and the organophilic modifications contain organic cations, such as ammonium substituted with long aliphatic carbon chains. The composition shown below refers to the present manufactured hydrophilic gel-forming modification, Laponite B.

Laponite B. Typical chemical analysis

	Per cent
SiO_2	55.9
MgO	26.7
Li_2O	1.9
F	8.3
Na_2O	4.3
Fe_2O_3	0.04
CaO	0.10
SO_3	0.05
CO_2	0.24
Structural H_2O	3.6

The pH of a 2 per cent dispersion is about 9.

The physical structure of a solid, i.e. the atomic arrangement of its smallest structural units, may be studied by X-ray analysis. In the case of clays, this usually means powder diffraction or diffraction by oriented aggregates. These methods can only give a unique solution of the atomic arrangement within

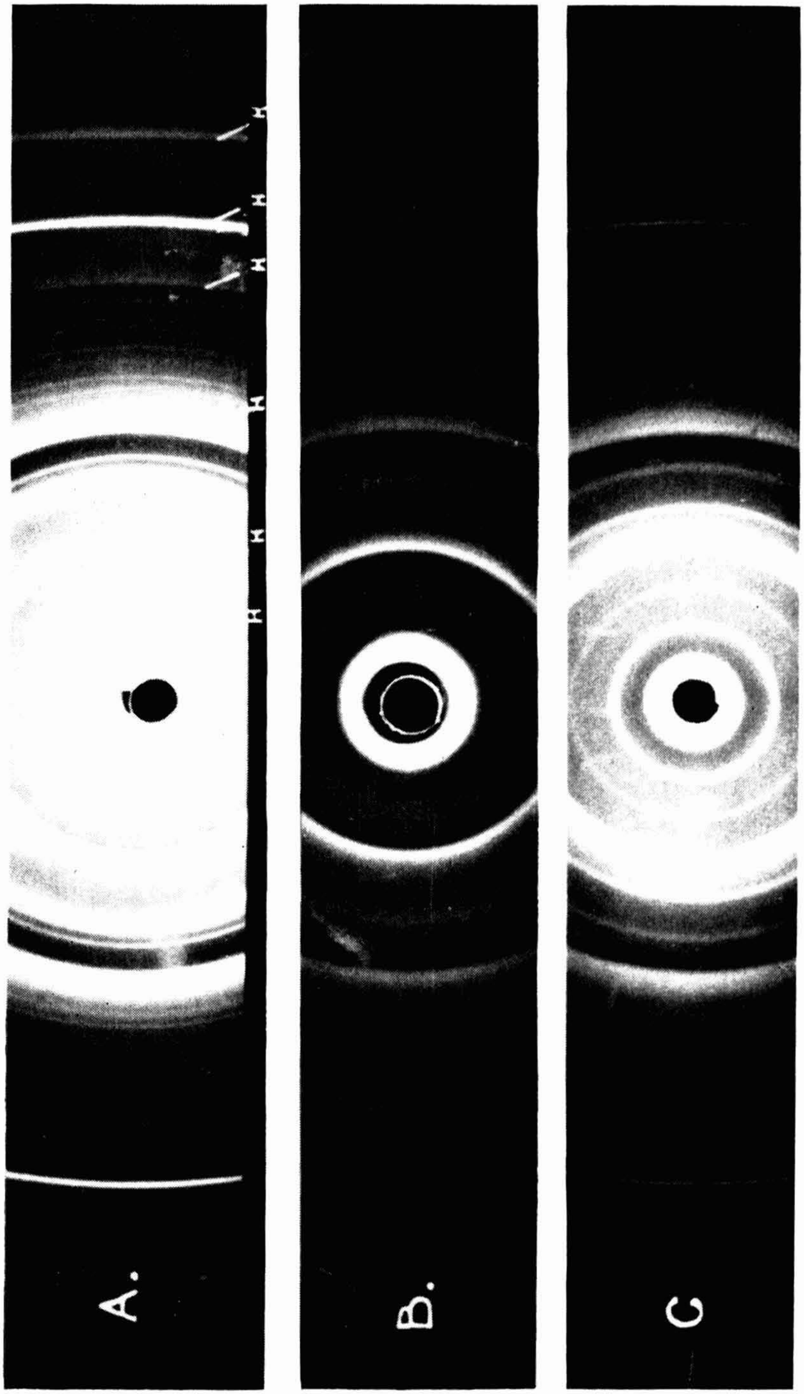


Fig. 1. X-ray powder diffraction pictures (Cu Ka radiation)
A. Commercial purified Hectorite. H lines due to Hectorite, other impurities.
B. Laponite B.
C. Laponite saturated with glycerol, showing rational series of basal reflections.

the unit cell of the crystal structure if highly refined techniques are applied, including intensity measurements. This work has been done for purified hectorite and, by analogy, the results may be applied to Laponite, since the X-ray diffraction patterns of the two materials are practically identical, as shown in Fig. 1. The structural arrangement is based on four close packed sheets of oxygen and hydroxyl or fluoride ions with magnesium and lithium ions in octahedral co-ordination in the centre, between the two internal sheets, and silicon ions in tetrahedral co-ordination between the outer and inner sheets. If all such positions were occupied by Si and Mg ions, the structures would be electrically neutral. Since some magnesium is substituted by lithium and some positions are unoccupied, the structure has, in fact, a negative charge which is balanced by exchangeable cations situated outside the lattice structure, between the multiple layers. The presence of fluoride does not affect the charge. Apart from the exchangeable cations, there is also a variable amount of water between the layers. The latter are thus separated, but retain their parallel orientation up to quite large distances. Only when the clay is " fully dispersed " in water will the layers be completely disoriented and move as free, separate particles. In the case of the gel-forming untreated clay, this state of dispersion can only be temporary, and a new voluminous, card-house like structure, based on attraction between the edges and the faces of the layers, is readily set up. This structure may be repeatedly broken down by shearing, but is re-established on standing ; in other words, it is thixotropic.

In the case of the sol-forming varieties, structure formation in water is inhibited. Surface treatment has neutralised the local positive charges on the edges of the layers and so edge-to-face bonding does not take place until other materials are introduced to counteract the surface treatment.

In the fully dispersed state, the individual particles can be observed by electron microscopy. Fig. 2 shows some well-formed specimens of thin hexagonal plates of nearly 0.5μ diameter, and also some " fuzzy " material consisting of much smaller particles. These small particles are probably responsible for the large surface area (more than 300 m^2g^{-1} when measured by low temperature nitrogen adsorption) of the powder. The surfaces of the apparently thin plate-like particles contribute relatively little to the total area since only the outer surfaces of the layers are accessible to nitrogen, and in the dry state many layers are aggregated in parallel orientation.

Rheological properties

The dispersions can be made up by mixing the dry powders with hot or cold water. Hot water is more rapid in its dispersing action, but the properties are the same at equilibrium, regardless of the method of dispersion used.

Dispersions containing 1-4 per cent Laponite B have been examined by a concentric cylinder viscometer (Fann VG) and in a few cases also by a cone and plate viscometer (Ferranti Shirley with automatic plotter). Shearing stress versus rate of shear curves were determined on the Fann viscometer after standardised shearing treatment designed to eliminate the thixotropic effect. This examination gave results typical of Bingham plastic systems, as shown in Fig. 3. The intercepts on the stress axis of the straight line sections of the

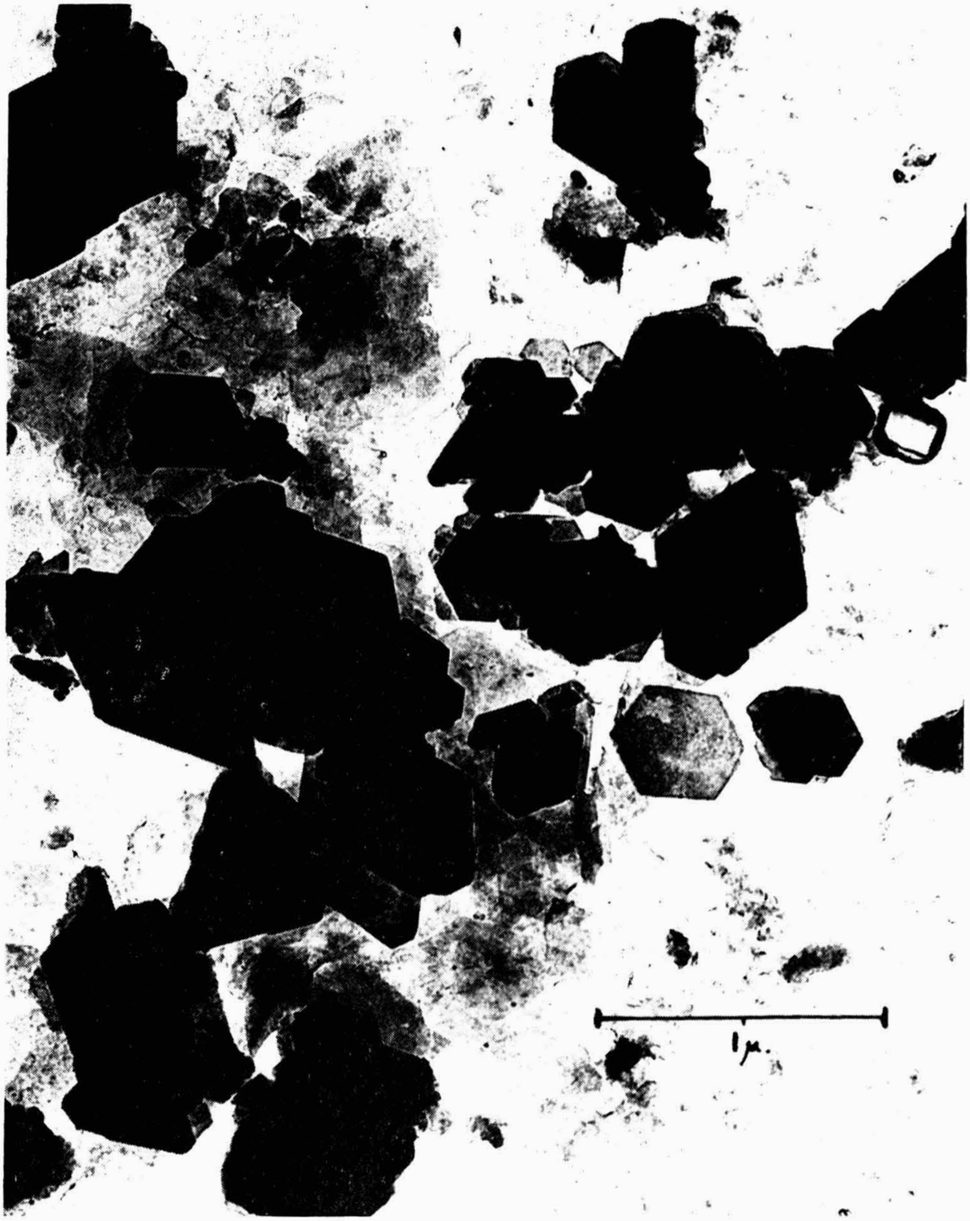


Fig. 2. Electron micrograph of Laponite B

curves, i.e. the Bingham Yield Values, increase rapidly with concentration. The slopes of the straight lines, i.e. the plastic viscosities, are less dependent on concentration. Generally, the plastic viscosities remain low even at high concentrations. It is seen that purified natural hectorite and bentonite have much lower Bingham Yield Values than Laponite for a given concentration.

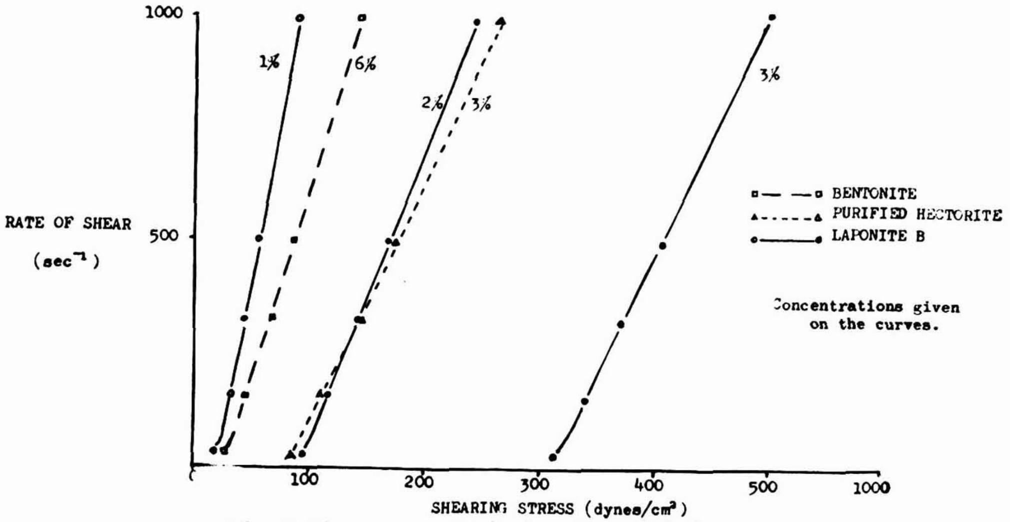


Fig. 3. Flow curves obtained on Fann VG viscometer

To study the thixotropic behaviour, three methods have been used and thanks are due to Professor Carless of the Chelsea School of Science and Technology for permission to quote the results he obtained on the Ferranti Shirley viscometer. The three methods were :

(a) hysteresis loop method,
(b) determination of the decay on shear at constant shear rate.
(c) thixotropic build-up of gel,

The first method is based on measuring the shear stresses, first at gradually increasing, then at decreasing rates of shear. In thixotropic materials, the structure breaks down during the " up curve " of shearing, and, if the time cycle is short, it is not supposed to reform again when the shear rate is decreased, i.e. in the " down curve." So, if a rigorously standardised procedure is applied, the area between the two curves, i.e. the hysteresis loop, is related to the thixotropic property of the material. Fig. 4 shows such a hysteresis loop obtained for a suspension of Laponite B of 2 per cent concentration. Before recording the curve, the gel was sheared from zero to 1759 sec^{-1} and back to zero in 120 seconds. The gel was then allowed to stand in the viscometer for three minutes before starting the measurement. It is seen that a very high stress is recorded on the " up curve " before the structure begins to break down. This is referred to as the static yield value.

The hysteresis loop method is not considered to be suitable for very highly thixotropic materials which may recover too rapidly to give a loop. It is suspected that, in the testing of the Laponite B dispersion, such rapid recovery occurred during decreasing the shear rate and caused diminution of the recorded loop area.

The determination of the decay of stress as a function of time is usually carried out at a constant, fairly low rate of shear, and the results give a useful

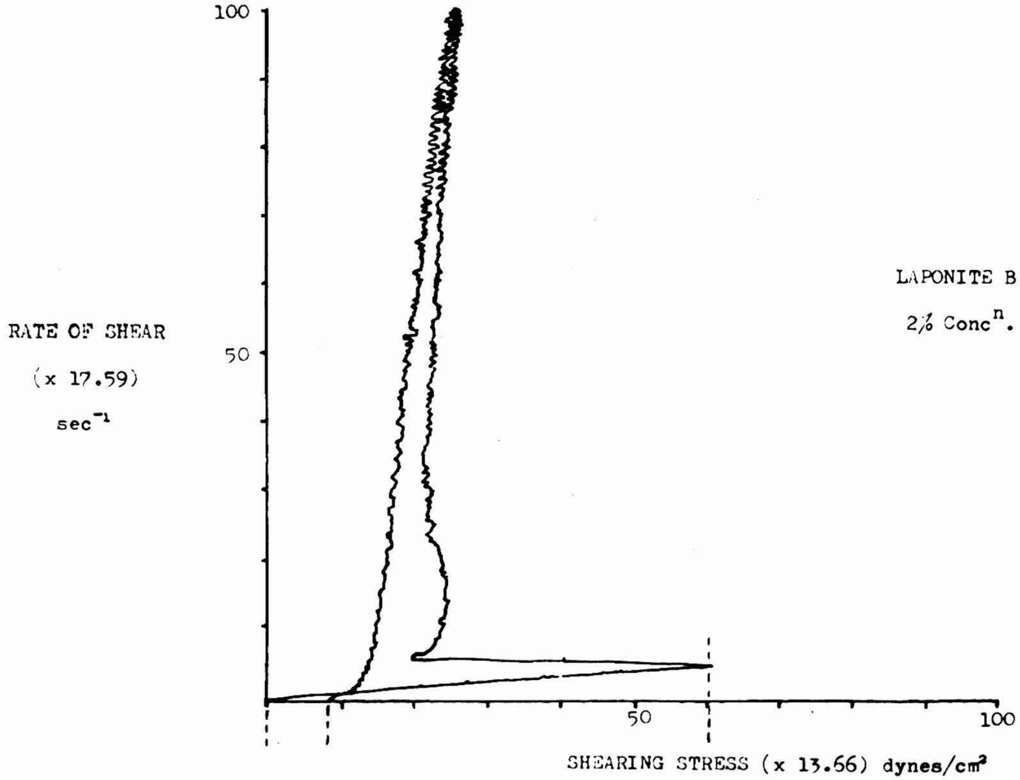


Fig. 4. Automatic recording of hysteresis loop on Ferranti Shirley cone and plate viscometer

indication of the gradual breakdown of the structure, a thixotropic effect. As expected from the previous result, a very rapid breakdown was observed for Laponite B, to such an extent that the response time of the viscometer appeared to be too long for accurate results to be recorded. As a first approximation, it was noted that the decrease of shear stress with time was logarithmic, and, at a shear rate of 17.59 sec^{-1}, the change in ten seconds amounted to a tenfold reduction of shear stress.

The fairly rapid reformation of the gel structure which had been broken down by shearing was assessed in terms of the increase in the static yield value. The gel was sheared by standard procedure and allowed to stand in the viscometer for different periods of time. The static yield value was then measured. Fig. 5 shows that, although the change was very rapid during the first minute, there was some further, slower build-up during the eight minutes of observation.

