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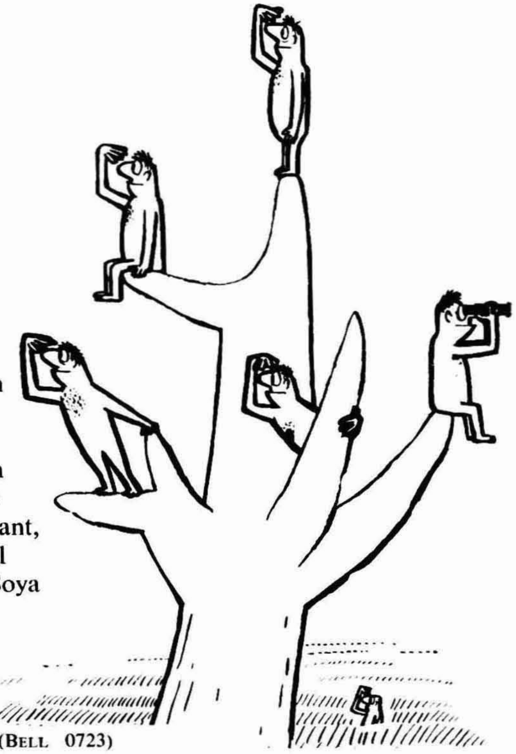

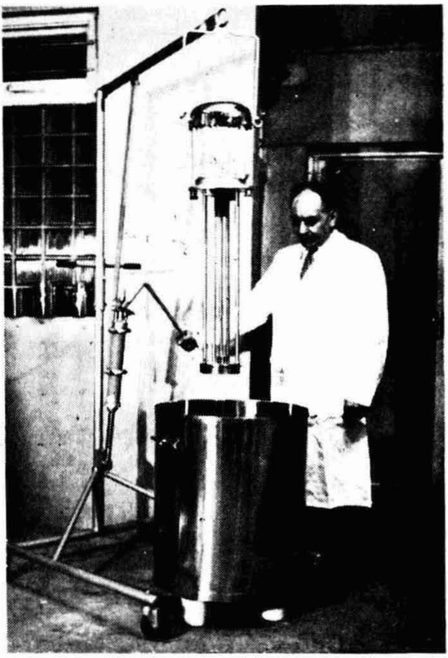


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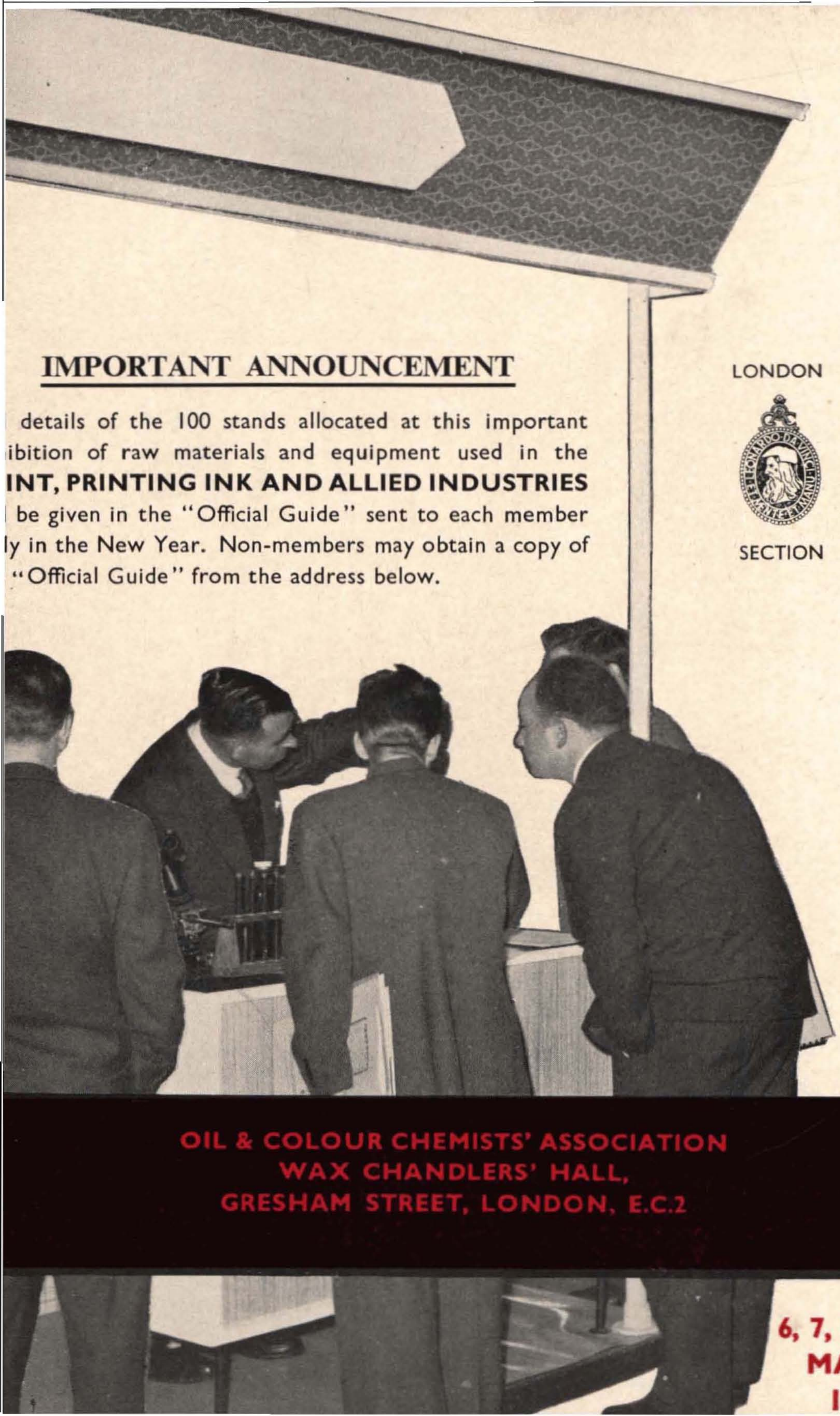