The influence of electrolytes on the rheological properties of Laponite has been made the subject of a separate study. In general, it has been found that addition of electrolyte at first increases, then decreases both the Bingham Yield Value and the plastic viscosity of the system, and flocculation of the clay occurs only when a fairly high electrolyte concentration—more than

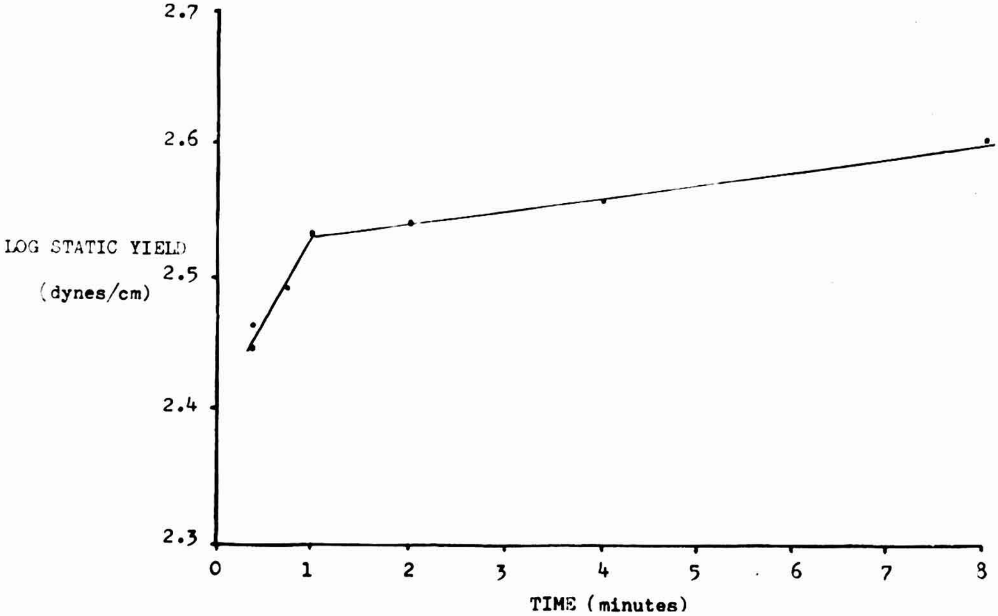


Fig. 5. Increase of static yield value with time: Laponite B 2 per cent concentration

100 milliequivalents per litre of liquid—is present. The presence of peptising chemicals increases the electrolyte tolerance. No flocculation has been observed in pigmented systems on addition of Laponite clays.

Behaviour in paints

The foregoing data regarding the gel forming properties of these synthetic swelling clays suggest that they could be of real value as thixotropic gelling agents for emulsion paints. Being of synthetic origin they are capable of modification during manufacture in order to produce a number of different forms which may be better suited for a variety of applications. The properties of two of these forms will now be discussed in relation to their use as gelling additives in emulsion paints. The one form, Laponite B, may be regarded as the standard base, whilst the other, Laponite S, is a modification in which some of the charges on the clay plates become neutralised when solution in water takes place. As will be seen, this modification, whilst allowing substantially the same gelling properties in the finished paint, give the possibility of easier handling during manufacture.

Preparation of dispersions

The most rapid methods of forming sols or gels in water from both Laponite B and S involve boiling and high shear agitation. However, in works practice, it will be more convenient to use slower speed stirring equipment and avoid the heating operation. The speed of dispersion will be increased by raising the temperature, but even at room temperature Laponite B will disperse in hard water in 15 minutes to give a gel at 2 per cent concentration, whilst Laponite S will disperse in 30 minutes at concentrations of 10-15 per cent to give mobile to viscous liquids which are easily handled. With Laponite S it is recommended that stirring should continue beyond the time for the apparent " solution " of the product, as the sol obtained after a short period of agitation is likely to be fairly viscous and could gel if agitation is withdrawn. Continued agitation prevents the formation of structure in water and maintains the dispersion in a mobile form. With hard water the dispersion after a short agitation period is slightly opaque and agitation should continue until it becomes almost clear.

As mentioned above, the electrolyte concentration, and therefore the type of water used, has some effect on the gel strength and type of dispersion obtained. With Laponite B in water, for example, gelation is almost immediate until a hardness level of less than 70 ppm is reached. Below this a slight delay in gelation occurs. Laponite S is only slightly affected by the water used. In the total paint system there are enough soluble ions present to allow gelation to take place almost independently of the water used.

Whilst in most paint works gels and sols of Laponite will not be stored more than a few days, storage tests have confirmed that no significant change occurs on storage up to one month.

Performance as thickener/gelling agent

Previous work, confirmed by an investigation carried out at the Paint Research Station, Teddington, suggested that in normal gelled emulsion paints Laponite would best be used together with a cellulosic thickener of either the anionic or nonionic type. A comparison of the properties of paints containing mixtures of Laponite B and a hydroxy ethyl cellulose, Natrosol 250 MR, was made working with a total thickener content of 0.5 per cent throughout. The following formula was used in the experiment :

Formula 1	*pts. by wt*
Runa RE30	170.0
Snowcal 6ML	60.0
5% Tetron	12.0
Fomescol U50	0.5
3% Natrosol 250 MR/Laponite B	100.0
Water	100.0
Vinamul N 6815	164.0
Nuodex 321E	1.5

After allowing to stand at 25°C for seven days the following properties were examined :

(a) gel condition.
(b) ease of penetration by brush.
(c) syneresis.
(d) ease of application by brush.
(e) flow properties of film.
(f) non-drip property.

The gel state was classified as firm, weak or none, depending on the deformation of a wedge-shaped sample of the paint cut out of the surface by means of a palette knife, which was observed over three minutes. The non-drip property was assessed by vigorously loading a brush three times and noting the degree of " drip " after the third loading. These observations are recorded in Table 1.

Table 1

Laponite B (replacing Natrosol)	Gel condition	Brush penetration	Syneresis	Brush-ability	Flow	Non-drip
0	none	easy	none	very good	good	fail
14.3	none	fairly easy	none	ditto	good	fail
16.7	weak	ditto	none	good	satisfactory	fail
20	weak	ditto	none	good	ditto	fail
25	firm	ditto	none	good	ditto	pass
33.3	firm	ditto	trace	good	ditto	pass
50	firm	slightly difficult	trace	good	poor	pass
66.7	firm	difficult	slight	good	poor	pass
75	firm	ditto	slight	fairly good	poor	pass
80	firm	very difficult	moderate	slightly sticky	very poor	pass
100	firm	ditto	considerable	sticky	ditto	pass

It was concluded that in the range 25-45 per cent Laponite B and 75-55 per cent Natrosol 250 MR satisfactory properties could be obtained. This suggests as a starting point, that in any formulation the Laponite should be present at approximately 0.2 per cent concentration, slight variations being made to suit particular formulations and requirements.

Point of addition

In view of the differences, previously described, which exist between dispersions of Laponite B and S, an investigation of the best point of incorporation of these grades during the manufacturing process was carried out.

With Laponite B the following formula was used :

Formula 2	*pts. by wt*
Runa RE30	121.5
China clay	36.0
Blanc fixe	60.0
2% Natrosol 250 HR	77.5
4% Calgon S	9.0
Hard tap water	114.4
Fomescol U50	0.7
Butyl carbitol acetate	10.0
Emulsion Revertex Beta AC43	112.5
Nuodex 321E	1.5

Laponite B was added as a replacement of 45 per cent of the Natrosol 250 HR either dry or as a 2 per cent gel.

A similar exercise was carried out with Laponite S, but using the following basic formula :

Formula 3	*pts. by wt*
Runa RE30	148.2
10μ Talc	72.6
2% Natrosol 250 HR	73.2
20 Calgon S	1.8
Ethylene glycol	6.0
Butyl carbitol acetate	12.0
Fomescol U50	0.7
Hard tap water	137.9

Laponite was added either dry or as 10 per cent sol as a 45 per cent replacement of the Natrosol 250 HR. The following table summarises the observations made :

Table 2

Point of addition	Laponite B (2% gel)	Laponite S (10% sol)
(1) Dry to mill base	immediate gel	delayed gel
(2) sol or gel to mill base	ditto	ditto
(3) after emulsion (low shear stirring)	gel undispersed	well dispersed delayed gel
(4) after tinter (low shear stirring)	ditto	well dispersed delayed gel some alteration to shade

It is obvious from these results that Laponite S has considerable advantages in use over Laponite B in the following respects :

(1) the slower development of the gel state avoids the need to use machinery which can handle thixotropic systems.

(2) this delay also allows containers and mills to be more completely emptied.

(3) because a 10 per cent sol of Laponite S is mobile, no difficulties occur in dispersing gel particles even when added under low shear mixing.

(4) if the Laponite S is added immediately before the final tinting, sufficient time, about one hour, should be available to complete the operation and can the product before the full gel structure develops.

Viscosity characteristics

A comparison of the viscosity characteristics of emulsion paints made from Laponite B and S was made using the Fann VG viscometer. The basic paint formula was as follows and Laponite B and S were added at 0.2 per cent on the total :

Formula 4	*pts. by wt*
Runa RE30	148.2
10μ Talc	72.6
2% Natrosol 250 HR	73.2
20% Calgon S	1.8
Ethylene glycol	6.0
Butyl carbitol acetate	12.0

Fomescol U50	0.7
Hard tap water	136.4
Epok V8007	147.6
Nuodex 321E	1.5

In the first place standard flow curves were obtained using the method recommended by the manufacturers of the instrument by reading alternately at high and low shear rates, viz. 600, 6, 300, 3, 100 rpm. The values of shear stress obtained are given in Table 3 and plotted in Fig. 6.

Table 3

Shear rate, sec^{-1}	Shear stress ($dyne\ cm^{-2}$)		
	Blank	Laponite B	Laponite S
5.1	119.3	239	334
10.2	119.3	262	334
170.3	668	739	811
511	1193	1288	1359
1022	1717	1932	2027
Plastic viscosity (poise) ..	5.25	6.44	6.68
10 minutes gel ($dyne\ cm^{-2}$)	215	785	1025

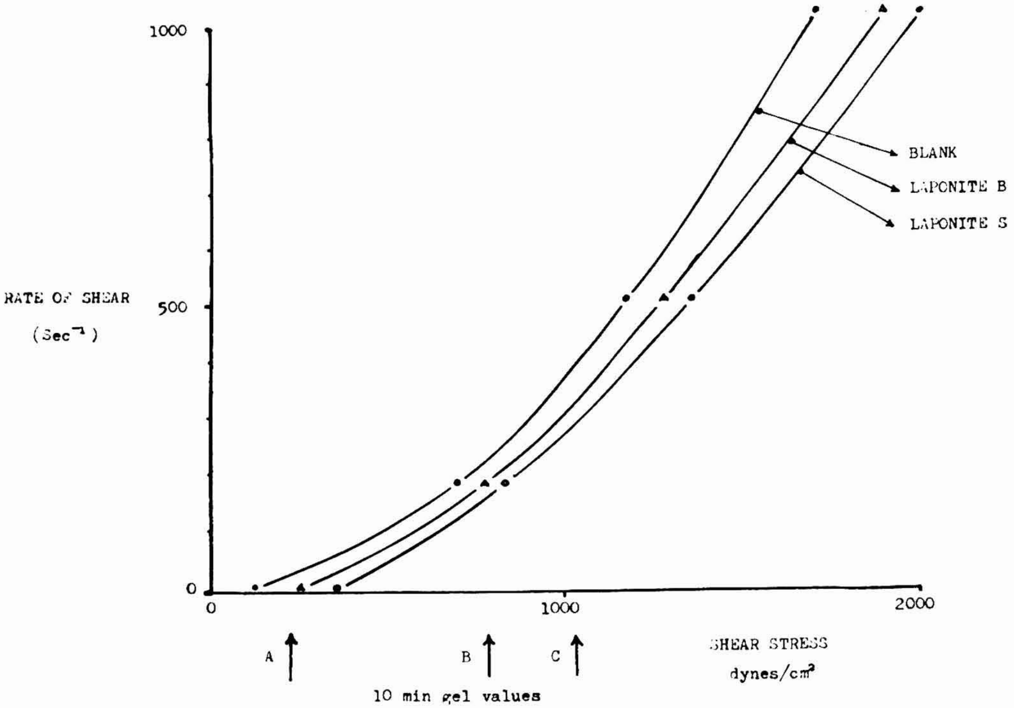


Fig. 6. Standard flow curves

The plastic viscosity was calculated from the readings at 600 and 300 rpm. The " ten-minute gel strength " was measured after stirring at maximum speed and then standing in the instrument cup for ten minutes.

The redevelopment of initial gel strength after shearing was followed by shearing the paints for two minutes at 600 rpm and then leaving undisturbed in the viscometer cup for varying times. See Table 4 and Fig. 7.

Table 4

Standing time	Initial gel strengths in dyne cm^{-2}		
	Blank	Laponite B	Laponite S
15 sec	170	360	405
30 sec	170	405	455
1 min	190	475	550
2 min	215	550	645
5 min	240	665	785
10 min	240	785	960
1 hr	335	1100	1215
3 hr	—	—	1400
17 hr	—	—	—

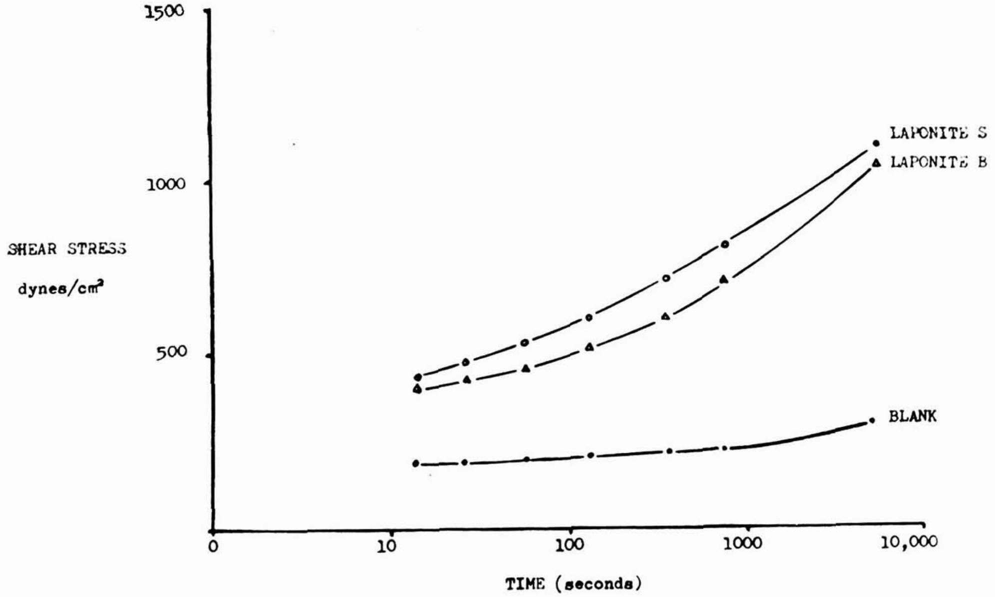


Fig. 7. Redevelopment of gel strength after shearing

As was expected, thixotropic effects are observed in all paints, but they are distinctly more pronounced when Laponite is present.

Time for gelation

As the time for gel formation is of obvious importance in the paint manufacturing operation a study was made of rate of gel formation under static conditions after the Laponite S clay had been added as a 10 per cent solution to an emulsion paint, Formula 4, at a concentration of 0.2 per cent on the total. The gel strength was followed by means of a penetrometer and the results are plotted in Fig. 8. In Fig. 9 the early part of the curve is expanded and the comments of visual observation are recorded in relation to the gel strength. It will be seen that a reasonable delay occurs before the gel develops its full strength. The gel formation is reversible and can be avoided by continued agitation until the paint is transferred to its packages.

Interaction with other ingredients

In introducing a new material into a formulation it is desirable to investigate its interaction with the other ingredients of the system in order to bring to light any special precautions which might have to be taken or any situation which limits the use of the new product. Ideally this should be covered in a statistically designed series in which the variables can be isolated. Failing this, some ingredients can be kept constant and the variation limited to extender, emulsion and thickener. A series of 20 paints were made according to the formulae in Table 5.

Table 5

	1	2	3	4
Runa RE30	148.2	148.2	121.5	121.5
10μ Talc	72.6	72.6	—	—
China clay	—	—	36.0	36.0
Blanc fixe	—	—	60.0	60.0
Natrosol 250 HR 2%	73.2	—	77.5	—
Courlose F700 3%	—	140	—	128.4
Calgon S 20%	1.8	1.8	1.8	1.8
Ethylene glycol	6.0	6.0	—	—
Butyl carbitol acetate	12.0	12.0	10.0	10.0
Fomescol U50	0.7	0.7	0.7	0.7
Water (hard)	136.4	72.6	128.6	80.2
Epok V8007	147.6	147.6	—	—
Revertex Beta AC43	—	—	112.5	112.5
Nuodex 321E	1.5	1.5	1.5	1.5

5-8, 1-4	+	1.1	Laponite	B powder	
9A,-1	+	7.5	10% sol	Laponite S	with appropriate reductions in water content
9B,-1	+	11.0	”	”	
9C,-1	+	17.5	”	”	
10A,-2	+	7.5	”	”	
10B,-2	+	11.0	”	”	
10C,-2	+	17.5	”	”	
11A,-3	+	7.5	”	”	
11B,-3	+	11.0	”	”	
11C,-3	+	17.5	”	”	
12A-4	+	7.5	”	”	
12B,-4	+	11.0	”	”	
12C,-4	+	17.5	”	”	

These paints were examined for non-drip, brushing and flow properties after standing for one week at 25°C and for storage properties after eight weeks at 40°C. The results are in Table 6.

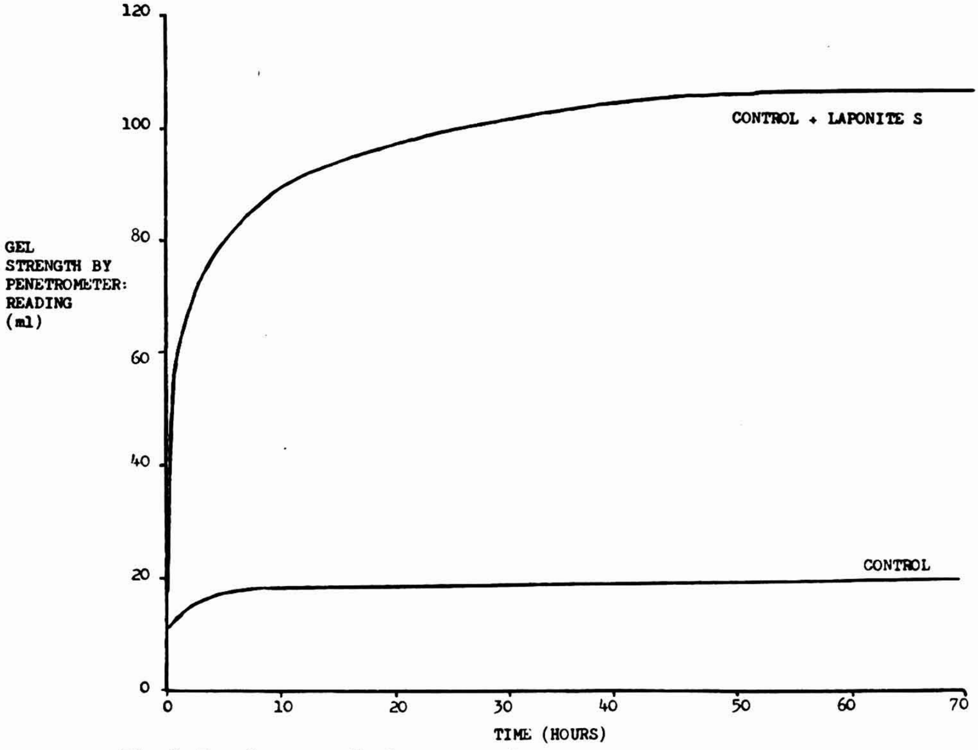


Fig. 8. Development of gel strength after Laponite addition to a paint

These results indicate :

(a) that Laponite gives similar thixotropic properties with both sodium carboxy methyl cellulose and hydroxy ethyl cellulose.

(b) that less Laponite is needed with china clay present because of the contribution made to structure by this extender.

(c) that in all these systems satisfactory gelled paints can be made by adjustment of Laponite S concentration in a paint around a mean value of 0.2 per cent.

(d) differences in stability arising from the emulsion might be countered by adjustments in sequestering agent, but, in any case, Laponite S enhances stability.

Table 6

Paint No.	Non-drip	Brushability	Flow	Storage 8 weeks at 40°C	
				Syneresis (mls in ½ pint)	Rotothinner viscosity change poise
1	fail	excellent	excellent	8	+ 7.5
2	fail	excellent	excellent	0	− 1.1
3	fail	excellent	very good	2	+13.2
4	fail	excellent	excellent	trace	− 1.0
5	pass	good	very good	3	+ 3.7
6	pass	fairly good	very good	0	+ 0.8
7	pass	ditto	fairly good	3	+12.3
8	pass	very good	good	0	+ 1.5
9A	fail	excellent	excellent	4	+ 6.8
9B	pass	excellent	excellent	3	+ 3.0
9C	pass	good	very good	1	+ 3.5
10A	fail	excellent	excellent	trace	− 0.4
10B	pass	fairly good	very good	trace	0
10C	pass	ditto	good	none	+ 0.2
11A	pass	very good	good	2	+12.0
11B	pass	good	good	2	+10.2
11C	pass	fairly good	fairly good	2	+ 9.7
12A	pass	very good	excellent	none	− 1.0
12B	pass	ditto	good	none	− 2.0
12C	pass	good	fairly good	none	− 1.2

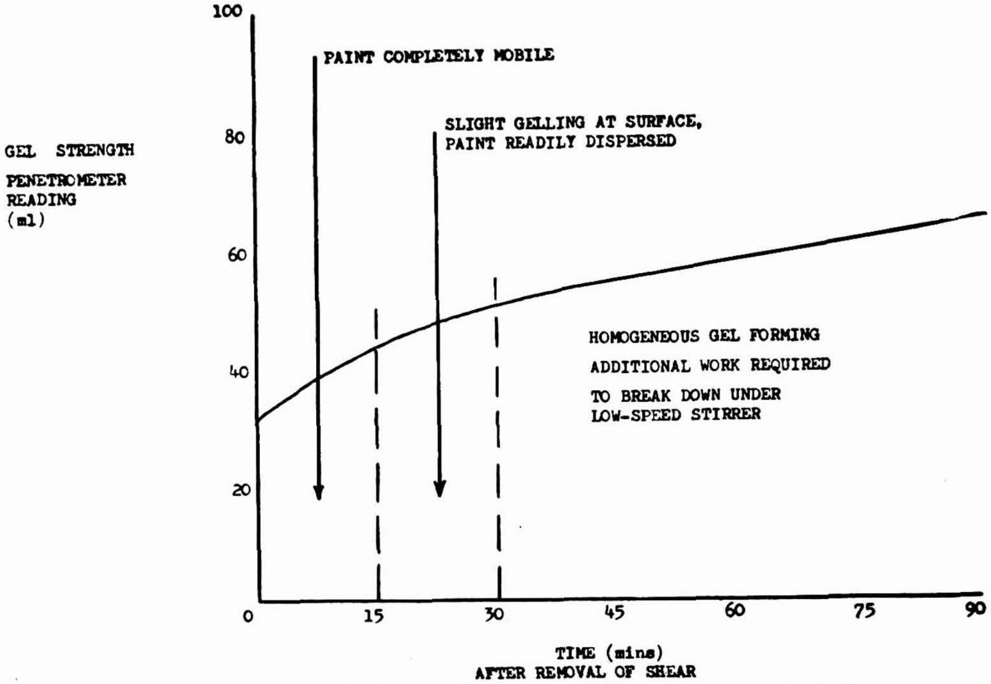


Fig. 9. Development of gel strength. Paint mobility after removal of shear

Effect of emulsion

It is believed that some gelling agents currently in use fail to give gels in certain circumstances, e.g. with surfactant or polyvinyl alcohol stabilised emulsions or when the pH is outside a limited range. Paints were prepared from emulsions listed in Table 6 using hydroxyl ethyl cellulose as thickener throughout and adding 0.2 per cent Laponite S. The basic formula is given below :

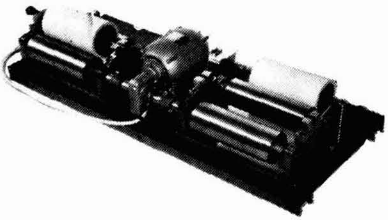
Formula 5	*pts. by wt*
Runa RE30	148.2
10μ Talc	72.6
2% Natrosol 250 HR	73.2
20% Calgon S	1.8
Ethylene glycol	6.0
Butyl carbitol acetate	12.0
Fomescol U50	0.7
Hard tap water	114.4
Emulsion	147.6 or equivalent
Nuodex 321E	1.5
10% Laponite S	11.0

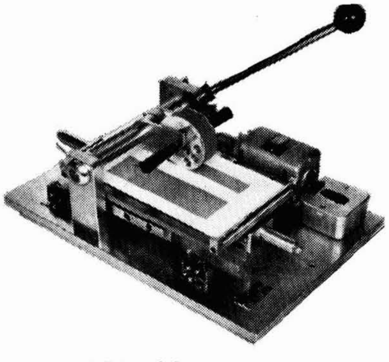
A constant PVC and a constant solids content were maintained, but no adjustments of water content were made to take care of differences in viscosity. The gel condition of the paints is recorded in Table 7.

Table 7

Grade No.	Polymer type	Emulsion stabiliser	Gel condition
Vinamul N 6265	vinyl acrylic copolymer	hydroxy ethyl cellulose	satisfactory
Vinamul N 6515	vinyl acetate/ vinyl caprate	ditto	slightly weak (1)
Vinamul N 6525	ditto	ditto	satisfactory
Vinamul N 6815	vinyl acetate/2 ethyl hexyl acrylate	ditto	ditto
Vinamul N 6825	ditto	ditto	ditto
Vinamul N 6930	vinyl acetate/ VeoVa 911	probably surfactant	very weak (2)
Vinacryl 4000	acrylic copolymer	ditto	very weak (3)
Vinamul 9700	polyvinyl acetate	hydroxy ethyl or ethyl hydroxy ethyl cellulose	satisfactory
Vinamul 8800	ditto	polyvinyl alcohol	ditto
Vinamul 9900	ditto	ditto	ditto

Except in two cases satisfactory gels were obtained. See notes 1, 2 and 3.






PURE BRISTLES

(1) It was found that the level of Laponite S required to give satisfactory gel was 0.3 per cent.

(2) The paint made to this formula was unstable and increased in viscosity rapidly. Another emulsion based on a Versatic acid polymer, Polimul 1175, presumably cellulose stabilised, gave a satisfactory gel. In order to determine the correct conditions for gelation with Vinamul N 6930 two paints were made using Vinapol 1640 as dispersant and Modocoll E600 and a 3 : 1 mixture of Modocoll E600 and Cellofas B3500 as thickeners. With Modocoll alone no gel was obtained, but with the mixed celluloses a satisfactory product resulted.

(3) This emulsion is normally used at higher PVCs than in the formula employed which was 50 per cent. Working at levels of 65 per cent to 80 per cent PVC with Snowcal 6ML as extender and hydroxy ethyl cellulose as thickener, satisfactory gels were obtained. If it is required to work at lower PVC levels, it becomes necessary to increase the cellulose thickener content in order to obtain a gel.

It was concluded from these results that, in most cases, satisfactory gels would develop by the addition of Laponite S to an emulsion paint formula. However, in a limited number of cases, and, in particular, with surfactant stabilised emulsions, modifications in the formulation will be necessary to suit the peculiar conditions obtaining.

Dispersants

A range of sequestering and dispersing agents in common use were examined as additives to a basic formula using Epok V8007 and hydroxy ethyl cellulose. The agents used and the paint conditions are listed below.

Table 8

	No Laponite	0.2% Laponite
Vinapol 1640	no gel	satisfactory gel
Orotan 731	flocculated	satisfactory gel deflocculated
Dispex 115	no gel	satisfactory gel
Calgon S/Orotan 731 ..	no gel	ditto
Calgon S	no gel	ditto
Tetron	no gel	ditto
Lissapol NX	flocculated	satisfactory gel deflocculated

It will be seen that in every case a satisfactory gel was obtained and in two cases an inprovement in the state of dispersion was made. The strength of the gel varied slightly from one agent to another.

Thickener or protective colloid

In the course of the work on Laponite S the range of thickeners examined has included the following :

Hydroxyl ethyl cellulose	Natrosol 250 MR Natrosol 250 HR
Ethyl hydroxy ethyl cellulose	Modocoll E 600

Sodium carboxy methyl cellulose	Courlose F 700 Cellofas B 3500
Methyl cellulose	Celacol M
Hydroxy propyl cellulose	Celacol HPM 1500 DS Celacol HPM 5000 DS

In all cases satisfactory gels have been obtained, the strength of which has been dependent on the thickener and the emulsion used in the formula.

Extenders

The extenders used in the work have included :

Calcium carbonate	Snowcal 6 ML Queensgate Whiting 25μ
China clay	Supreme ECC
Blanc fixe	Barium Chemicals Ltd.
Talc	Microtalc AT 10μ

In no case is there evidence that any extender interferes with the gelling properties of Laponite S, although the level of structure obtained is influenced to the same extent as in a paint containing no Laponite S.

Effect of pH

Paints were prepared according to Formula 5 using emulsion Vinamul N 9700 and employing talc as the extender. Alterations to pH of the paint were made by additions of acetic acid or ammonia and the effect on gel strength noted.

From pH 6.5 to 9.9 the gel strength remained firm. At pH 5.9 a considerable reduction of gel strength occurred which was probably due to flocculation of the Laponite. It is concluded, therefore, that in the range of pH obtained in most emulsion paints, Laponite will give satisfactory gels.

Laponite in textured emulsion coatings

With the present grades of Laponite the optimum results in normal emulsion paints are obtained in mixture with cellulosic thickeners. However, in some types of paint based on synthetic emulsion which are stippled or trowelled after application to develop a pattern or texture on the surface, the fairly rapid gel recovery of Laponite can be used to advantage. In this case Laponite would be used in the absence of added thickener or by additions of up to 0.75 per cent of the total formula. As an example the Formula 1 was modified by the addition of Laponite S at 0.75 per cent.

During mixing of the mill base the viscosity was low and remained so even after the addition of the emulsion. On standing in the can the paint developed a high level of thixotropy and was completely dripless. Application properties were easy and the film set up rapidly, ensuring a heavy coating which did not flow. The film could be stippled to give a fairly sharp textured surface.

Storage properties of the paint were satisfactory, giving no syneresis and no change in viscosity after four weeks.

Conclusion

Synthetic swelling clays, Laponite B and Laponite S, of which the latter is preferred, are useful products for the development of gel structure in a wide range of emulsion paint formulations.

Acknowledgments

Acknowledgments are made to colleagues for most of the practical work and to the Directors of Laporte Industries Limited for permission to publish this paper.

[*Received 12 October 1967*

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J. Oil Col. Chem. Assoc. 1968, 51, 254-255

Short Communication

Rapid esterification of fatty acids

By E. Sunderland

Aktiebolaget, Alfort & Cronholm, Stockholm, Sweden

A great deal of attention has been devoted to the esterification of fatty acids, particularly since the advent of GLC. Many methods and reagents have been recommended, including methanol alone in micro-autoclaves, methanol with catalysts such as *p*-toluenesulphonic acid and BF_3 complexes, dimethyl sulphate, diazomethane. Curiously enough one of the most effective agents, anhydrous methanolic HCl, seems to have been entirely neglected.

The author first used this reagent some 30 years ago—by accident! Whilst recrystallising stearic acid to take a melting point, methanolic HCl was used for the final crystallisation instead of petroleum ether. A single crystallisation gave pure methyl stearate.

In connection with the preparation of methyl fatty acid esters for GLC the use of this reagent has been investigated in more detail. The following table shows the degree of esterification of a technical linoleic acid after various times at room temperature (23°C) with 1.2 per cent methanolic HCl. Determinations were by direct titration against blanks, 0.5 g fatty acid + 5 cc methanolic HCl.

Time (minutes)	*% Esterification*
2	43.4
5	71.7
10	89.4
15	95.2
20	97.9
30	98.6
45	99.0

A single experiment with 0.6 per cent methanolic HCl gave 98.0 per cent after 45 minutes.

The reaction may be speeded up by gentle warming, and at 50°C some 5 minutes appears to suffice. Small losses of HCl make the determination less accurate with the simple technique used, but 5 minutes at 50°C would certainly appear to be adequate judging by the room temperature results.

Similar experiments with a low rosin tall oil fatty acid gave a maximum esterification figure of 98.3 per cent (after 160 minutes) and it seemed likely that the resin acids were not being esterified. A synthetic mixture of technical linoleic acid and WW rosin was esterified under the same conditions (1.2 per cent HCl/MeOH) for 60 minutes. Rosin content of mixture 15.7 per cent ; found :

15.8 per cent (assuming 100 per cent fatty acid esterification). It therefore appears that the reagent may also be useful for the determination of rosin in the presence of fatty acids.

An attractive feature of the method is the ease with which the reagent may be prepared. Unfortunately, after the lapse of many years one cannot give due credit to the originator of the elegant method for producing dry HCl, other than by describing it. It deserves to be more widely known.

A distilling flask is half filled with concentrated sulphuric acid and fitted with a cork through which a tap funnel is inserted. To the end of the funnel tube a piece of glass capillary tube is attached (by fusion or a short rubber tube) of such a length that it reaches the bottom of the flask. The capillary bore may be between 0.2 and 1.0 mm. Concentrated hydrochloric acid is added to the tap funnel and runs slowly down into the sulphuric acid. HCl is generated instantaneously and dried by the sulphuric acid to issue from the side arm. If high rates of HCl production are used it is advisable to use a spray trap, most simply made by putting a glass wool plug into the flask neck below the side arm, or into the side arm itself.

[*Received 18 December 1967*

Next month's issue

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

" **Some features of ship painting,**" *by P. J. Gay.*

" **Discoloration of paint films by traces of mercury, lead and iron compounds,**" *by E. Hoffmann.*

" **Mechanical properties of alkyd resin varnish films and the effect of different weathering conditions on them,**" *by R. Vittal Rao, Mohd. Yaseen and J. S. Aggarwal.*

" **Dehydropolymerisation and copolymerisation of drying oils and other reactive compounds exhibiting α-methylene activity,**" *by R. Lambourne.*

Reviews

INDUSTRIAL FILTRATION OF LIQUIDS

By D. B. PURCHAS. London : Leonard Hill, 1967. Pp. xxiv + 463. Price 84s.

This book is directed at users or potential users of filtration equipment rather than designers, and emphasis is placed on the industrial or practical aspects of filtration. Some theoretical relationships are introduced in the final chapter but these are selected and presented so as to show their practical application to industrial problems.

The scope of the book is wider than might be suggested by the title and is based on a definition of filtration as the separation of a discontinuous from a continuous phase by any means. Gravity and centrifugal settlers and thickeners are, therefore, included in addition to permeable membrane filters.