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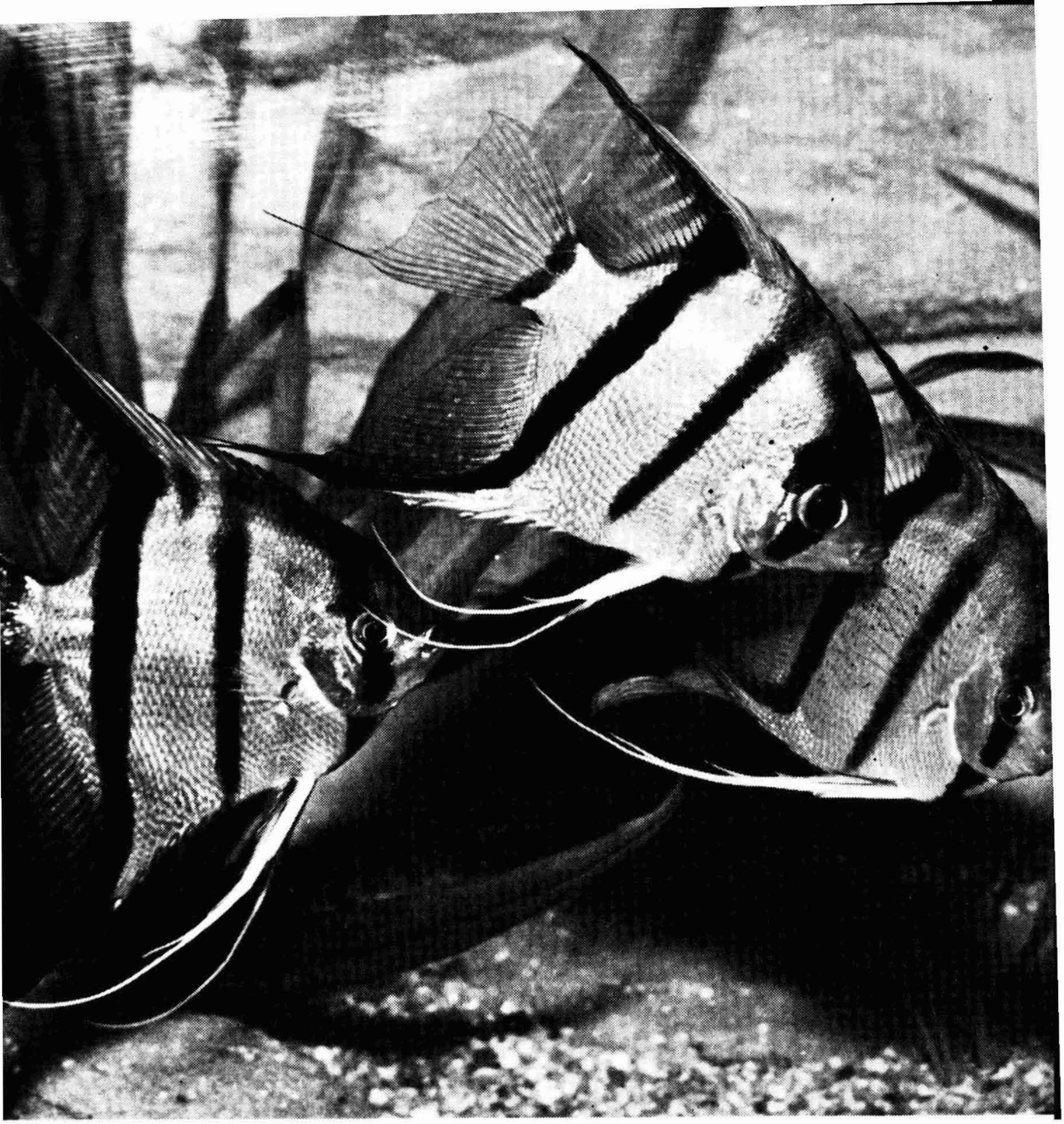

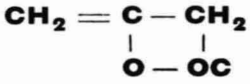

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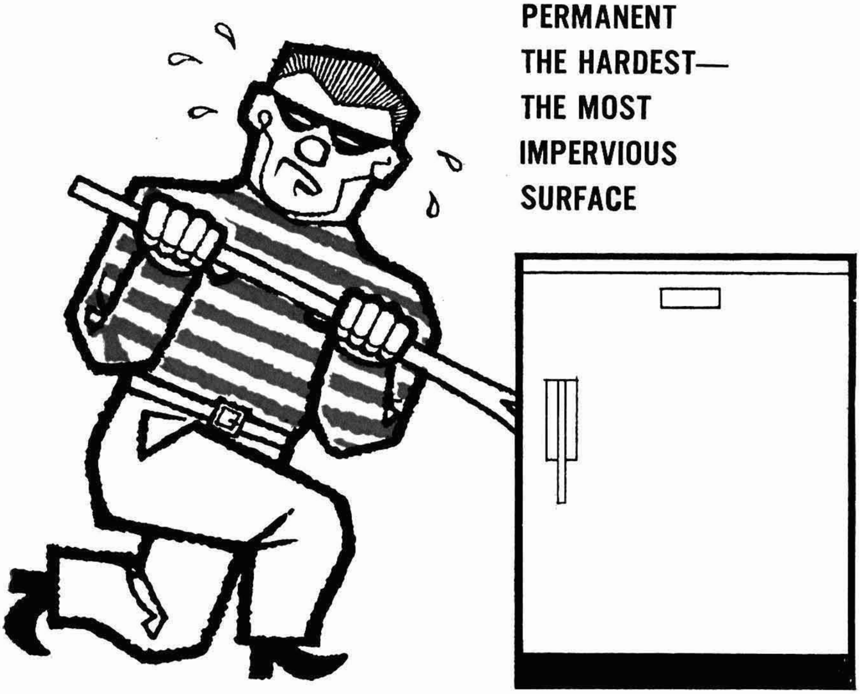