The first three chapters are general. Chapter 1 discusses the nature of filtration problems and introduces the approach to their solution which is adopted in the book. Chapter 2, under the title " Pre-treatment of Solid/Liquid Mixtures," describes various methods by which filtration problems can sometimes be made easier. Most of these treatments aim at increasing the settling rate or the solids concentration of a system. A large chapter on filter media arranges the numerous different types into ten groups, on the basis of their rigidity. These groups are described separately. It is fortunate that the selection of a suitable medium is often dictated by the type of filter selected.

The next four chapters fulfil the main objective of the book, which is to help the user to select the right filter for the job. Equipment is classified according to the general characteristics of the slurry, namely,

1. rapid settling systems
2. systems with medium or low settling rates
3. clarifying systems
4. pastes, pulps and non-fluid systems.

Further sub-division is as follows : with or without washing of the solids ; batch or continuous discharge of the solids ; separating forces, viz. gravity, vacuum, pressure or centrifugal. The description of available equipment is comprehensive and, like the rest of the book, is liberally illustrated by photographs, diagrams and graphs. References are conveniently given at the foot of the page on which they are quoted.

There has been a dearth of books on filtration and with this admirable and painstaking book, Mr. Purchas has provided a useful complement to the recent Ministry of Technology review and bibliography, Solid-Liquid Separation (1966).

D. J. MORRIS.

LABORATORIUMSBUCH FUR DIE LACK UND ANSTRICHMITTEL INDUSTRIE. ZEIDLER-BLEISCH

3rd Edition, completely revised by Gertrud Bleisch. Pp. 426. Dusseldorf: Wilhelm Knapp Verlag, 1967. Price DM 58.

The accepted German simple tests for paints and varnishes, and their raw materials, which do not require costly equipment, are collected in this well produced laboratory manual. As it was originated by Hans Wolf in 1924 and later repeatedly brought up to date by G. Zeidler, this book has been widely used on the Continent. It does not claim completeness, or the inclusion of all the modern tools of analysis and research such as gas spectroscopy, but it is very comprehensive as its 426 pages indicate.

The main headings are :

General analytical methods.

Specific determinations for oils, solvents, pigments and resins.

Chemical analysis of paints.

Technical—practical methods and instrumentation for testing paint and paint film properties.

Practical suggestions.

Explanations of principles involved are given, and procedures are described clearly. Often a number of methods have been included, and attention is drawn to short cuts.

Most of the more recent synthetic resins, etc., are covered.

The majority of testing methods tallies with those known in this country, but laboratories seeking additional information or which have Continental interests may like to have at hand this work which has been thoroughly brought up to date by Miss Bleisch.

MANFRED HESS.

Information Received

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

A new range of *Lodematic* " *Up-Enders* " for barrels and drums has been introduced by **Lodematic Limited.**

Up-ending of barrels, received horizontally " on the roll " on the *Lodematic* forks, is claimed to be achieved in seconds, actuated automatically by barrel weight or controlled by a single manual valve lever. The *Up-Ender* can also be used for work positioning and turning of reels. Off-loading is at heights between ground level and 20 feet, according to model.

Hydraulics are powered by mains electric motor, but battery or diesel powered mobile, manually-propelled models are also available. The *Lodematic Power-Pack* can be supplied integral with the *Up-Ender*, or independent, linked by a hydraulic hose.

As from 1 January 1968 **National Adhesives Limited** is to be named **National Adhesives and Resins Ltd.** The new name reflects the increased importance of synthetic resins in the company's business.

British Resin Products Limited have recently published their technical booklet *E11/2*, giving detailed information on their range of *Epok* amino resins. A section of the booklet also covers *Epok* alkyds, which are used with the aminos.

An improved low-price periodical black for large letterpress half-tone work on flatbed machines has recently been announced by the **Fishburn Printing Ink Company Ltd.**

Designated *AP 3244*, the new ink has been formulated to give a high density blue tone, quick setting, sharp half-tone capabilities and a good level of finish on a wide variety of stock. The ink is claimed to be particularly valuable when printing a mixture of new and used electros, stereos and zinc blocks.

G. Bopp & Co. Ltd., London, claim to produce the world's finest wire filter cloth. Woven in stainless steel wire, the cloth has almost 3,000,000 pores to the square inch. These materials are designed for ultra-fine filtration in hydraulically controlled systems, air and gas filtration, and use in fuel filters for critical applications.

A new grade of *Elvacite* acrylic resin is now available from **Du Pont Company (United Kingdom) Limited.** The new grade, *Elvacite 2043*, is said to be particularly suited as a resin base for gravure and flexographic inks for packaging materials, as it has properties of high alcohol solubility, resistance to discolouration on heating, and adhesion to most plastic films and aluminium foils.

Also from **Du Pont** is a new class of interpolymer resins for hot-melt adhesives, coatings and lacquers. The new range of *Elvax* acid terpolymer resins incorporate an organic acid into the ethylene and vinyl acetate (EVA) structure of the established *Elvax* vinyl resin range. Superior oil and grease resistance, improved " hot tack " properties, greater adhesion to non-porous substrates and improved seal strength are claimed.

Additions to the *Irgalite PDS* range of easily dispersible organic pigments for paint have recently been announced by **Geigy (UK) Limited.** These are *Irgalite Red PDS4*, a blue shade toluidine red, claimed to have excellent fastness to light and good fastness to chemicals, and *Irgalite Blue PDS5*, a beta-form copper phthalocyanine blue, claimed to have outstanding lightfastness, and excellent fastness to chemicals, solvents and overpainting. Both pigments embody the rapid dispersion characteristics of the *Irgalite PDS* range, and are available in granular form for easy dust-free handling.

J. M. Steel & Co. Ltd. have recently introduced a new grade of their thixotropic alkyd *Alkydal F 67 TIX*. The new grade, *Alkydal F 67 TIX 40 per cent*, has lower solids to ease formulating and processing. It is claimed that any desired gel structure can now be obtained in a normal decorative or urethane paint, without loss in properties, using the new grade and the existing grade, *Alkydal F 67 TIX 60 per cent*.

L. A. Mitchell Limited have recently produced a new eight page leaflet describing their weight control system for bulk material handling. The leaflet, ref. CP10, contains comprehensive information on the range and design of the equipment.

A new etch primer, which, it is claimed, answers the need for a one-pack material with the high performance of a two-pack etch primer, has been developed by **Hadfields (Merton) Limited.** The primer can be used on a wide range of ferrous and non-ferrous metals, and is made from polyvinyl butyral resin with anticorrosive pigment. High resistance is claimed to alkalis, cutting oils and lubricating oils.

Imperial Chemical Industries Ltd., Dyestuffs Division, has applied for planning permission to build and equip a £3-million technical service centre at Blackley, Manchester, adjoining the headquarters of Dyestuffs Division, Hexagon House.

The new centre will replace several old laboratories, and will provide the most up-to-date facilities. All the present staff of the Division's Application Research and Technical Service Department, more than 700 people, will be accommodated, and it is claimed that it will be one of the largest of its kind in the UK. Every opportunity is to be taken to utilise **ICI** materials in the building of the new block.

The Mastermix Engineering Co. Ltd. has recently introduced a new machine, the *Paste Mixer/Disperser*. The design combines a slow speed agitator with a high speed cavitation disperser, which, it is claimed, eliminates the difficulties involved in the production of large quantities of dispersion, solutions or mixes, particularly with materials having poor flow characteristics, and produces absolute uniformity of product in the minimum of processing time.

A new range of *Mixer Granulator Dryers* is announced by **Calmic Engineering Co. Ltd.** The standard range is of 5 and 10 cu ft capacity, but a 20 cu ft machine is in use.

The mixer mixes, granulates and dries automatically, without the need to transfer material from one machine to another, and is in line with the requirements of the Factory Inspectorate.

A new vinyl coating for collapsible tubes has been introduced by **Pinchin Johnson & Associates Limited.** The formulation is said to give optimum balance of resistance to cold crushing as well as to solutions of the product packed, and high degrees of adhesion and flexibility are claimed.

A new miniature proofing press, the *Minipress*, manufactured by **Alfr. Andersson & O. Sorensen,** is now available in this country from **H. E. Messmer Ltd.,** the sole distributors.

The *Minipress* is said to fulfil a demand for a small efficient and economically priced apparatus for evaluation of printing inks and papers prior to the printing process.

Hull

Dehydropolymerisation and copolymerisation of drying oils

The third ordinary meeting of the session was held at the College of Technology, Hull, on 4 December 1967. The Chairman, Mr. L. W. Wynn, introduced Mr. R. Lambourne, who gave a paper on the " Dehydropolymerisation and copolymerisation of drying oils." The meeting was the annual joint lecture in conjunction with the Oil and Fats Group of SCI.

The work described by Mr. Lambourne stemmed from the investigation by Karash and by Farmer and Moore of the reactions of aromatic compounds with peroxides. This work had later been extended to fatty acid esters by Sutton and Klingman. It was shown that a hydrogen atom was abstracted from an α-methylene group in an unsaturated fatty ester, giving rise to a number of resonance forms, one of which could contain conjugated double bonds, and which would give rise to a number of dimers.

The work carried out by Mr. Lambourne had explored the possibility of using this reaction to upgrade the drying properties of semi-drying oils. In these reactions stoichiometric amounts of peroxide were employed and the reaction bore no relation to the usual peroxide initiated polymerisation of unsaturated monomers.

The oil to be treated was heated with peroxide in sealed tubes in the temperature range 145-200°C, and after removal of tertiary butanol, formed from the tertiary butyl peroxide, oils of about 20 poise viscosity and iodine value 120 could be produced, since there was no loss of unsaturation during dehydropolymerisation.

The reaction with peroxide might be carried out with myrcene or *allo*ocimene in the presence of fatty acid esters, leading to what could be described as a " myrcenated oil." With linseed oil and myrcene, reaction with peroxide led to a range of products of viscosity 10-12 poise, which, in the presence of Co and Pb driers, yielded very rapid drying, hard glossy films.

The lecture was followed by a discussion in which Messrs. Wynn, Lythgoe, Finn and Dr. Warson took part. A vote of thanks was proposed by Mr. Lythgoe and was warmly supported.

Two members of SCI and 19 members of OCCA were present.

S.R.F.

Irish

The measurement of colour by instrumentation

The second meeting of the session was held in the Imperial Hotel, Cork, on Friday 27 October 1967. This was the first occasion that a section meeting has been held outside Dublin.

Mr. R. Adam, the Chairman, presided and 22 members and 13 guests were present.

We were honoured to have as one of our speakers our Immediate Past President, Dr. S. H. Bell, who introduced the main speaker, Mr. P. V. Foote of the Paint Research Station, who presented a first class paper entitled " The measurement of colour by instrumentation." Mr. Foote explained in great detail the theory of colour measurement and the equipment which is used.

The paper aroused considerable discussion and those taking part included Messrs. O'Callaghan, Quick, Jones, Patton, Caughlan, Adam, Morrish and Kieran.

A vote of thanks was proposed by Mr. Walsh and seconded by Mr. N. Morrish.

Prior to this 15 members visited Mitchelstown Creameries and spent an enjoyable afternoon learning and seeing the art of cheese making.

F.D.H.S.

Paint testing for a purpose

The third technical meeting of the Irish Section in the present session was held at the Clarence Hotel, Dublin, on Friday 24 November 1967 at 8 p.m. The Chairman, Mr. R. Adam, presided and 27 members and three visitors were present.

The chairman introduced one of our own members, Mr. R. B. Simon of the Institute of Industrial Research and Standards, who presented a stimulating paper entitled " Paint testing for a purpose." He described the two approaches to the testing of paint. Namely " *ad hoc* testing " and " testing to a standard."

He explained the implications of each in detail and this brought forth a lively discussion. Those taking part were Messrs. O'Callaghan, Murdoch, Smith, Farmer, Jones, R. Murphy and A. P. Murphy.

A vote of thanks was proposed by Mr. M. O'Callaghan.

F.D.H.S.

London

Room temperature curing acrylic systems

A meeting was held on 14 December 1967 at East Ham Technical College, London, E.6, with Mr. R. N. Wheeler in the chair, to hear a lecture on " Room temperature curing acrylic systems," by Mr. A. G. North, of Cray Valley Products Ltd.

Mr. North discussed the theoretical choice of polymer structure which could be used in solvent based acrylic systems which would cure at ambient temperatures. With acrylics, one had the ability to vary independently both the backbone and the pendant groups. There were three alternative basic linkages for the polymer backbone, C-C, C-N, or C-O. Ideally one would choose a C-C linkage, which was highly resistant to attack, as in polyethylene and various substituted polyethylenes, e.g. polypropylene or polymethylpentene. C-N linkages were difficult to obtain without oxygen linkages, e.g. as in polyamides. C-O linkages, as in ester or ether groups, had comparatively poor resistance. Combinations of C-O and C-N linkages occurred e.g. in polyurethanes.

In addition there was a need to obtain certain essential properties such as solubility, compatibility, cross-linking potential, and sufficient polarity to obtain adequate adhesion. Solubility and compatibility were generally attained by the introduction of oxygen and nitrogen containing groups. Again for polarity one would incorporate OH, COOH, or NH_2 groups, which could be incorporated in the side chains.

For the backbone, there was not a great deal of choice,when economic considerations were involved, and one would use esters unsaturated either in the acid or the alcohol components, e.g. vinyl acetate, or methyl acrylate. The backbone of the polymer was resistant to attack ; it was not so important if the side chains showed some tendency to hydrolysis, but the relative ease of hydrolysis needed to be studied, and one would not put in more hydrolysable material than was necessary. Styrene could be used to fill up spaces on the backbone (good solubility in aromatics, no hydrolysis, and low cost). This tended to show poor impact resistance, however, and one would not use more than was necessary, other groups, such as ethylene or propylene being used where possible (this was already being done in emulsions).

It was then necessary to introduce reactive groups for crosslinking. There were, for instance, the " stoichiometric " reactions, e.g. epoxy/amine, and isocyanate. Or those which could be regarded as " catalytic," e.g. the curing of methylol groups by acids, or epoxy groups by tertiary amines.

A practical room-temperature curing system which had been developed was then described. This involved a hydroxyl containing acrylic resin cross-linked with an aromatic isocyanate. The backbone was highly resistant, and only the side chains were liable to hydrolytic scission. Admittedly, if one had used a polyester or polyether backbone, any weakness could be partly overcome by the introduction of a greater number of cross links, but this would mean the introduction of groups to confer flexibility, which was expensive. Mr. North then showed the results of practical evaluation of the new system, in comparison with other conventional systems. Good resistance to chemicals and staining was obtained, and impact resistance and adhesion were high.

The lecture was followed by an extensive discussion, in which contributors included Mr. S. E. Francis, Mr. C. E. Hoey, Mr. D. F. Archer, Mr. D. Stewart, Mr. H. Adler, Mr. K. W. Green, Mr. L. D. Eccleston, Dr. H. Warson, Mr. F. A. Clarke, Mr. T. A. Chessman, Mr. E. R. Buck, and Mr. A. Panayiotides. A vote of thanks was proposed by one of the students at East Ham Technical College, Mr. E. Buck, who expressed the pleasure of those present at having such an interesting and well prepared lecture presented in East London.

V.T.C.

Manchester

Development in the paint industry

The January meeting was held at the Manchester Literary and Philosophical Society's premises on Friday the 12th, Mr. I. S. Moll being in the chair and 83 members and guests being present.

Mr. J. H. Butler, Chairman and Managing Director, Berger-J. & N. Paints Limited, delivered a lecture, " Development in the paint industry." Mr. Butler first reviewed the statistics and growth of the world's paint production. Although national production in the UK placed the industry fourth in world rank (after the US, Western Europe and Japan), inclusion of overseas affiliates of UK companies raised the industry to second place (US 2.4 m ton, UK+overseas 1.0 m ton). In ten years from 1955, US production increased from $1.5 billion value to $2.0 billion and the projection to 1970 was $2.5 billion. Industrial paints had a similar growth rate in the past to trade and decorative lines, but in future industrials were expected to gain an increased share of the total ; a similar growth pattern was expected in Western Europe.

In the last ten years growth of production in Western Europe and Scandinavia had been at 7 per cent per annum, in the UK at 2.8 per cent per annum. In both the UK and Europe paint trade growth had been much slower than in the chemical or plastics industries.

Mr. Butler noted that TiO_2 consumption in the UK per unit volume of paint was higher than in Western European countries, and that while in the UK of all raw materials 14 per cent was TiO_2 and 17 per cent extenders, in the US, 11 per cent was TiO_2 and 17 per cent extenders. He questioned whether the UK TiO_2 usage was at a higher rate than necessary.

By examining past statistics and extrapolating these, Mr. Butler predicted the changes to be anticipated in raw material usage in the US and in the UK, grouping these raw materials under the headings of pigments, additives, intermediates, resins and polymers.

Turning next to investment in development, the lecturer drew attention to the time scale involved in a major project. Taking research, development, plant construction and commissioning together, there might be a period of 4 to 4½ years during which a project was in the red ; only after this time would profits be earned to pay off the original investment.

Mr. Butler then compared the operating costs of the UK and US paint industries broken down into raw materials, production, and distribution costs. Rebates apart, there were no large differences in the break-down of costs in the two countries and the final profits expressed as a percentage of selling price were very close. Attention was drawn to raw materials—43 per cent of selling price, and selling, distribution and general expenses totalling 20 per cent of selling price and to the fact that a 2 per cent reduction in raw material costs could increase the profit by 10 per cent. In these two high areas of expenditure, a continuous watch on costs was very necessary and would bring worthwhile returns.

It was pointed out that the selling price of paint was about one-fifth of the total cost of a painting job and that application methods need improvement to bring down total costs. This applied mainly in the decorative trade ; in industrial finish applications, improved methods such as electrostatic spraying, curtain and coil coating were in use and expanding.

Mr. Butler concluded by listing and discussing management systems which could be used to assist research and development procedures. Many methods were available such as project analysis, network analysis, resource allocation, statistical design, optimisation, method study, linear and non-linear programming and simulation. The object of selecting and using any of these techniques was to reduce the time taken to complete a project.

A lively and informative discussion followed in which some 20 members contributed. At the close the formal vote of thanks was proposed by Mr. N. Ashworth.

D.A.P.

Scottish

Towards automation

" Towards automation " was the title of a stimulating lecture given by Mr. H. R. Touchin, Consultant Scientist, to the members of the Scottish Section on 18 January in the Lorne Hotel, Glasgow.

Mr. Touchin opened by stressing the need for the paint industry to look for the modern and future tools of production, as exemplified elsewhere in the chemical industry, of which automation was one. In spite of the small size of most paint manufacturing units, automation could usefully be applied. It did not necessarily involve the use of a computer, and could well be introduced in stages, after a critical review of present methods to show where these could be adapted for instrumentation and control.

Automation was distinguished from mechanisation, and defined by the four stages of measurement, comparison with standard, feed to a controller and adjustment of the process. It could give benefits in productivity, elimination of human error, less batch to batch variation, economy of materials and safety. Each stage in the paint-making process required examination for suitable variables, either of the " input " type, e.g. speed or power, or of the " quality " type, e.g. colour, viscosity, specific gravity.

Mr. Touchin then took examples from each part of the paint-making process to illustrate his views. Weighing and measuring of raw materials could be very expensive in manpower, but could easily be automated by using load cells, weighing vessels or level controllers, perhaps linked to a programmed controller to charge premixes in the correct order. On a grinding mill, control of such variables as speed, power consumption or temperature could help to ensure consistent production. At the let-down stage, the " quality " variables entered the picture, and an example of solvent addition controlled by mix viscosity was described. One could visualise the fully automated operation of a change pan mixer, with mechanised guidance of the pan, lowering and starting of the mill head, followed by additions of resin, solvent etc. to a standard viscosity or specific gravity, and completed by removal of the pan and restarting of the cycle. Some types of programme controller which might be suitable for such a unit were illustrated.

A hypothetical continuous plant for a simple white emulsion paint was then outlined and Mr. Touchin pointed out that if a product such as beer could be produced continuously on an automated plant, as had been done, it should not be impossible to make progress in the paint field. Such progress was essential if the best use was to be made of all the industry's assets—men, machines, materials and money.

The discussion covered such matters as the use of load cells, the problems of cleaning complex equipment between products, the ability of automated systems to cope with unexpected emergencies and batch vs. continuous production. Members felt that they had been given much food for thought, and a vote of thanks proposed by Dr. J. D. Easton was carried with acclamation.

J.D.E.

The composition and some uses of tall oil products

The December meeting of the Scottish section was held in the Lorne Hotel, Sauchiehall Street, Glasgow, on the 14th of the month, when Dr. K. S. Ennor of British Oxygen Chemicals Ltd. spoke on " The composition and some uses of tall oil products."

Dr. Ennor first discussed occurrence and separation. Crude tall oil was a by-product of the sulphate paper process and was the world's cheapest source of organic acids. The first effective separation of tall oil fatty acids and rosin on a commercial scale took place in the USA in 1949. About two years ago the Chemicals Division of BOC commissioned the United Kingdom's first tall oil fractionation plant. The process employed two columns in a three pass blocked operation. The most valuable products were tall oil fatty acids, rosin and distilled tall oil. Dr. Ennor then described the composition and uses of these three products separately.

Tall oil fatty acids were composed mainly of linoleic and oleic acids. In long oil length alkyds, Scandinavian tall oil fatty acids of low rosin content gave air drying rates similar to soya bean oil. These products gave a much faster dry than tall oil fatty acids of American origin. Scandinavian tall oil fatty acids gave less yellowing in the dark than soya bean oil in alkyd paints. A possible explanation was the presence in Scandinavian tall oil fatty acids of cis-5, 9, 12-octadecatrienoic acid, which reacted like a dienoic acid. On exterior exposure, tall oil alkyds gave slightly better results than soya bean oil alkyds. In the preparation of long oil length alkyds, a maximum of about 30 weight per cent of the tall oil fatty acids could be replaced by linseed oil without using an alcoholysis step. This unusual procedure retained the short processing time of the fatty acid process.

Tall oil rosin contained mainly abietic and dehydroabietic acids. A convenient gelometer test had been developed to assess different methods of inhibiting rosin crystallisation. The most effective method was the reaction of rosin with paraformaldehyde. The applications of rosin were described briefly.

Distilled tall oil was a mixture of fatty acids and rosin acids, the fatty acid portion containing mainly C_{18} unsaturated acids. The chief rosin acids were tetrahydroabietic and pimaric acids, which were both relatively resistant to autoxidation. Distilled tall oil was used in low cost rosin-modified alkyds with good yellowing resistance. Limed distilled tall oil was used in marine paints and drum coatings.

The lecture was listened to with interest, and a brisk discussion followed. The meeting was closed by a vote of thanks proposed by Mr. C. MacLean.

J.D.E.

Eastern Branch

Dispersible organic pigments

The second technical meeting of the session, with Mr. G. H. Hutchinson in the chair, was held at the Wee Windaes Restaurant on Wednesday 22 November 1967 when Mr. A. Gray and Mr. P. Birrell spoke on " Dispersible organic pigments " to 25 members and guests.

Mr. Birrell introduced the subject by outlining various aspects of pigment dispersion, and the nomenclature associated with the subject, which was exemplified by a practical demonstration.

The need for dispersible pigments was considered in the light of the manufacture and use of conventional organic pigments, with emphasis on the tendency of the pigment to flocculate, aggregate, and agglomerate during the filter pressing, drying and grinding operations.

Mention was made of pre-dispersed pigments (chips, flushed bases, master-batches etc.) and Mr. Birrell went on to review the progress made to date in producing easily dispersible pigments in relation to the paint, printing-ink and plastic industries and mention the various pigment ranges produced for each industry.

Mr. Birrell concluded his talk by outlining the most suitable types of dispersing equipment, emphasising the changes in present-day equipment by the advent of these pigments.

The lecture was illustrated with a large number of excellent slides and finished with a short film showing the dispersion of an easily dispersed pigment.

A lively discussion followed. The vote of thanks was proposed by Mr. C. Cochrane who congratulated the lecturers on an interesting and well-presented paper.

J.H.S.

A general review of resins for printing inks

The third technical meeting of the session was held at the Wee Windaes Restaurant on Wednesday 13 December 1967 with Mr. G. H. Hutchinson in the chair. Mr. R. J. Joulier spoke on " A general review of resins for printing inks " to 26 members and guests.

Mr. Joulier introduced the subject by outlining the five major printing processes, namely :—letterpress (conventional and web fed), direct and offset lithography (including heat-set web offset), photogravure, flexography, and silk-screen. The main outlets for these processes were discussed with regard to economy, and material to be printed—plastic, paper and metal.

After describing the basic formulation principles of inks for the various processes, he went on to discuss, at length, the resin requirements for these different types of ink, from the viewpoint of running on the press, the period just after printing, and long-term in the dried film, with particular respect to paper substrates.

Mr. Joulier concluded his talk by examining future resin requirements with regard to increased press speeds and the increasing variety of substrates to be printed.

A lively discussion followed, and the vote of thanks was proposed by Mr. J. H. Stewart who congratulated the lecturer on the manner in which he had covered a diverse subject.

J.H.S.

Thames Valley

The examination of pigments used by artists in paints

The third meeting of the 1967/68 winter programme was held at Beaconsfield on 29 November. This well attended gathering heard Miss Plesters, from the Scientific Dept. of the National Gallery, speak on " The examination of pigments used by artists in paints."

Miss Plesters said she intended to confine her talk to consideration of easel painting. First of all she traced briefly the history of pigments used by the painters since the earliest times, much of it being derived from careful studies of historical texts, e.g. the writings of Theophilus around AD 1200 and of Cennino Cennino of the early 1400's. Moving toward the present day, much more was known and by the 18th-19th century the amount of recorded data on the birth of pigments was extensive. She noted the interesting story of J. M. W. Turner's paint box which had been fully analysed by an industrial concern for presentation in the 1951 Festival of Britain.

Miss Plesters proceeded to outline the source and characteristics of many early pigments, amounting to about 30 in all, up to the year 1700. They included verdigris, natural ultramarine, umbers, carbon black, vermilion, lead white, dragons blood resin, gamboge, naturally occurring bitumens, and gold and silver leaf. For fresco and wall painters the list was restricted to the earth pigments. Like the history of pigments itself, the range available to the artist from this date was ever-widening and viridian, prussian blue, chromes, cadmium red and cobalt blues became used, until finally, in the present day, many brighter toned pigments were available, such as quinacridones, monastrals and much improved lakes.

The physical and chemical examination was done by removing ½-1 mm sections normally from a crack in the painting and, after freeing the pigment from its medium with either solvents or alkali, it was usually readily identified by means of optical and chemical tests. In the optical examination the binocular and polarising microscope played an important part, while chemical examination followed fairly traditional lines, being a combination of the precipitation and crystallisation reactions used in chemical microscopy and spot tests using organic reagents for metals. Some excellent slides backed up Miss Plesters' points on the identity of individual pigments, among them being enlarged cross-sections (layers), in colour, of samples from actual pictures and a number showing the form of some pigments after extraction. Other slides showed well the inability of restorers in distant or more recent times to exactly reproduce the original either in texture or particle size. Again the old masters had shown great skill in producing dramatic visual effect by means of transparent colour over solid colour, thus enhancing the saturation and brightness, which also contributed to difficulty in restoration.

Interesting examples of some difficulties encountered in cleaning and restoration were given, e.g., A Tintoretto Allegory, after removal of discoloured varnish, still had leaden grey skies, the reason probably being accounted for by an increase in the refractive index of the medium, thus lowering the opacity, combined with chemical reaction of the blue pigment with the oil medium. A further example was the Duchess of Milan painting by Holbein, which, having been slightly damaged comparatively early on in its existence, had had the blue background entirely repainted twice in the past, once in the 17th or 18th century (using the original pigment, but in a resin medium which had rapidly discoloured) and again in the 19th century (when the paint used contained prussian blue, a pigment unknown in Holbein's time). In a recent restoration carried out at the National Gallery, both layers of repaint were removed, revealing not only the original blue background but also a cast shadow of the figure which had previously not been visible.

Miss Plesters had, not surprisingly, amassed considerable specialised data, both on the pigment compositions of notable paintings and on the physical and chemical characteristics of the pigments themselves. She was thus, on occasion, able to help the criminal investigation authorities when questions of identity arose, as in cases of fake, forgery and theft.

More recent and sophisticated identification techniques were briefly referred to, such as X-ray spectrophotometry and an interesting new neutron activation analysis by some Dutch and German workers which would help to establish more accurately the original date of a picture and the geographical origin of some of the pigments.

Question time reflected the interest shown by the audience in what Miss Plesters had said. Dr. Mayne asked what the particle size range of pigments used by the old masters was, and the speaker replied that the range was such that restorers and forgers were given considerable difficulty using present-day pigment qualities. Some mineral pigments in old paintings tended to be coarsely ground to conserve their maximum colouring power, while other pigments were not greatly different in particle size from those used today. Mr. Hill asked about the incidence of mould on pictures, and in reply Miss Plesters referred to the Florence disaster, where she had seen many pictures with mould attack. Strangely the mould was usually growing on the surface soil or the wax or varnish coatings, and not on the paint itself.

Other questions were about the reasons for the impressive durability of vermilion before 1800 ; the difficulty restorers who did not reproduce the original pigments had; what was gamboge; the extent of use of poisonous pigments.

Mr. Manfred Hess proposed the vote of thanks, which was heartily endorsed by the audience.

R.E.G.

Notes and News

20 OCCA Technical Exhibition

More than 150 firms mentioned in the " Official Guide "

This year's Exhibition will be the largest in terms of ground space since the Technical Exhibitions began in 1949. There will be 108 exhibitors, with stands covering an area of 27,000 sq ft, and over 150 firms are mentioned in the *Official Guide* to the Exhibition.

The venue is Alexandra Palace, and the times of opening will be as follows :

Monday 25 March	3 p.m. to 6.30 p.m.
Tuesday 26 March	10 a.m. to 6 p.m.
Wednesday 27 March	10 a.m. to 6 p.m.
Thursday 28 March	10 a.m. to 6 p.m.
Friday 29 March	10 a.m. to 4 p.m.

A Plan of the Hall can be found on page 275, and a numerical list of exhibitors on pages 271-3. A map showing the main routes to Alexandra Palace is on page 274 ; free car parking space is available, and a free bus service will run at frequent intervals between Wood Green station, on the Piccadilly Line, and the Exhibition.

The Exhibition Luncheon will be held at the Savoy Hotel at 12.45 on Monday 25 March, when the Rt. Hon. Lord Erroll of Hale will reply to the Address of Welcome by the President. Lord Erroll will then open the Exhibition at 3 p.m.

Copies of the *Official Guide*, which has already been sent to all members of the Association and exhibitors, will be available free of charge at the Information Centre, Stand 51, telephone 01-883 4285, or advance copies may be obtained from the Association's offices.

Invitations have again been extended to parties of senior science students from local schools to visit the Exhibition on the mornings of 25, 27 and 28 March. The visitors will be given a short introductory talk by a member of the Association before touring the stands. The Technical Education Stand, manned by representatives of technical colleges and from industry, will feature in the tour.

To assist the growing numbers of overseas visitors, and the exhibitors, interpreters will be available on Stand 50, or at the Information Centre.

In case of accident, British Red Cross personnel will be present on Stand 30.

Refreshment facilities at Alexandra Palace include several buffets and bars, which will be open throughout the period of the Exhibition, and two restaurants with full dining facilities : the Edinburgh Room, where tables can be reserved and which offers a wide choice of dishes, and the Alexandra Room, where tables cannot be reserved.

A fully illustrated report of the Exhibition will appear in the May issue of the *Journal*, and will contain extracts from the speeches at the luncheon, and a review of the exhibits.

Latest Information

Since the *Official Guide* was printed the following information has been received :

Stand 14B—De La Rue Frigistor Limited

The main feature of the De La Rue Frigistor Company's stand will be the De La Rue *Climatest*. This is an advanced weathering machine available for use on external or internal facing materials. Present samples under test include many types of paints on a range of surfaces, plastics and anodic films.

Special features of the *Climatest* unit are : rapid weathering—ten years of natural weathering can be condensed into one " *Climatest*-year," close UV correlation—for accurate simulation of the sun's most harmful and penetrating rays, large sample capacity—to ensure that true effects are discovered for the backing as well as facing materials, and accurate controllable climatic conditions at the sample surfaces. In addition to sunshine, temperature and humidity levels, the De La Rue *Climatest* can simulate rainfall, frosting and dewing. The full technical details of the unit and the tests already completed will be available on the stand.

Stand 7—Pure Chemicals Limited

On 1 January 1968, Pure Chemicals Limited was sold by Rio Tinto Zinc to Noury and Van Der Lande NV, a major chemical subsidiary of the recently formed Dutch KZO Group, and parent company of Novadel Limited (Stand 78). In addition to acquiring PCL, Noury have also bought its joint venture in Germany, Interstab Chemie, together with its parent, Chemische Fabrik Hoesch.

Pure Chemicals Limited will continue to produce and sell not only their own stabiliser range but also their other products.

Between them Novadel and Pure Chemicals now market the UK's widest range of stabilisers for PVC, the two companies' ranges being complementary to each other.

Novadel will also continue to produce organic peroxides, accelerators, bulk chemicals and raw materials for the paint industry, and PCL their organic bromides, organo phosphorous compounds and special organic chemicals.

Stand 19—Ciba Clayton Limited

As well as the items described in the *Official Guide*, *Cromophtal* pigments in air-drying decorative paints will be shown.

Stand 78—Novadel Limited

Novadel will be featuring *Noury Drier 10* on their stand in addition to the products mentioned in the *Official Guide*.

A barium base composite drier, free of lead and calcium, *Noury Drier 10* is recommended for safflower, sunflower and other semi-drying oil alkyd paints which are liable to deterioration due to lead stearate formation, and in paints where loss of drying due to cobalt adsorption is evident.

Preliminary details will be presented of a newly-developed water soluble thermosetting acrylic resin, *Nourycryl*. It is hoped to show panels illustrating performance of stoved films.

Stand 68—Berk Limited

Berk *Fire Retardents* have now been classified under the general trade name *Flammex*. The general purpose grade, *Flammex 5BT*, will be shown. It is claimed that this product can be incorporated into emulsion systems without disturbing the manufacturing process, and with no loss of desirable paint properties.

Stand 52—British Titan Products Co. Limited

A feature of the BTP stand will be a display of the use of high-durability *Tioxide* pigment in coil-coating with PVC finishes to give at least a 15 year maintenance-free life outdoors.

Also shown will be a display of *Tioxide R-XL* as a base for tinting latex paint, demonstrating the economy resulting from the use of a white of high reducing power rather than a larger amount of a white of lower reducing power in this application.

Stand 21—Cray Valley Products Limited

It is now intended to show the following additional products on the Cray Valley Stand.

Synocryl acrylic resins. Two new crosslinking resins, a room temperature crosslinking acrylic resin for high performance coatings, and a low cost thermosetting acrylic resin which crosslinks with amino resins at 120°C giving performance equivalent to high quality alkyd melamine systems.

Resydrol water soluble resins, with emphasis on electrodeposition.

Synolac and *Gelkyd* resins.

Three new epoxy curing agents in the *Versamid* and *Synolide* ranges.

(a) A polyamide formulated principally for use in epoxy flooring compositions and solvent-free, high-build coatings.

(b) A water soluble polyamide type epoxy curing agent which extends the use of two-pack epoxy coatings to water-based systems.