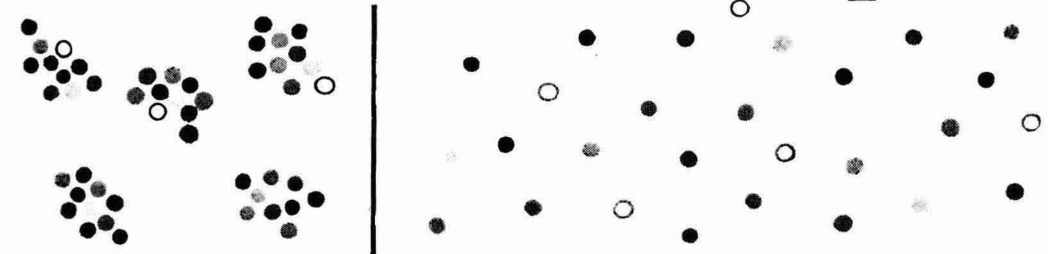

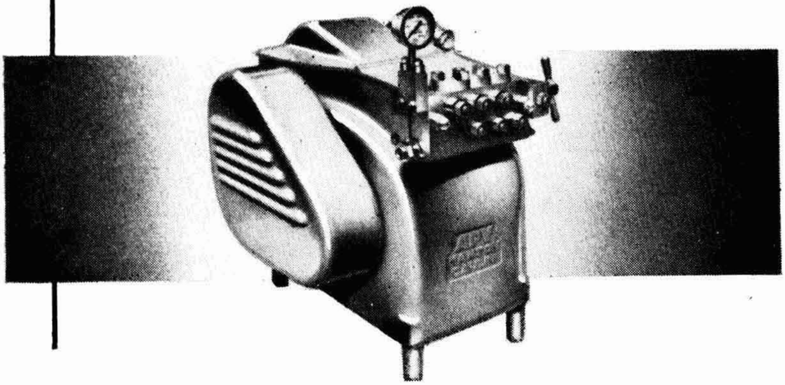

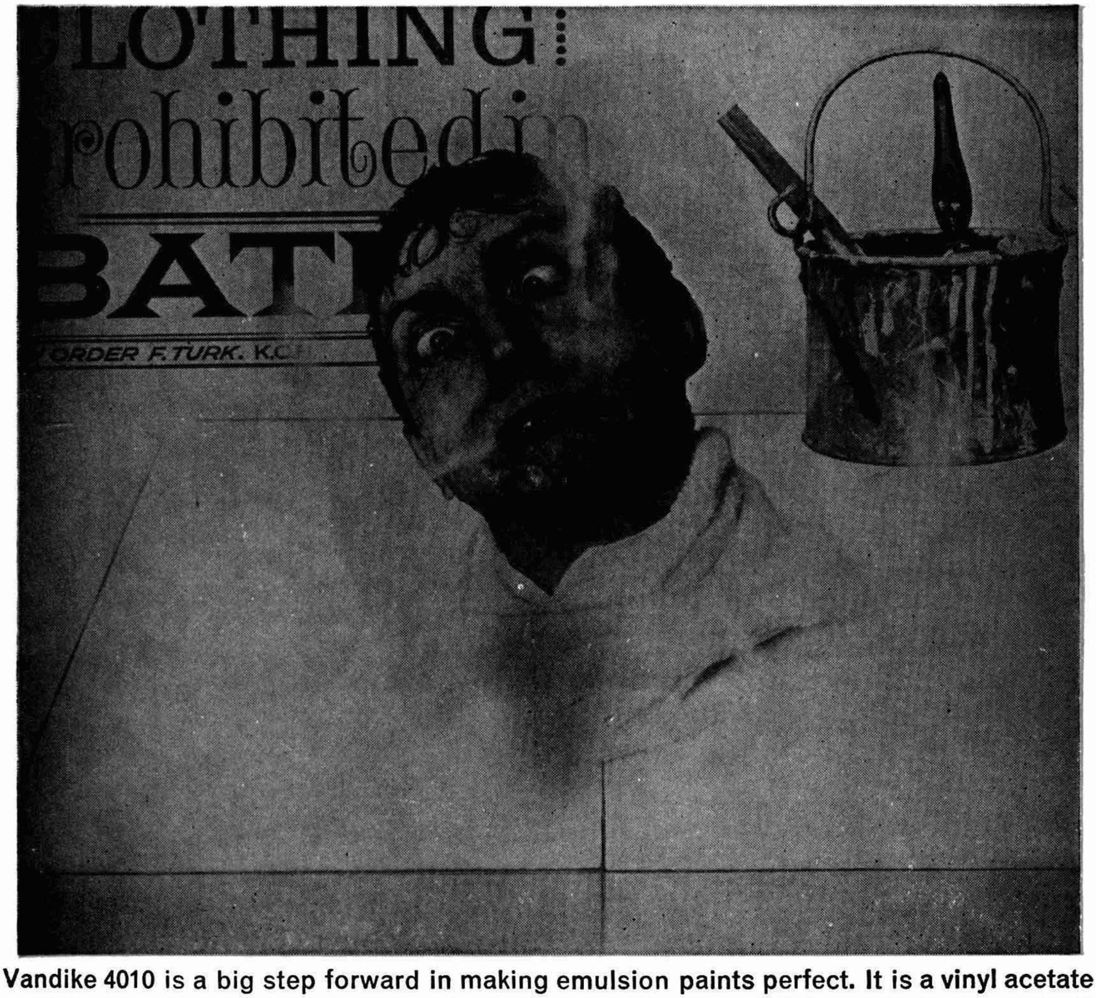
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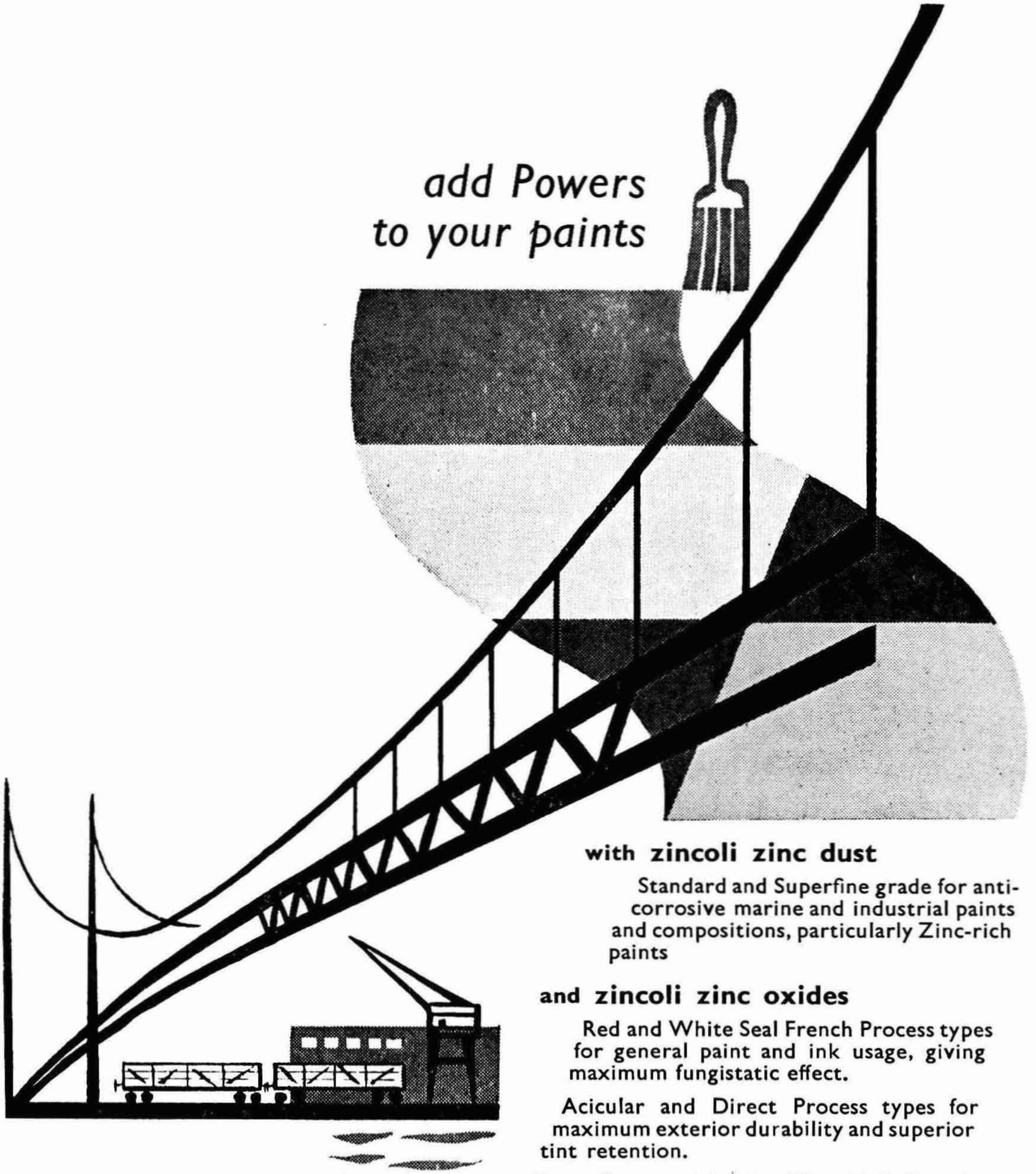
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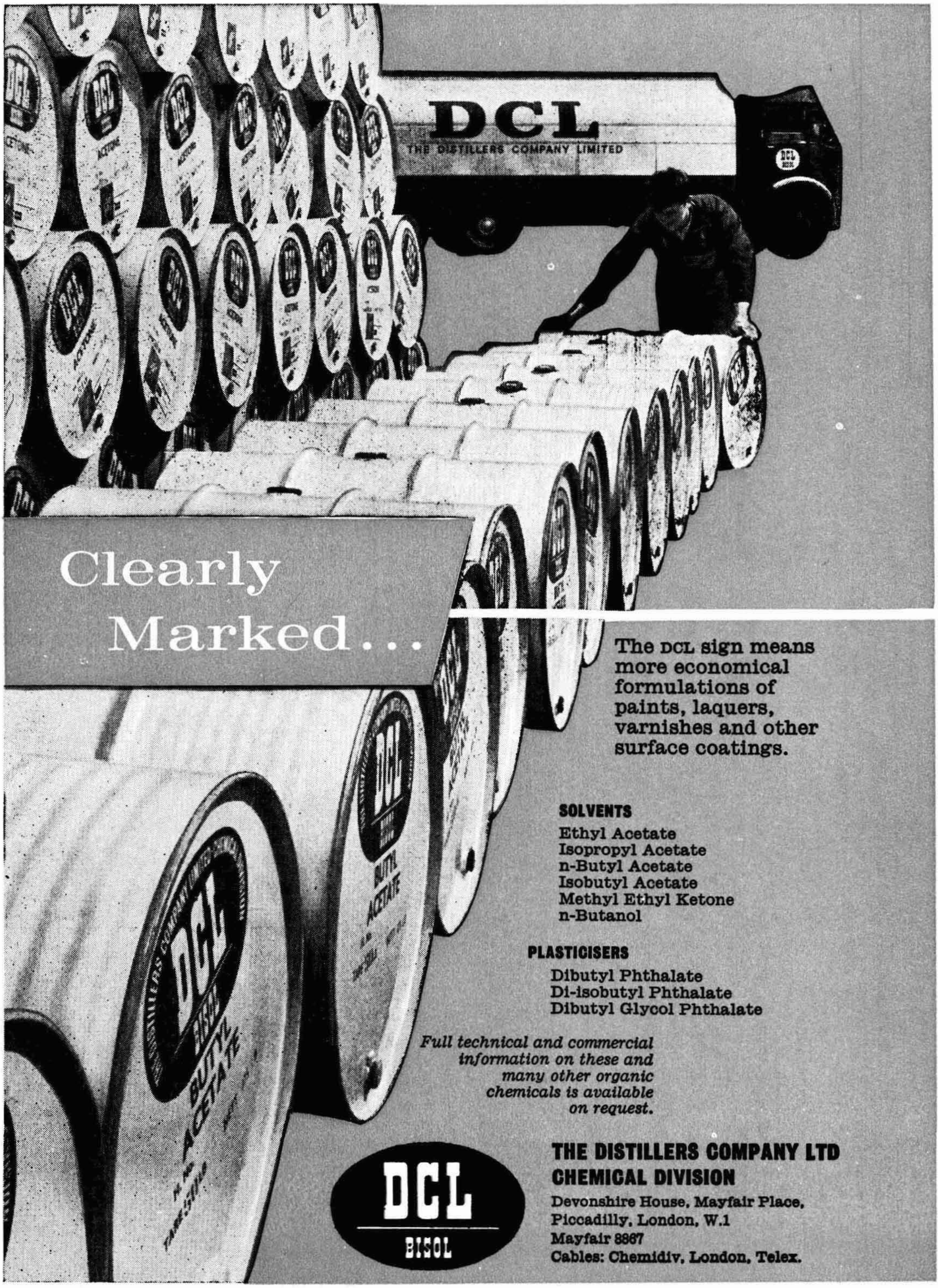
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TRANSACTIONS AND COMMUNICATIONS

Gypsum Plaster as a Substrate for Paint Films*

By M. J. Ridge

Division of Building Research, C.S.I.R.O., Melbourne, Australia

Summary

An account is given of the production of gypsum plaster and its fabrication into fibrous plaster sheets. Special reference is made to the steps in these processes that influence the structure of the final product. The phenomena and kinetics of setting, which is essentially a process of recrystallisation, are also described. Pastes of plaster and water may be regarded as seeded supersaturated solutions, and the influence of density and type of seeding nuclei on the rate of setting is discussed. Particular attention is paid to factors that have strong effects on the microstructure developed on setting.