(c) A novel epoxy curing agent which enables epoxy coatings to be applied and cured underwater to give tough coatings of good adhesion.

Versadyme dimer acid, for use in the production of various polymers.

Stand 22—Polyvinyl Chemie Holland NV

The Neorez range of moisture curing urethanes for use in seamless floors will be shown, together with examples of flooring.

Stand 38—Geigy (UK) Limited

Additional products to be shown by Geigy include :

Irgalite Pink FB, a ferrocyanide cationic dye complex with exceptional print brightness, which is economical compared to conventional PMA or PTMA equivalents.

Use of *Irgafin S* predispersed pigment powders in rigid pvc, showing the excellent transparency obtained.

A range of *Irgaphor* rubber masterbatches. End uses of the *Irgalite Paper* range of colours for paper coating and wallpaper printing.

Stand 77—Union Carbide Europe Inc.

New developments in additives, *Resin Grade Asbestos 144* and *244*, will be shown, as will EVA-co-mer resins, and self-adhering organosols and plastisols.

Stand 44—PIRA

Pira are now able to show recent work on the accuracy of ink-makers' fineness-of-grind gauges.

Stand 20—Ciba (ARL) Limited

Ciba will introduce a new grade of solid epoxy resin, *Araldite 7300*, with particularly good solubility properties and very compatible with other resins.

The latest developments in epoxy-based pretreatment primers and solvent-free underwater coatings will be shown.

Stand 65—BASF United Kingdom Limited

The address of BASF is Earl Road, Cheadle Hulme, Cheadle, Cheshire, and not as stated in the *Guide*.

Since the *Official Guide* was printed, the following stand telephone numbers have been received from the GPO :

Stand 14a—Hubron Sales Ltd.	01-883 5581
Stand 17—Bernd. Schwegmann KG	01-883 5523
Stand 20—Ciba ARL	01-883 5575
Stand 64—ICI Ltd.	01-883 5703
Stand 95—Jenag Equipment Ltd.	01-883 5599
Stand 106—Cornbrook Resin Co. Ltd.	01-883 5670

Change of Exhibitor

Stand 14—Kollmorgen (UK) Ltd.

Kollmorgen (UK) Ltd. has replaced the listed exhibitor for Stand 14 and is showing the same range of equipment for colour measurement and control as is described in the *Official Guide*. Kollmorgen (UK) Ltd. has assumed the United Kingdom interests for the Macbeth Corporation, Davidson and Hemmendinger, Inc., and the IDL division of Kollmorgen Corporation USA. A complete range of equipment for the evaluation, measurement and control of colour is now offered by Kollmorgen and will include the following well-known instruments :

1. Comic 1—analogue colour mixture computer.
2. Comic 2—digital colour mixture and general purpose computer.
3. Tristimulus integrator and spectrophotometer readout systems.
4. Colour difference, analysis and display computers.
5. On line colour control systems and shade sorting equipment.
6. Colour Eye range of abridged spectrophotometers and colorimeters.
7. The General Electric recording spectrophotometer.
8. The Trilac recording spectrophotometer.
9. The complete range of Macbeth daylight equipment and lighting cabinets for visual colour evaluation.

As well as sales and applications the new company will handle service and spare parts from its office presently located at 46 London Road, Reading, Berkshire.

Numerical List of Exhibitors

Stand	
1	Winkworth Machinery Ltd.
2	Hardman & Holden Ltd.
3	Vuorikemia OY
4	Pretema AG
5	Hygrotherm Engineering Ltd.
6	Joyce, Loebl & Co. Ltd.
7	Pure Chemicals Ltd.
8	Churchill Instrument Co. Ltd.
9	Tintometer Ltd.
10	John L. Seaton & Co. Ltd.
11	Chemische Werke Huels AG
12	Research Equipment (London) Ltd.
13	Herbert Smith & Co. (Grinding) Ltd.
14	Kollmorgen (UK) Ltd.
14a	Hubron Sales Ltd.
14b	De La Rue Frigistor Ltd.
15	Peter Silver & Sons (Engineers) Ltd.
16	DIAF A/S
17	Bernd. Schwegmann KG
18	Farbwerke Hoechst AG
18	Hoechst-Cassella Dyestuffs Ltd.
18	Hoechst Chemicals Ltd.
19	Ciba Clayton Ltd.
20	Ciba (ARL) Ltd.
21	Cray Valley Products Ltd.
22	Polyvinyl Chemie Holland NV
23	Shell International Chemical Co. Ltd.
24	British Celanese Ltd.
25	Grampian Press Ltd. (*Paint Manufacture*)
26	Albright & Wilson (Mfg.) Ltd.
27	William Boulton Ltd.
28	Titaandioxydefabriek Tiofine NV
29	Durham Raw Materials Ltd.
30	British Red Cross Society
31	Amalgamated Oxides (1939) Ltd.
32	Draiswerke GmbH
33	Resinous Chemicals
34	Sterling Colour Co. Ltd.
35	BP Chemicals (UK) Ltd.
35	British Resin Products Ltd.
36	Spelthorne Metals Ltd.

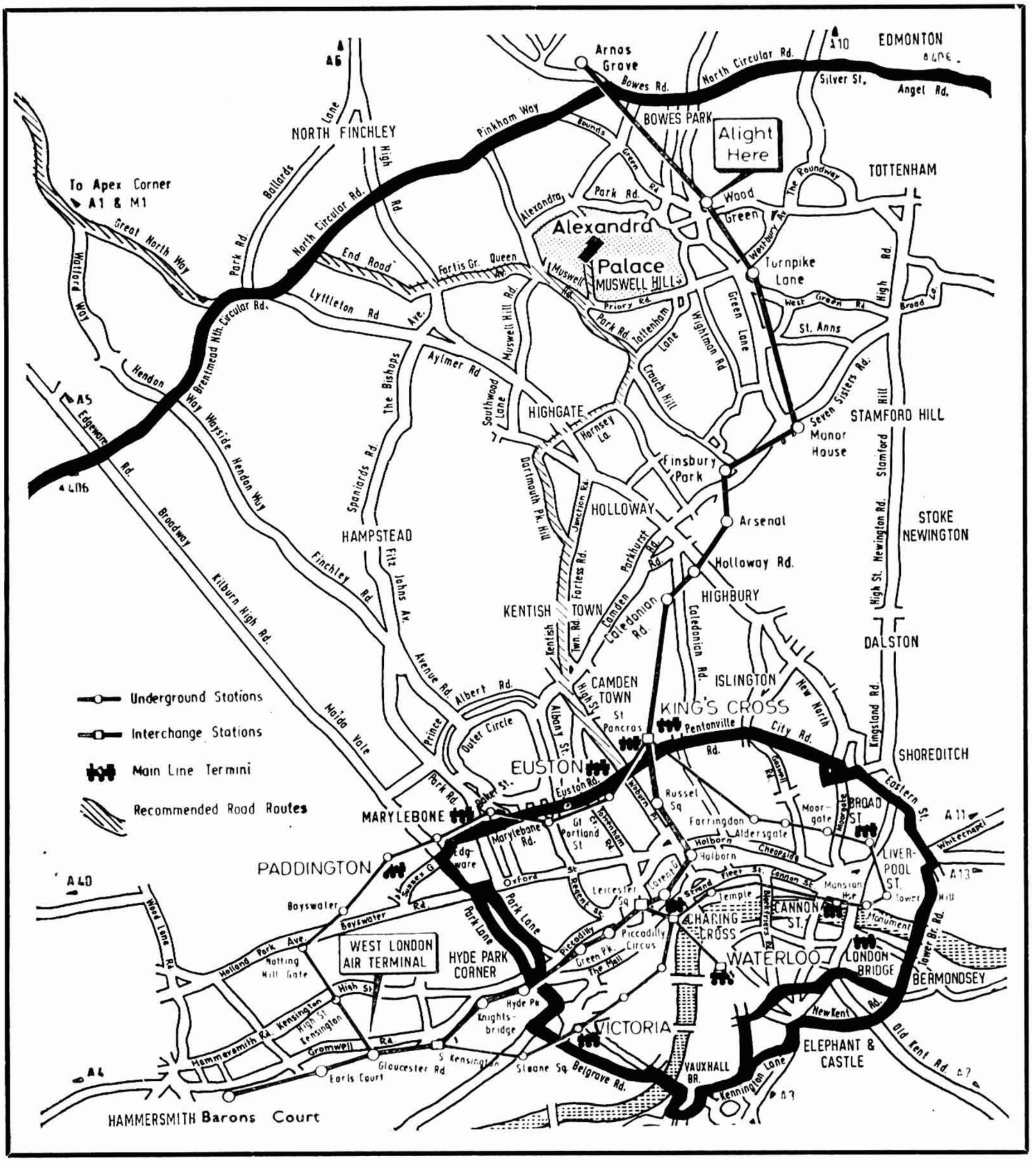
Numerical List of Exhibitors—*continued*

Stand	
37	Farbenfabriken Bayer AG
38	Geigy (UK) Ltd.
39	Surface Coating Synthetics Ltd.
40	Beck, Koller & Co. (England) Ltd.
41	Amoco International S/A
42	Kingsley & Keith (Chemicals) Ltd.
43	Allied Chemical Corporation
44	PIRA
45	DH Industries Ltd.
46	Kronos Titanium Pigments Ltd.
47	Paint Research Station
48	Dow Chemical (UK) Ltd.
49	National Provincial Bank Ltd.
50	Interpreters
51	Oil & Colour Chemists' Association Information Centre
52	British Titan Products Co. Ltd.
53	Titanium Intermediates Ltd.
54	Joseph Crosfield & Sons Ltd.
55	*Paint, Oil & Colour Journal*
56	Laporte Industries Ltd.
57	Sheen Instruments (Sales) Ltd.
58	Sawell Publications Ltd. (*Paint Technology*)
59	Scado-Archer-Daniels NV
60	Rex Campbell & Co. Ltd.
60	The Chemical Supply Co. Ltd.
61	Anchor Chemical Co. Ltd.
62	Rhone-Poulenc
63	Holmes Bros. Paint Machinery Ltd.
63	Torrance & Sons Ltd.
64	Imperial Chemical Industries Ltd.
65	BASF United Kingdom Ltd.
66	Silverson Machines Ltd.
67	Microscal Ltd.
68	Rudolph Meyer's Inc.
69	Columbian International (GB) Ltd.
70	Vinyl Products Ltd.
71	J. H. Little & Co. Ltd.
72	BIP Chemicals Ltd.
73	The Baker Castor Oil Co.
74	Ferranti Ltd.
75	Molteni Off. Mecc. of Milan
75a	DIA Chemie
76	AMF International
77	Union Carbide Europe Inc.
78	Novadel Ltd.
79	Technical Education
80	Harlow Chemical Co. Ltd.
81	Modo Products (Chemicals) Ltd.
82	Torsion Balance Co. Ltd.
83	Svenska Oljeslageri Aktiebolaget
84	Elcometer Instruments Ltd.
85	Carless, Capel & Leonard Ltd.
86	Berk Ltd.
87	Synthese, Kunstharsfabriek, NV
88	International Colloids Ltd.
89	Dott. Edoardo Meazzi
90	A. V. Pound & Co. Ltd.
90	Tenneco Chemicals Inc.
91	British Iron and Steel Research Association
92	Mastermix Engineering Co. Ltd.
93	Shawinigan Ltd.
94	Metal Propellers Ltd.
95	Jenag Equipment Ltd.
96	Bush, Beach & Segner Bayley Ltd.
96	Degussa
96	Wacker Chemie
97	Maschinenfabrik Heidenau, Veb.
97	Unitechna
98	Marchant Bros. Ltd.
99	Chemieanlagen GmbH
99	Plastmaschinenwerk Freital
100	Esso Chemical Ltd.
101	Lennig Chemicals Ltd.
102	Cox's Machinery Ltd.
103	Cornbrook Resin Co. Ltd.
104	Sachtleben AG
105	Fredk. Boehm Ltd.

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

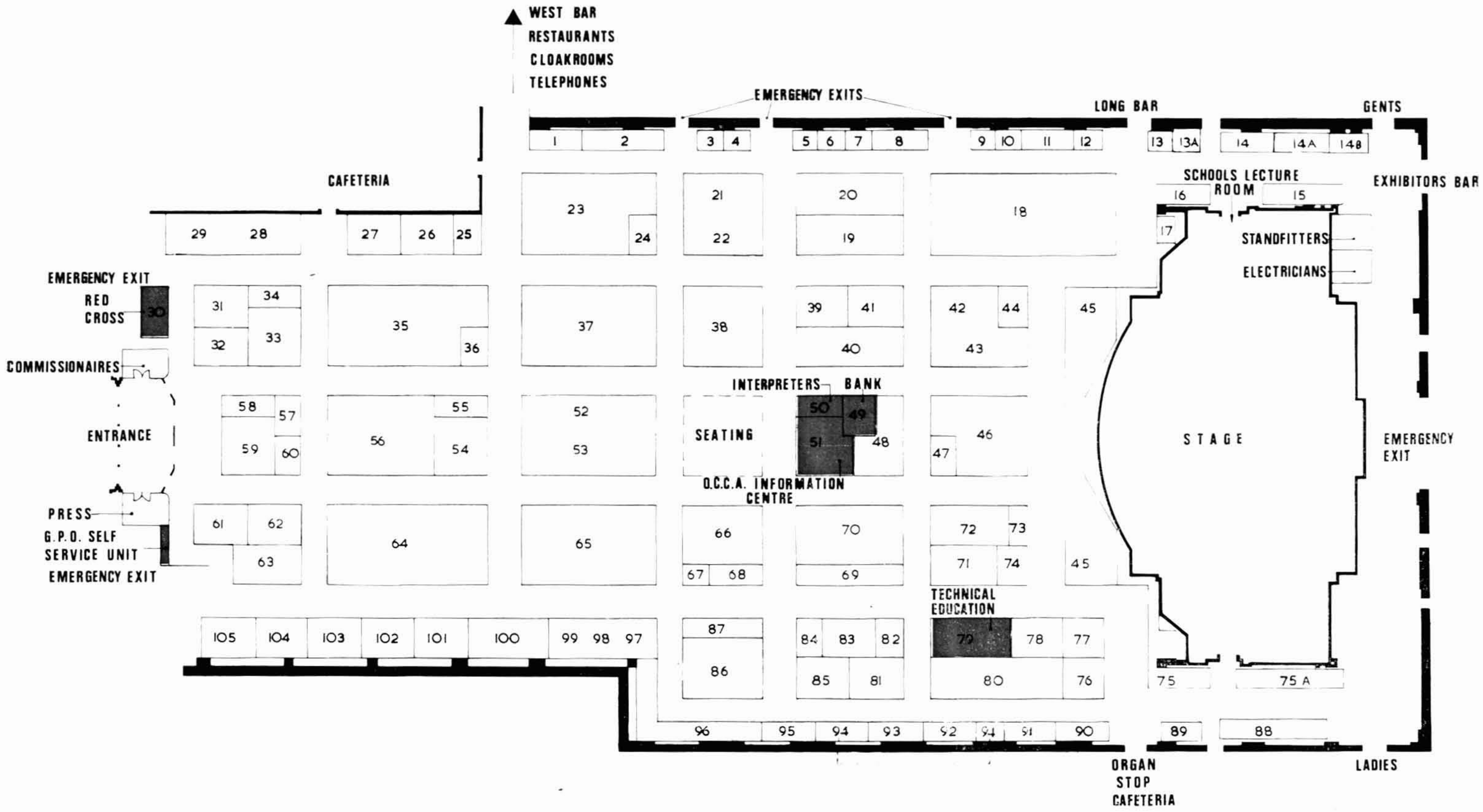
Stand	*Company*	*Official Guide Page No.*
59	ADM Chemicals	140
45	Ateliers Sussmeyer	84, 102
13	Barker & Aspey Ltd.	148
81	Berol Company	119
33	British Paints Ltd.	134
90	Butler Chemicals Ltd.	156
90	Butler Malros Ltd.	156
46	Canadian Titan Pigments Ltd.	106
31	Canadian Zincoli Pigment Ltd.	50
18	Chemische Werke Albert	93
19	CIBA Ltd.	76
46	Derives du Titane SA	106
2	du Pont de Nemours, E.I., & Co.	98
89	Erlich, G. J., Ltd.	115, 120
86	Georgia Kaolin Co.	62
41	Greeff, R. W., & Co. Ltd.	52, 132, 152
11	Greenham, Herbert G. F., & Co. Ltd.	78
43	Harmon Colours Ltd.	49
71	Henkel International GmbH	110
45	Hockmeyer USA	84
45	Kupper, Peter, AMK	84
32	KW Chemicals Ltd.	86
17	Lovelock, E. J. R.	143
61	Marbon Chemical Division of Borg-Warner	56
81	Mo och Domsjö AB	118
29	Morehouse International Ltd.	89
92	Myers Engineering Ltd.	114
46	National Lead Group	106
60	Necof NV	74
14	New Instrument Laboratories Ltd.	57
78	Noury & Van der Lande NV (Holland)	121
78	Oelwerke Noury & Van der Lande GmbH (West Germany)	121
45	Pamasol GmbH	84
62	Pechiney-St.-Gobain	132
42	Pontyclun Chemical Co.	105
90	Pound, A. V., & Co. Ltd.	155
80	Revertex Ltd.	98
69	Runnymede Dispersions Ltd.	81
45	Schold Welding Ltd.	84
42	Sherwin Williams Co.	106
62	Societe Redis	132
31	Stolberger Zincoli GmbH	50
42	Thos. Swan and Co. Ltd.	105
46	Titan Co. AS	106
46	Titangesellschaft mbH	106
90	NV Transicol	156
42	Trojan Powder Co.	105
96	Vickers Ltd. Engineering Group	113
45	Vree, J. de, & Co. Ltd.	84
42	Warwick Chemical Co. (Yorkshire) Ltd.	105