INTRODUCTION

The purpose of this paper is to give an account of the nature and structure of gypsum plaster, which is an important substrate for paint films. In order to gain an insight into the nature of a gypsum plaster surface it is necessary to go back in its biography and trace the processes that have resulted in the formation of the solid material that confronts the painter. In making this study it must be borne in mind that the production of plaster by the calcination of gypsum (calcium sulphate dihydrate) and the setting reaction that occurs when the calcined gypsum (calcium sulphate hemihydrate) is mixed with water, involves the reactions of solids. The reactions of solids are often dominated by trace constituents of the system and by structural imperfections. The latter factor is particularly important in connection with the dynamics of such processes as crystal growth, which is the basis of the setting of plaster.

Before discussing the technology and physical chemistry of gypsum plaster, it is worth considering the question of structural imperfections which was raised in the last paragraph. The ideal crystal of classical chemistry is a three-dimensional array of atoms, ions, or other groups, in which the unit pattern is repeated with unerring regularity. Real crystals are not like this. The simplest defect that can occur in a crystal is that a lattice site remains unoccupied, and a consideration of the statistical mechanics of the matter reveals that for each temperature there is an equilibrium concentration of lattice vacancies. Vacancies are point defects.

Very important defects known as dislocations also occur in real crystals. These are extended defects and can span the entire width of the crystal. Dislocations can be resolved into two basic types. The structure of one of these types, an edge dislocation, is shown in Fig. 1. Examination of the figure

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*Read at the Second Australian Convention, Oil and Colour Chemists' Association, Warburton, Victoria, June, 1960.

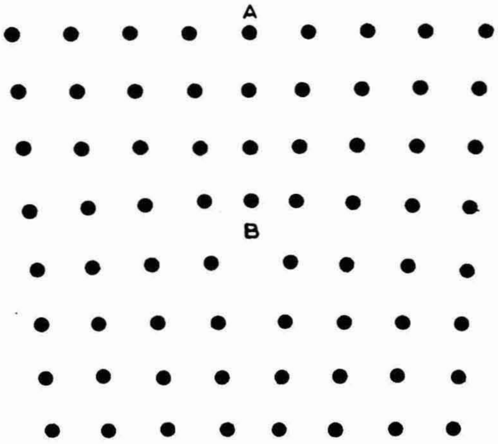
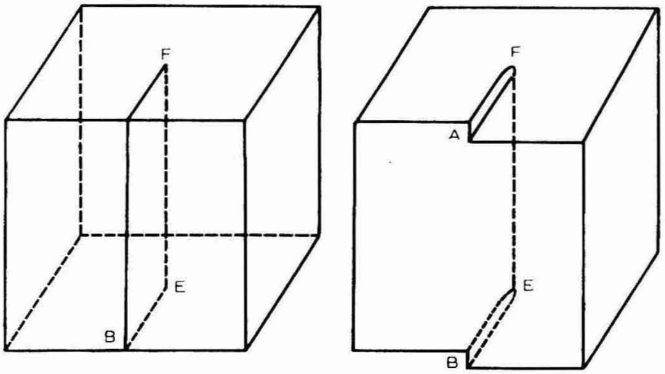


FIG. 1. STRUCTURE OF AN EDGE DISLOCATION

shows that the lattice contains an incomplete sheet of atoms AB. At a few lattice spacings away from B the lattice is nearly perfect, but in the immediate vicinity of B there is a region of distortion which extends into the lattice. The other type shown in Fig. 2 is a screw dislocation, the structure of which can be visualised as follows. A cut ABEF is made in the crystal (Fig. 2a) and the matter of the crystal is subjected to a shear to give a displacement of one lattice spacing (Fig. 2b). Once again at a few lattice spacings away from the line FE the lattice is nearly perfect, whereas there is a region of distortion in the immediate vicinity of FE.

The bearing of dislocations on crystal growth, which is the basis of the hardening of plaster, is apparent when the following facts are considered. It is probable that crystals grow by the spreading of layers across their surfaces; such a layer is shown in Fig. 3. When the edge of the crystal has been reached, a new layer, *i.e.* a two-dimensional nucleus, must form, and this is the bottleneck in the process of growth. The probability of a two-dimensional nucleus growing further increases with the size of the nucleus. When it is considered that the formation of a nucleus of quite a number of lattice spacings is usually necessary for the probability of further growth to be appreciable, it becomes clear that the rate of nucleation as a result of the chance aggregation of a sufficient number of right groups in the correct positions is likely to be low. In the model of the screw dislocation (Fig. 2b), the crystal has on its surface a step that is anchored at F. If this crystal were placed in a supersaturated solution the step would advance and wind itself into a spiral staircase of lattice that could grow indefinitely without further necleation. Consequently, the growth of a crystal containing screw dislocations would be expected to be very much more rapid than that of a perfect crystal. Furthermore, this is not unsupported speculation, as spiral features on the surface of crystals have been found and an example is shown in Fig. 4. The height of the steps in growth spirals is, of course, likely to be small, and special techniques are often needed to reveal these features.