HOW TO REACH ALEXANDRA PALACE



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

Plan of Exhibition



Analysis of Exhibits

Exhibitor	Stand No.	Resins	Pigments	Extenders	Additives	Solvents	Oils	Machinery	Lab. Machinery	Instruments	Misc.
Albright & Wilson (Mfg) Ltd.	26		●		●						Chemicals
Allied Chemical Corporation	43		●		●						Low MW polyethylenes
Amalgamated Oxides (1939) Ltd.	31		●								
AMF International ..	76							●	●		
Amoco International S/A	41	●									Chemicals
Anchor Chemical Co. Ltd.	61	●	●		●						Curing agents
AVO International ..	14									●	
Baker Castor Oil Co., The	73				●						Thixotropic agents
BASF United Kingdom Ltd.	65	●	●								Adhesives
Beck, Koller & Co. (England) Ltd. ..	40	●									
Berk Ltd.	86		●	●	●						Filter cartridges
BIP Chemicals Ltd. ..	72	●									
Boehm, Fredk., Ltd. ..	105	●			●						
Boulton, William, Ltd. ..	27							●	●		
BP Chemicals (UK) Ltd.	35					●					Plasticisers, monomers
British Resin Products Ltd.	35	●									
British Iron & Steel Research Association	91										Research Association
British Celanese Ltd. ..	24	●			●						
British Titan Products Co. Ltd.	52		●								

Exhibitor	Stand No.	Resins	Pigments	Extenders	Additives	Solvents	Oils	Machinery	Lab. Machinery	Instruments	Misc.
Bush, Beach & Segner Bayley Ltd.	96	●	●	●	●						
Campbell, Rex, & Co. Ltd.	60	●	●		●	●	●				
Carless, Capel & Leonard Ltd.	85	●				●					
Chemische Werke Huels AG	11	●				●					PVC, plastisols, organosols, copolymers, latices
Churchill Instrument Co. Ltd.	8									●	
Ciba (ARL) Ltd. ..	20	●									
Ciba Clayton Ltd. ..	19		●								
Columbian International (GB) Ltd.	69		●								
Cornbrook Resin Co. Ltd.	103	●									
Cox's Machinery Ltd. ..	102							●			
Cray Valley Products Ltd.	21	●									
Crosfield, Joseph, & Sons Ltd.	54			●	●						
De La Rue Frigistor Ltd.	14b									●	
D. H. Industries Ltd. ..	45							●	●		
DIA Chemie	75A	●	●				●				Import and export organisation
Diaf A/S	16							●	●	●	
Dow Chemical (UK) Ltd.	48	●			●	●					
Draiswerke GmbH ..	32							●	●		
Durham Raw Materials Ltd.	29	●			●			●	●		
Elcometer Instruments Ltd.	84									●	

Exhibitor	Stand No.	Resins	Pigments	Extenders	Additives	Solvents	Oils	Machinery	Lab. Machinery	Instruments	Misc.
Esso Chemical Ltd.	100					●					
Farbenfabriken Bayer AG	37	●	●								
Ferranti Ltd.	74									●	
Geigy (UK) Ltd.	38		●								
Grampian Press Ltd.	25										Technical journal
Hardman & Holden Ltd.	2		●		●						
Harlow Chemical Co. Ltd.	80	●									
Hoechst Chemicals Ltd.	18	●	●								
Hubron Sales Ltd.	14A	●	●								
Hygrotherm Engineering Ltd.	5							●			
Imperial Chemical Industries Ltd.	64	●	●		●	●					
International Colloids Ltd.	88		●								
Jenag Equipment Ltd.	95							●	●		
Joyce, Loebl & Co. Ltd.	6									●	
Kingsley & Keith (Chemicals) Ltd.	42	●	●								Resin intermediates
Kronos Titanium Pigments Ltd.	46		●								
Kunstharsfabriek Synthese NV	87	●									
Laporte Industries Ltd.	56	●	●		●	●					Organic and inorganic peroxides, thixotropic agents
Lennig Chemicals Ltd.	101	●									
Little, J. H., & Co. Ltd.	71				●	●					Wetting agents, thixotropic agents

Exhibitor	Stand No.	Resins	Pigments	Extenders	Additives	Solvents	Oils	Machinery	Lab. Machinery	Instruments	Misc.
Marchant Bros. Ltd. ..	98							●	●		
Maschinenfabrik Heidenau, Veb ..	97							●			
Mastermix Engineering Co. Ltd., The	92							●	●		
Meazzi, Dott. Edoardo ..	89							●			
Metal Propellers Ltd. ..	94							●			
Meyer's, Rudolph, Inc. ..	68									●	
Microscal Ltd.	67									●	
Modo Products (Chemicals) Ltd.	81				●	●					Plasticisers
Molteni Off Mecc. of Milan	75							●	●		
Novadel Ltd.	78				●						Components for polyurethane, resin intermediates
Paint, Oil & Colour Journal	55										Technical journal
Paint Research Station ..	47										Research Association
PIRA											Research Association
Plastmaschinenwerk Freital	99							●			
Polyvinyl Chemie Holland NV	22	●									
Pretema AG	4									●	
Pure Chemicals Ltd. ..	7				●						Catalysts
Research Equipment (London) Ltd.	12									●	
Resinous Chemicals Ltd.	33	●									

Exhibitor	Stand No.	Resins	Pigments	Extenders	Additives	Solvents	Oils	Machinery	Lab. Machinery	Instruments	Misc.
Rhone-Poulenc	62	●			●	●					
Sachtleben AG	104		●	●							
Sawell Publications Ltd.	58										Technical publications
Scado-Archer-Daniels NV	59	●									
Schwegmann, Bernd., KG	17				●						
Seaton, John L., & Co. Ltd.	10						●				
Shawinigan Ltd.	93	●									
Sheen Instruments (Sales) Ltd.	57									●	
Shell International Chemical Co. Ltd.	23	●				●					
Silver, Peter, & Sons (Engineers) Ltd.	15							●	●		
Silverson Machines Ltd.	79							●	●		
Smith, Herbert, & Co. (Grinding) Ltd.	13							●			
Spelthorne Metals Ltd.	36		●								
Sterling Colour Co. Ltd.	34		●								
Surface Coating Synthetics Ltd.	39	●									
Svenska Oljeslageri Aktiebolaget	83	●									
Tenneco Chemicals Inc.	90	●			●	●					Plasticisers, catalysts
Tintometer Ltd.	9									●	
Titaandioxydefabriek Tiofine, NV	28	●									
Titanium Intermediates Ltd.	53				●						Media for heat-resistant paints

Exhibitor	Stand No.	Resins	Pigments	Extenders	Additives	Solvents	Oils	Machinery	Lab. Machinery	Instruments	Misc.
Torrance & Sons Ltd.	63							●	●		
Torsion Balance Co. Ltd.	82									●	
Union Carbide Europe Inc.	77	●									
Vinyl Products Ltd.	70	●									
Vuorikemia Oy	3		●								
Wacker Chemie	96	●									
Winkworth Machinery Ltd.	1							●	●		

Fiftieth Anniversary Celebrations

The details of the celebrations arranged for the Association's Fiftieth Anniversary in May were given in the February issue of the *Journal* and also on a form circulated to all Members in January. Members are reminded that the closing date for applications for tickets is 25 April. The Dinner on 10 May at the Savoy Hotel, London, W.C.2 replaces the Association's normal biennial dinner and applications for this function are *not restricted to members*. Single tickets cost £3 13s. 6d. and applications should be sent to the Association's office as soon as possible.

The Council is pleased to announce that The Rev. Joseph Robinson, M.TH., Canon Residentiary at Canterbury Cathedral, has agreed to conduct the Commemorative Service at the Parish Church of St. Vedast-alias-Foster on Thursday 9 May.

Amongst those who have accepted the Council's invitation to join in the Fiftieth Anniversary celebrations are the following :

The Presidents of :

- Parliamentary and Scientific Committee
- Institute of Metal Finishing
- British Plastics Federation
- Paintmakers Association
- Plastics Institute
- Institution of the Rubber Industry
- Federation of Societies for Paint Technology
- Society of Chemical Industry
- Research Association of British Paint, Colour and Varnish Manufacturers
- Chemical Industries Association
- Royal Institute of Chemistry
- Society of Dyers and Colourists

The Chairmen of :

- British Standards Institution
- Colour Group
- British Colour Makers Association
- Council of PIRA

The Masters of :

- Worshipful Company of Painter-Stainers
- Worshipful Company of Wax Chandlers.

Many members of the Press have accepted an invitation to be present at the Reception and Exhibition on 10 May at Wax Chandlers' Hall.

The printing of the History of the Association is now taking place and copies will be despatched to all Members of the Association early in May. In addition a Commemorative Booklet has been prepared which will be issued to all those joining in any of the celebrations on 9 and 10 May.

Association Conference, 1969

The Association conference in 1969 is to be held at Eastbourne from 17-21 June. The title chosen is " Film formation and curing," and several papers have already been promised. Further details will appear from time to time in the Journal.

IXth FATIPEC Congress

A provisional programme has now been produced for the IXth FATIPEC Congress, to be held at the Palais des Congres, Brussels, from 12-18 May 1968.

Nine plenary lectures have so far been scheduled, by speakers from Germany, Belgium, France, Italy, Holland, Switzerland, Scandinavia and the USA, and a paper from Great Britain, to be given by Mr. L. A. Tysall, an Ordinary Member attached to the London Section, and his co-author, Mr. A. J. Snowden. Eighty-seven communications will also be presented. The subjects covered under the main title of " Fundamental aspects of test methods peculiar to the industry of paint, varnish, printing inks and related products," are broadly subdivisible into three sections :

1. Tests on raw materials.
2. Tests on applied products.
3. Tests on liquid products—rheology —mechanical properties, general science, methodology.

The Congress will open at 4 p.m. on Sunday 12 May, and close on 5 p.m. on Friday 17 May. A full programme of social events has been arranged. Application and forms and further details are available from the Congress Secretariat, 49 Square Marie-Louise, Brussels, 4.

New Zealand Convention, 1968

The 6th Annual Convention of the New Zealand Sections is to be held at the Wairakei Hotel from 21-23 June 1968.

Papers covering any technical or general interest subject pertinent to the surface coating industry are invited, and should preferably occupy about 45-60 min. for presentation.

It will be noted that the New Zealand Convention follows the Australian Function by one week and therefore presents a unique opportunity for visitors to meet OCCA delegates from both countries within a brief period.

Inquiries should be addressed to :

G. F. Browne,
Hon. Publications Officer,
Wellington Section,
C/o Stauffer Chemical Co. (NZ) Ltd.,
P.O. Box 30343,
Lower Hutt,
New Zealand.

Plastics Institute—New Headquarters

The Director and Secretary represented the Association at the official opening, by Sir Peter Allen, of the new headquarters of the Plastics Institute at 11 Hobart Place, London, S.W.1, on 23 January. The Association congratulates the Institute on the acquisition of the lease of this imposing building, which has been made possible by covenants and donations from members and companies.

Irish Section

Left to right: Mrs. Kershaw, Mr. Kershaw (Hon. Treasurer, Irish Section), Mr. R. N. Wheeler (Chairman, London), Mrs. Wheeler, Mrs. Sharp, Mr. F. D. H. Sharp (Hon. Sec., Irish Section), Mrs. Sowerbutts, The President (Mr. F. Sowerbutts), Mr. R. Adam (Chairman, Irish Section), Mrs. Adam, Mr. R. H. Hamblin (General Secretary), Mrs, Hutchinson, Mr. G. H. Hutchinson (Chairman, Eastern Branch, Scottish Section)

Annual Dinner and Dance

The Irish Section's Annual Dinner and Dance was held at the Clarence Hotel, Dublin, on Thursday 14 December 1967, in the presence of the Chairman, Mr. R. A. Adam, and Mrs. Adam. The principal guests were Mr. F. Sowerbutts (President) and Mrs. Sowerbutts. The other guests were Mr. R. H. Hamblin (General Secretary), Mr. R. N. Wheeler (Chairman, London Section) and Mrs. Wheeler, Mr. G. H. Hutchinson (Chairman, Eastern Branch, Scottish Section) and Mrs. Hutchinson.

At the Official Reception before the dinner, Mrs. Sharp presented bouquets to Mrs. Adam and Mrs. Sowerbutts.

The toast to the Irish Section was proposed by Mr. Sowerbutts, who outlined the history of the Association.

The response was given by the Chairman, Mr. R. Adam, who thanked our cross-channel visitors for their attendance and co-operation with the Department of Agriculture over the precautions against foot and mouth disease, saying that he was delighted that they had all been able to make the journey. He pointed out that they must be the only delegation to an OCCA function who had had to be disinfected before being allowed to attend.

The final speech of the evening was the toast to our guests, which was ably proposed by the Treasurer, Mr. J. Kershaw.

The room was then cleared, and dancing commenced and continued until the early hours of the next morning.

Once again a happy, lively party where informality was the main theme.

Obituary

Mr. James Ward

It is with regret that we record the death on Christmas morning of Mr. James Ward, an Ordinary Member attached to the Irish Section and a founder member of the earlier Irish Branch of the Bristol Section.

Mr. Ward, who was 57, had come to Dublin from Darwen, Lancashire, in

1933 as Works Manager of The Walpamur Co. (Ireland) Limited. However, due to ill-health he had resigned from the Board of the Company some months ago. He had served a total of 42 years with The Walpamur Co., during which time he had made many friends in the industry.

He will be sadly missed by his colleagues and all who knew him.

News of Members

Mr. J. E. Mitchell, Ordinary Member, and member of the Manchester Section Committee, Chief Chemist, Decorative Division, Walpamur Co., has been elected a Fellow of the Royal Institute of Chemistry.

Mr. W. P. J. Baily and Mr. A. C. Fletcher, Ordinary Members attached to the London Section, have been appointed to the Board of Vinyl Products Limited.

Mr. Baily is Assistant to the Technical Director, and Mr. Fletcher is Technical Service Manager.

Mr. G. H. Marshall, an Ordinary Member attached to the London Section, has been appointed a Director of Horace Cory & Co. Ltd. Mr. Marshall is Technical Manager of the Company.

Mr. L. O. Kekwick

Mr. L. O. Kekwick, an Ordinary Member attached to the London Section, retired from his position of Chairman and Managing Director of Amalgamated Oxides (1939) Ltd., at the end of 1967. Mr. Kekwick was President of the Association from 1951-53, Vice-President from 1957-59, and Australian Section Representative on Council from 1954-63. He also held the offices of Treasurer and Chairman of London Section, and during his term as Chairman was instrumental in the staging of the small Technical Exhibition at Borough Polytechnic, the first of the OCCA Technical Exhibitions. Mr. Kekwick is currently Chairman of the Dartford Hospital Management Committee, a governor of the North West Kent College of Technology and a Commissioner of Dartford and Crayford Navigation, and so will have plenty to occupy his time in retirement.

Dr. H. W. Keenan

Dr. H. W. Keenan, an Ordinary Member attached to the London Section, has retired from his position as Technical Director of Beck, Koller & Co. Ltd.

Dr. Keenan served on Council from 1939-40, and was Honorary Research and Development Officer in 1944 until his election as President in the same year, which office he held until 1947. He was also a Vice-President from 1955-57, Vice-President on Council from 1961-63, and Chairman of London Section from 1940-42.

Dr. Keenan has long taken an interest in education in the industries, and is at present Chairman of the Technical Education Committee.

SLF changes name

Skandinaviska Lackteknikers Forbund (SLF) has changed its English name to " Federation of Scandinavian Paint and Varnish Technologists."

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

DEWHURST, SYLVIA MIRANDA, B.SC., Windermere, 168 Galleywood Road, Chelmsford, Essex. *(London)*

DUFVA, LENNART, B.A., AB Wilh Becker, Fack, Stockholm, 9, Sweden. *(Overseas)*

FOTHERGILL, JOHN ROBERT, L.R.I.C., International Paints Ltd., Stoneygate Lane, Felling, Gateshead, 10. *(Newcastle)*
HAILS, GEORGE, L.R.I.C., International Paints Ltd., Stoneygate Lane, Felling, Gateshead, 10. *(Newcastle)*
JOHNS, NICHOLAS, B.SC., 6 Thomas Street, Loughborough, Leics. *(Midland—Trent Valley)*
KELSEY, GEORGE COLIN, 2 Cedar Drive, Sutton at Hone, Nr. Dartford, Kent. *(London)*
KERRISON, DENNIS JOHN GRAHAM, L.R.I.C., 23 Horace Road, Forest Gate, London, E.7. *(London)*
LOWREY, KENNETH WILLIAM, International Paints Ltd., Stoneygate Lane, Felling, Gateshead, 10. *(Newcastle)*
POLSON, IAN SINCLAIR, L.R.I.C., 38 Mayfield Gardens, Brentwood, Essex. *(London)*
PORTEOUS, BARRIE MILROY, B.SC., 7 North Gyles Road, Edinburgh, 12. *(Scottish—Eastern)*
SVEDIN, CARL GUSTAV, c/o AB Wilh Becker, Postbox, Stockholm, 9, Sweden. *(Overseas)*

Junior Members

ELLIOTT, RICHARD, 44 McKerrell Street, Paisley, Scotland. *(Scottish)*
HILL, CLIVE LESLIE, 26 Vicarage Road, Winslow, Bletchley, Bucks. *(Thames Valley)*
MACKAY, ANGUS FRANCIS, Alexander, Fergusson & Co. Ltd., 50 Ruchill Street, Glasgow, N.W. *(Scottish)*
MORRISON, DONALD JOHN, 98 Glenmuir Drive, Glasgow, S.W.3. *(Scottish)*
NICHOLSON, COLIN, International Paints Ltd., Stoneygate Lane, Felling, Gateshead, 10. *(Newcastle)*

Forthcoming Events

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

Monday 4 March

Hull Section. " Towards Automation," by Mr. H. R. Touchin, to be held at the Hull College of Technology, at 7.00 p.m.