2(*a*) 2(*b*)

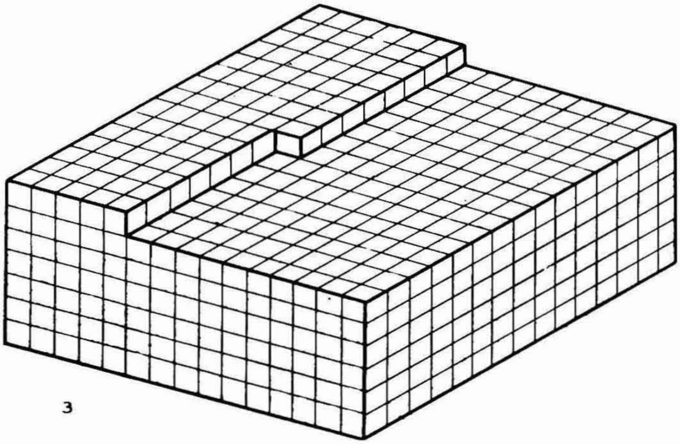


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FIG. 2. FORMATION OF A SCREW DISLOCATION (*After* Verma[1])

FIG. 3. CRYSTAL SURFACE WITH AN INCOMPLETE LAYER (*After* Verma[1])

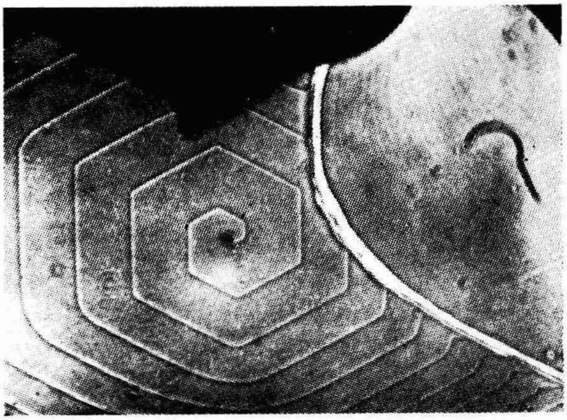


FIG. 4. A REGULAR HEXAGONAL SPIRAL ON A SiC CRYSTAL (*After* Verma[1])

Figs. 2, 3 and 4 on this page, are reproduced from *Crystal Growth and Dislocations*, by A. R. Verma, by kind permission of the publishers, Butterworth's Publications Ltd.

PRODUCTION OF GYPSUM PLASTER

The raw material for the plaster industry is gypsum, which in Australia is of recent geological origin. In North America and Europe the gypsum deposits upon which the plaster industry is based are geologically older, and may have been metamorphosed. After gathering, the gypsum is washed to remove harmful materials, crushed if necessary and calcined, either by a continuous process in a rotary calciner, or by a batch process in a kettle. In these two processes the material is subjected to considerable, but different, types of agitation and mechanical working. In Australia it is also usual to grind the plaster when the calcination is complete. Agitation and mechanical working during the production are of importance as they are liable to damage the crystals of gypsum and calcium sulphate hemihydrate, and the attendant lattice imperfections will leave some legacy in the properties of the finished product.

TABLE I

COMPOSITIONS OF SOME GYPSUM PLASTERS

Plaster	CaO (%)	SO_3 (%)	SiO_2 and other insolubles (%)	R_2O_3 (%)	MgO (%)	CO_2 (%)	NaCl (%)	Total H_2O (%)
A	36.8	49.4	2.9	0.7	0.8	2.3	0.01	6.6
B	39.3	49.4	0.24	0.3	0.3	3.3	0.08	6.5
C	39.0	51.6	0.20	0.3	<0.1	2.2	0.09	6.5
D	37.5	53.5	2.3	0.6	<0.1	0.0	0.05	6.4
E	38.1	54.5	0.32	0.5	<0.1	0.0	0.08	6.5
F	37.8	54.3	0.36	0.1	<0.1	0.0	0.07	6.5
G*	37.7	54.1	0.46	0.2	<0.1	0.1	0.06	6.2

*Plaster G was calcium sulphate hemihydrate exsiccated (laboratory chemicals) (*after* Ridge and Hill[2]).

The composition of some gypsum plasters is shown in Table I. Plaster G was a reagent grade calcium sulphate hemihydrate and it is notable that plasters E and F, which were commercial gypsum plasters, are of the same order of purity. Plasters B and C contain some calcium carbonate, but this acts only as an inert filler. It does not follow, however, that the properties of a plaster will be impaired if it contains material other than calcium sulphate. For example, plaster A contained more silica than the others, but the adhesion of latex paint to casts of this material was better than usual.

PRODUCTION OF FIBROUS PLASTER

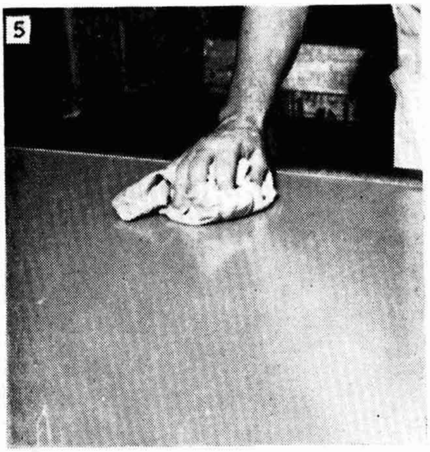
Plaster is mixed with water for fabrication as the material that the painter encounters, and the essential steps in the production of a fibrous plaster wall, which is a commonly encountered building surface in Australia, are worth considering. The first step that will interest the paint manufacturer is the application of a release agent to the casting table (a greased experimental casting table is shown in Fig. 5). It is highly probable that the casting surface will affect the properties of the surface of cast plaster. However, work done in the Division of Building Research has shown that the release agents in common use do not produce an adverse effect upon the surface of fibrous plaster sheet, unless application has been so heavy that a visible film of grease is left on the surface of the cast[3,4].

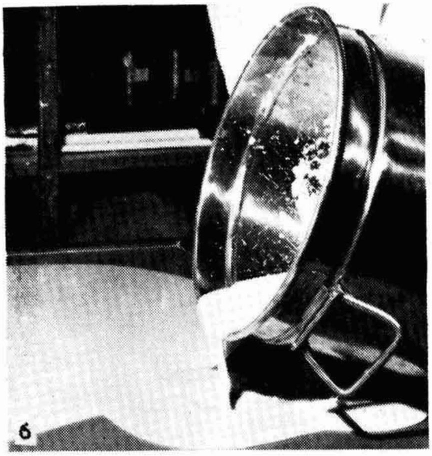
Plaster for the sheet is added to water and the mixture is agitated or stirred to render it smooth. This observation may be obvious, but it deserves mention because in practice the vigour of stirring differs greatly from factory to factory, and as discussed later this can result in marked variations in the structure developed on setting. However, variations in the microstructure do not seem to be accompanied by any marked effect in the adhesion of the paint films.[3] The gauge, the mixture of plaster and water, is shown in Fig. 6 being poured on the casting table, and it can be seen that the mixture is quite free-flowing. The photograph shows the production of a sheet by the one-gauge method, which is the common practice in New South Wales, but two-gauge sheets are made in the other States, particularly in Victoria. In this process a thin layer of plaster and water, *i.e.* a face gauge, is applied to the bench before the main gauge is poured and the sisal is placed. The plaster in the face gauge can be made denser than in the main gauge, so that two-gauge sheets have a harder, less porous surface than one-gauge sheets. The density of the plaster in the face gauge is often further increased by dusting with dry plaster before the main gauge is poured.

In Fig. 7 sisal is being spread over the plaster and Fig. 8 shows the incorporation of sisal into the plaster by running a fluted roller over the table. In this process the plaster is worked to a considerable extent at a time when the setting process is progressing. It has already been indicated that this affects the structure of the finished product.

The final stages in the fabrication of a fibrous plaster sheet consist in turning in the fibre at the edge of the table, and running a straight edge over the table to ensure that the sheet is uniform in thickness. Sometimes dry plaster is sprinkled over the back of the sheet to increase its density and hardness. When hardened, the sheet is lifted and placed in a rack to dry, some manufacturers using artificially heated driers to speed up this process.

Flush jointing is a most important process in the production of a fibrous plaster wall or ceiling, and is carried out as follows. Plaster, thicker in consistency than that used in sheet making, is trowelled into the gap between two adjacent sheets and the joint is allowed to stand, during which time water is absorbed by the sheets from the plaster that has been applied, rendering it denser. The joint is scraped down and more plaster is applied, and is then finished by sprinkling it with water and polishing with a trowel. These operations give a clue to the fundamental differences in nature between the plaster in the joint and that in the adjoining sheets, and they are of interest to those who have to formulate paints suitable for application to this important and popular





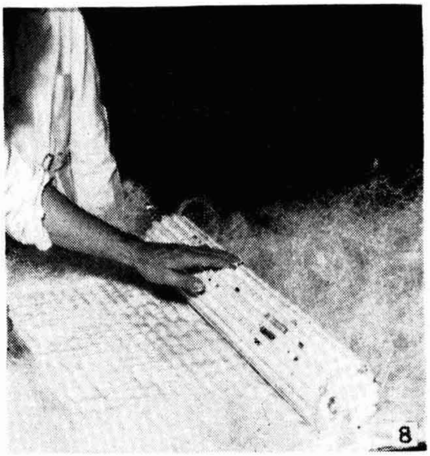


Fig. 5. An Experimental Casting Table for Fibrous Plaster Sheeting

Fig. 6. The Mixture of Plaster and Water Being Poured on the Greased Casting Table (Experimental)

Fig. 7. Sisal Being Spread Over the Plaster

Fig. 8. Sisal Being Incorporated Into the Plaster

substrate. Two of these differences are now apparent, *viz.* (1) the surface of the sheets is a cast surface, whereas the surface of the joint is a worked surface in which the micro crystals of the dihydrate have been seriously damaged ; and (2) the total porosity of the plaster in the joint is much less than in the sheet.

PHYSICAL CHEMISTRY OF GYPSUM PLASTER

Details of the mechanism of the setting process and development of structure may now be considered. The solubility of calcium sulphate dihydrate and of calcium sulphate hemihydrate is shown in Fig. 9; gypsum plaster consists essentially of the hemihydrate. The hemihydrate is more soluble than the dihydrate, suggesting a simple mechanism for setting, *i.e.* that the hemihydrate passes into solution and the dihydrate is precipitated. This is the theory that was advanced many years ago by such illustrious scientists as Lavoisier and Le Chatelier. Such a simple mechanism has been challenged and various colloidal processes have been suggested to explain the initial thickening, but growing crystals of the dihydrate can be found very early in the setting process and the important features of the kinetics of the reaction can be explained on the crystallisation theory. There seems, therefore, to be little need to consider the colloidal theories further.

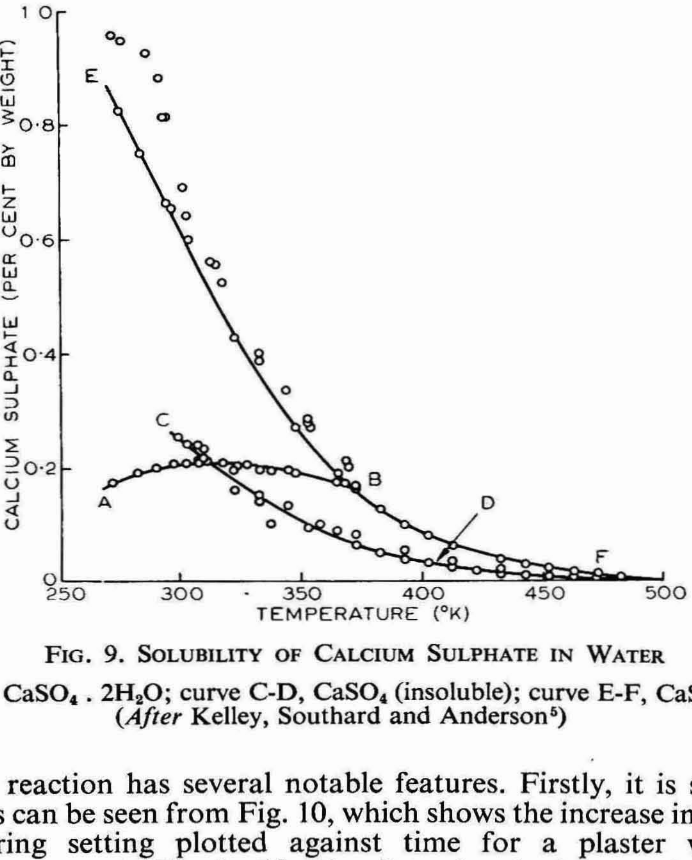


FIG. 9. SOLUBILITY OF CALCIUM SULPHATE IN WATER

Curve A-B, $CaSO_4 . 2H_2O$; curve C-D, $CaSO_4$ (insoluble); curve E-F, $CaSO_4 . \frac{1}{2}H_2O$
(*After* Kelley, Southard and Anderson[5])

The setting reaction has several notable features. Firstly, it is strongly self-accelerating as can be seen from Fig. 10, which shows the increase in temperature occurring during setting plotted against time for a plaster with various additives and treatments. The justification for using the increase in temperature as a parameter of the reaction will be discussed later. Such a reaction may be characterised by the length of the induction period, when suitably defined, and the maximum rate attained. The second point is that the structure developed on setting has usually a fairly even grain size (Fig. 11). This figure should be of interest to paint manufacturers because it shows that the surface of plaster is a type of sieve. The pore size is in the region of 5μ, although it will be shown later that considerable variations occur in the pore size. When considering the consequences of the porous structure, electrostatic effects must be borne in mind.