Midlands Section—Trent Valley Branch. " Work of the Forensic Science Laboratory," by a member of East Midland Forensic Science Laboratory, to be held at the British Rail School of Transport, London Road, Derby, at 7.30 p.m.

Friday 8 March

Manchester Section. Short Lectures and Discussion Evening, " Instrumental Colour Matching," with contributions by a number of firms using instrumental methods, to be held at the Liverpool Building and Design Centre, at 6.30 p.m.

Monday 11 March

London Section—Southern Branch. " Testing and Investigation of Marine Paints," by Dr. A. M. van Londen, Ing., to be held at the Keppel's Head Hotel, The Hard, Portsmouth, at 7.00 p.m.

Tuesday 12 March

Newcastle Section—" Testing and Investigation of Marine Paints," by Dr. A. M. van Londen (TNO), to be held in the Royal Turk's Head Hotel, Newcastle upon Tyne, at 6.30 p.m.

West Riding Section. " Dispersible Pigments," by a member of the staff of the Dyestuffs Division of ICI Ltd., to be held at the Griffin Hotel, Boar Lane, Leeds, 1, at 7.30 p.m.

Thursday 14 March

Scottish Section. " Colour Matching," by Mr. J. Beresford (Geigy (UK) Ltd., Pigments Division), to be held in the Lorne Hotel, Sauchiehall Street, Glasgow, at 6.00 p.m.

Thames Valley Section. " Choice of Colours in Automobiles," by a representative from Ford Motor Co., to be held at the Royal White Hart Hotel, Beaconsfield, Bucks., at 7.00 p.m.

Friday 15 March

Midlands Section. Newton Friend Lecture, " Furs," by Mr. G. A. Smith, to be held at the Chamber of Commerce House, Birmingham, 15, at 6.30 p.m.

Saturday 16 March

Scottish Section—Student Group. " Paint Specifications," by Mr. W. W. Galbraith (Montgomerie-Stobo Ltd.), to be held in the Lorne Hotel, Sauchiehall Street, Glasgow, at 10.00 a.m.

Wednesday 20 March

Scottish Section—Eastern Branch. Annual General Meeting at 7.00 p.m. Followed by Lecture " Work Study and Other Specialist Departments," by Professor Gloag (Heriot-Watt University).

Thursday 21 March

London Section. European Liaison Lecture, " Co-operation : A Necessity for Paint and Printing Ink Research," by H. K. Raaschou Nielsen, to be held in the New Engineering Block, University College, London, W.C.1, at 6.30 p.m.

Friday 29 March

Bristol Section. " Modern Analytical Techniques," by Mr. R. E. Coulson, Imperial Smelting Corporation Ltd., to be held at the Royal Hotel, Bristol, at 7.15 p.m.

Wednesday 3 April

Manchester Section—Junior Members. " Organic Pigments for Paint," by Mr. G. T. Williams (Geigy (UK) Ltd.), to be held at the Manchester Literary and Philosophical Society, at 4.00 p.m.

Thursday 4 April

Hull Section. Symposium—" Industrialised Building : Surface Coating Problems," to be held at the Department of Chemistry, The University, Hull, 9.30 a.m. to 4.30 p.m.

Midlands Section—Trent Valley Branch. Annual General Meeting.

Newcastle Section. Annual General Meeting.

Friday 5 April

Irish Section. " Wallpaper Manufacture," by Mr. S. K. Weir and Mr. M. Burke preceded by the Annual General Meeting at 7.30 p.m.

Monday 8 April

London Section—Southern Branch. Annual General Meeting.

Tuesday 9 April

Thames Valley Section. Annual General Meeting.

West Riding Section. Annual General Meeting.

Thursday 18 April

London Section. Annual General Meeting at the Criterion-in-Piccadilly, London, W.1, at 6.30 p.m.

Friday 19 April

Manchester Section. Annual General Meeting at the Lancashire County Cricket Club.

Wednesday 24 April

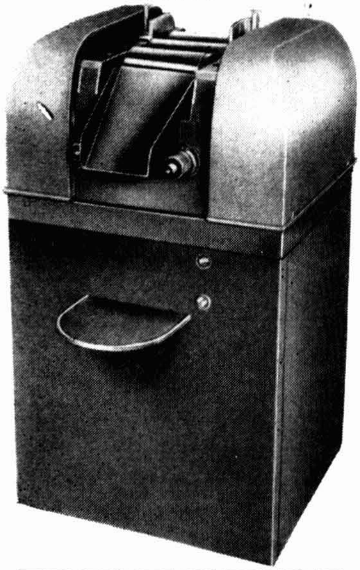
Scottish Section—Eastern Branch. Joint Lecture with Society of Dyers and Colourists. Subject to be decided. To be held at the ICI Recreation Hall, Grangemouth.

Friday 26 April

Bristol Section. Annual General Meeting.

Midlands Section. Annual General Meeting to be held at the Winston Restaurant, Balsall Heath Road, at 7.00 p.m.

SUPERFINE DISPERSION WITH



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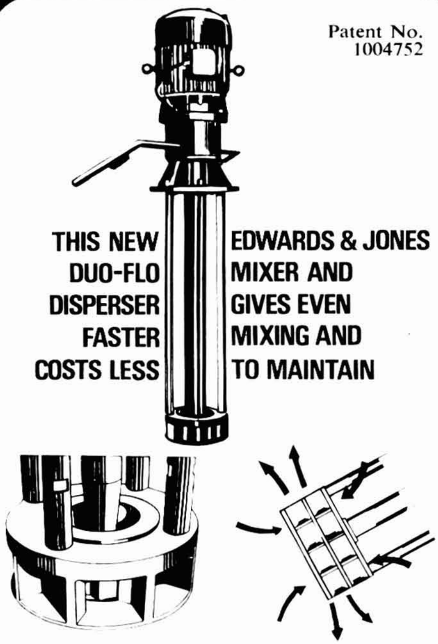
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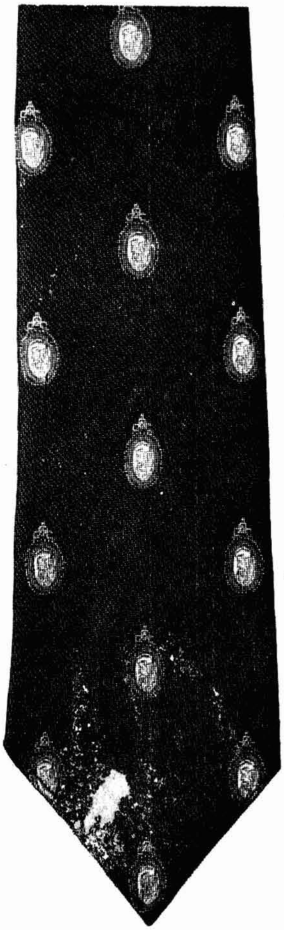

The tie is blue terylene, with the Association's insignia woven in red and gold silk.

The blazer badge has the insignia embroidered in silver and gold wire on a red and blue ground.

The plaque has the insignia hand-painted in red and gold on a blue ground.

The car badge has the insignia embossed in red and gold on a blue ground, the whole being covered in transparent polypropylene, with a thick chrome surround. Bar and grille fittings are available.

In order to commemorate the Fiftieth Anniversary of OCCA, Council has authorised the production of a tie, blazer badge, wall plaque and car badge bearing the Association's insignia. These items are available only from :—

THRESHER & GLENNY

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

To : Thresher & Glenny Ltd., Lancaster Place, Strand, London, W.C.2.
As a member of the Association, please accept my order for the following:

......OCCA tie @ 20/- ....
......OCCA blazer badge @ 63/- ....
......OCCA wall plaque @ 33/6 ....
......OCCA car badge @ 36/6 ....
(state bar or grille)

(All prices include postage and packing)

If tie is required airmail add 6s. 6d.

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Section/No. ....

Address ....

....

....

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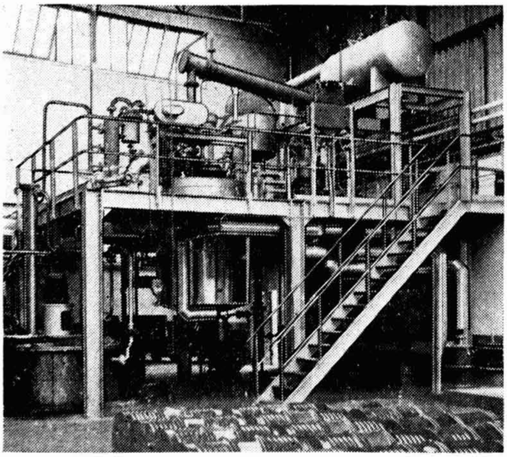
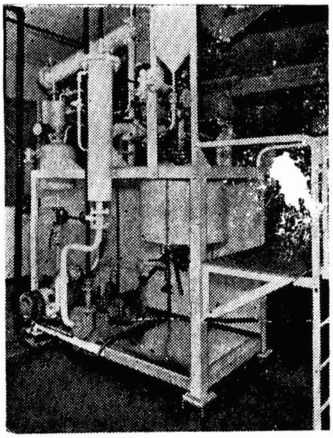
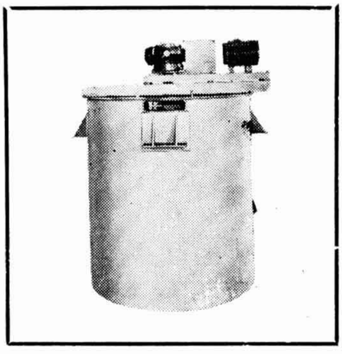
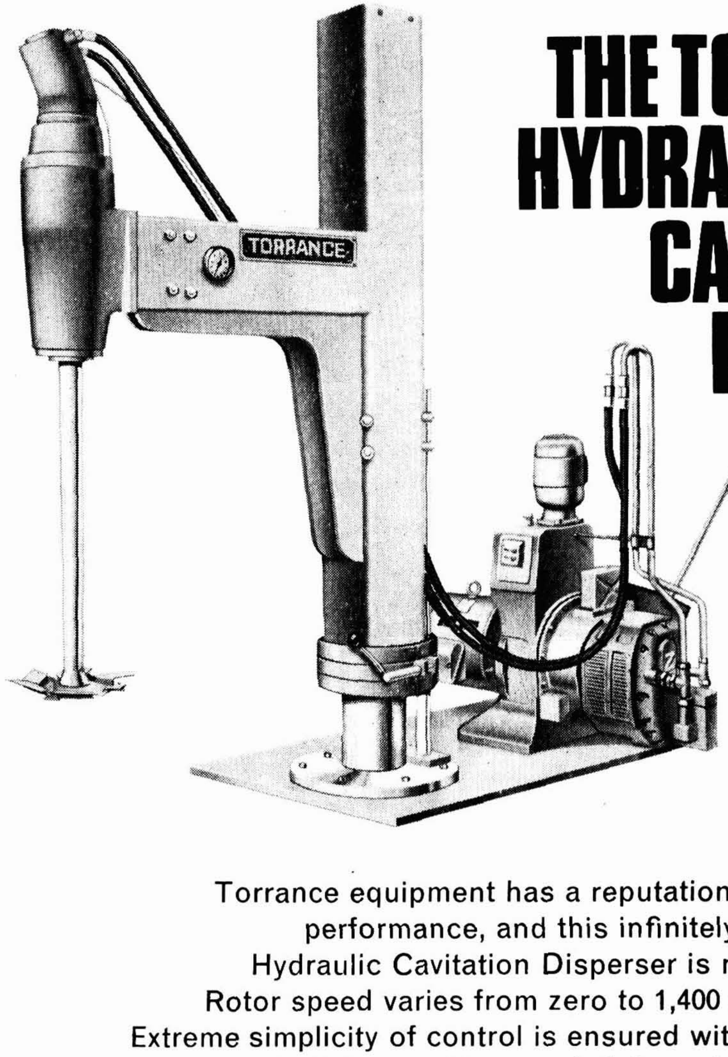
The ties will be supplied from stock; all other items are made to order. Only the tie can be supplied by airmail at an extra cost of 6s.









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CLASSIFIED ADVERTISEMENTS

Classified Advertisements are charged at the rate of 9s. per line. **Advertisements for Situations Wanted are charged at 2s. 6d. per line. A box number is charged at 1s. 0d.** They should be sent to the General Secretary, Oil & Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London, E.C.2. Telephone: **Monarch 1439**

SITUATIONS VACANT

PSF

Pressed Steel Fisher is a subsidiary of British Motor Holdings and is one of the largest Body Building organisations in Europe. Its Research & Development Dept. at Cowley, Oxford, has been instrumental in establishing the Electrocoat process for automobile bodies and components. We now need a Paint Technologist to head the team to evaluate new materials and develop other new techniques.

PAINT TECHNOLOGIST

- The attitude and past performance of the individual is as important as formal qualifications. The successful applicant could be a graduate with two or three years' relevant experience or a National Certificate man with a wide practical knowledge of paint.
- Cowley is situated in pleasant Oxfordshire countryside. Salary will match qualifications and assistance with house removals will be offered.

Applications To:

Senior Staff Recruitment Officer, PRESSED STEEL FISHER, Cowley, Oxford.

RESEARCH/DEVELOPMENT CHEMIST

THE SERICOL GROUP, a medium sized, very progressive and rapidly expanding company, wish to appoint a qualified chemist to lead a new research and development section in London.

The work will be varied and include customer contact, but will be concerned particularly with the application of the new polymers in screen printing inks. Experience in film formers will therefore be valuable.

This is regarded as a senior appointment and the successful candidate will have a very adequate salary and fringe benefits.

Write in complete confidence to :

The Technical Director (Colours),
24 Parsons Green Lane, London, S.W.6.

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Sales Manager

Experienced and able Sales Manager required by well-known company to be responsible for the sale of pigments and chemicals through existing sales team to the paint, printing ink and pigment industries. Applicants should have a sound knowledge of these industries combined with an appropriate educational background and qualifications. The appointment is based in the Manchester area and offers a good salary and other fringe benefits. A company car will be provided. Please apply in confidence to Box No. 286.

POLYMER CHEMISTS

with a good honours degree or equivalent are invited to apply for a position directly responsible to the Director of the BLMRA.

The successful candidate will be in charge of an R and D Section dealing with the finishing of all types of leather. Experience in surface coatings is essential ; some knowledge of surface active chemistry advantageous.

Initially the post will be at Egham but most probably the laboratories will be moved to Leeds in 1971/2.

The salary will be negotiable around £2,500 depending on age and qualifications.

Applications to the Director, British Leather Manufacturers Research Association, Egham, Surrey.

SITUATION WANTED

Young man, experienced paint chemist. Italian Nationality. Fluent English and French. Graduated in Chemistry—is seeking a situation. Now working in Milan for an American Company. Five years' formulating experience in the automotive and industrial fields.

Also interested in Raw Material Technical Service Field for Paint Industry.

Please address to :

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Dear Sir,

Thank you for your letter of yesterday's date and in answer to your first query I would recommend the purchase of ''Introduction to Paint Technology.'' I consider that it is an excellent book for introducing a person into the industry and at 15s. I feel that it is outstanding value. It can be obtained from the Oil & Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London E.C.2.

Now with regard to your second question . . .

COURSES AVAILABLE

THE UNIVERSITY OF LEEDS

DEPARTMENT OF COLOUR CHEMISTRY AND DYEING

M.Sc. COURSE IN COLOUR CHEMISTRY

Applications are invited for entry to a twelve month post-graduate course commencing October, 1968. The course, which will lead to the degree of M.Sc. by examination, will provide instruction in the branches of chemistry of particular concern to those whose interests lie in the field of colour. The course will consist partly of lectures on the chemistry of colour matters, the physical chemistry of absorption, photochemistry, colour physics and the chemistry of natural and synthetic polymers. The remainder of the course will be concerned with practical instruction and a research project. Applicants should normally possess a good Honours degree (or its equivalent) in chemistry or a related subject.

The course is accepted as suitable for the tenure of S.R.C. Advanced Course Studentships. Firms wishing to second members of their staff may reserve places for candidates to be named at a later date.

Applications and enquiries should be addressed to Professor I. D. Rattee, Department of Colour Chemistry and Dyeing, The University, Leeds, 2.

FIFTIETH ANNIVERSARY CELEBRATIONS DINNER

10 May 1968 at the
Savoy Hotel, London, W.C.2

Inner London Education Authority

LONDON COLLEGE OF PRINTING

SCIENCE DEPARTMENT

SENIOR LECTURER IN PHYSICAL CHEMISTRY

The London College of Printing is developing higher level courses which require more advanced teaching in basic science, and in science as related to printing technology. The courses running at present in the Science Department include the HND in Printing and an Advanced Course in Printing Technology for graduates. Applications have been made to run a CNAA B.Sc. degree in Printing, and courses in Packaging at HND level.

The Senior Lecturer is required to be responsible for the development of teaching in the field of Physical Chemistry. He should be a graduate, preferably with teaching and research experience and some knowledge of either the Printing, Packaging, Papermaking, Printing Ink manufacture or other allied industry.

Salary scale (under review) £2,140-£2,380, subject to addition of London Allowance £70. Assistance may be given towards household removal expenses.

Application forms (returnable by 29 March 1968) and further particulars obtainable from the Clerk to the Governors, London College of Printing, Elephant and Castle, London, S.E.1.

"..........and finally we filled the vacancy through a classified advertisement in J.O.C.C.A."

The *Journal* is read by technical personnel throughout the surface-coatings industries, and Situations Vacant may be advertised at the rates of 9s. per line, and 2s. 6d. per line for Situations Wanted.

Printed in England by Metchim & Son Ltd., 8 Storey's Gate, Westminster, London, S.W.1.