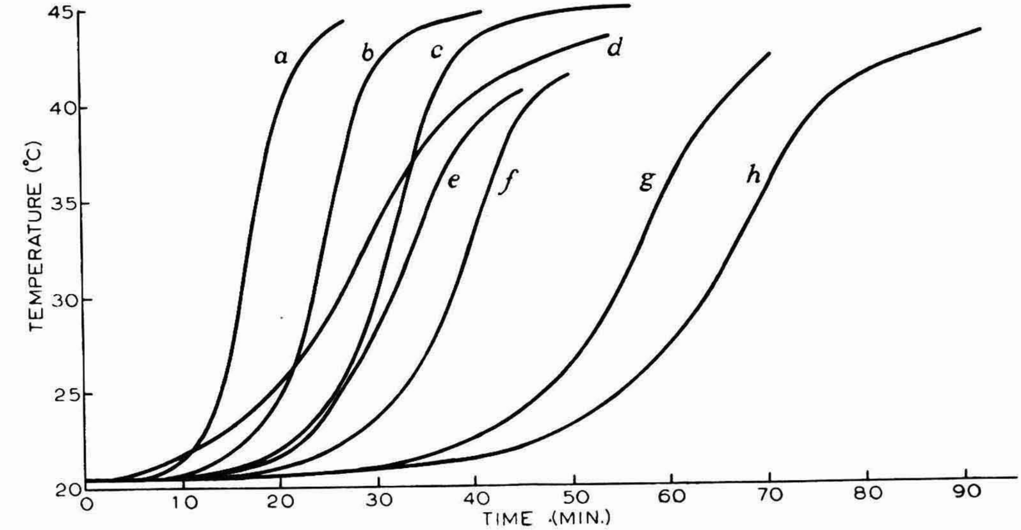


FIG. 10. CURVES OF TEMPERATURE AGAINST TIME FOR MIXTURES OF 60 G. WATER AND 100 G. PLASTER ACCELERATED AS INDICATED IN TABLE II

TABLE II

(*After* Ridge[6])

Curve	Additive	Weight (g.)
a	Sodium sulphate	1.0
b	Sodium chloride	1.0
c	Ammonium thiocyanate	1.0
d	"Killed" plaster	0.5
e	Mixture of plaster and water stirred vigorously for 6 minutes	
f	Dry plaster ground for 30 minutes before mixing with water	
g	Plaster ground 15 minutes before mixing with water	
h	No additive	

If crystals grow they must have a beginning, *i.e.* a stable nucleus must form. The same points that were considered in connection with two-dimensional nucleation on the surface of a crystal also apply to three-dimensional nucleation, that is to say, the formation of an effective nucleus is likely to be the bottle-neck in the crystallisation of calcium sulphate dihydrate. However, the considerations mentioned apply to homogeneous nucleation, in which the nucleus forms as a result of chance fluctuations in a homogeneous medium. A suspension of plaster in water is a heterogeneous system in which the interfacial area is high.

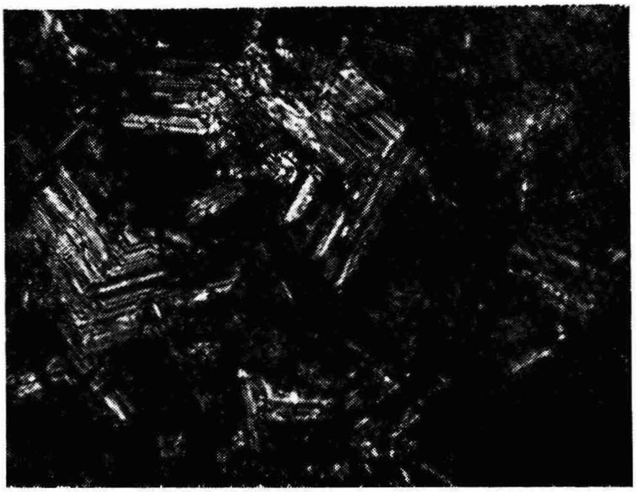


FIG. 11. STRUCTURE DEVELOPED BY 60 G. WATER AND 100 G. PLASTER AT 20.5°C (×400 approx.)

Moreover, the dispersed phase is a solid that has been subjected to grinding and agitation, both of which are liable to produce lattice damage and imperfections. The plaster may also contain residual crystals of gypsum, and such a system is likely to contain effective nuclei and potential nucleus-forming sites. Consequently, a slurry of plaster and water may be regarded as a seeded system, and spontaneous nucleation will not be expected to contribute much to the conversion of hemihydrate to the dihydrate. The uniform grain size of the plaster when set suggests that growth over the entire population of nuclei begins at the same time, presumably shortly after the plaster and water are mixed.

Since the nuclei of calcium sulphate dihydrate grow by the adsorption of calcium and sulphate ions and water molecules on their surface, it is reasonable to assume that

$$\frac{dx}{dt} = k''S$$

where x is the mass of dihydrate, S is the surface area of the nuclei, k″ is a constant and t the time.

As seen from Fig. 11, the growing phase in setting plaster consists of needles, plates and other forms that grow in one or two dimensions only. Consequently, the surface area of the growing phase may be regarded as being proportional to its mass rather than to the 2/3 power of the mass, therefore

$$\frac{dx}{dt} = k'x$$

from which is derived

$$x = Pe^{k't} \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where P is a constant[6].

It is rather convenient that neither the heat nor the rate of the reaction has a large temperature coefficient[7], which means that the reaction can be conveniently studied under conditions in which the loss of heat is minimised, and using ΔT, the increase in temperature, as a measure of the extent of the reaction. The effect of temperature on the rate of the reaction is illustrated in Fig. 12, from which it is seen that from 0°C to about 40°C the maximum rate of increase of temperature rises to a shallow maximum, the induction period is almost constant and the period of initial set falls slightly.

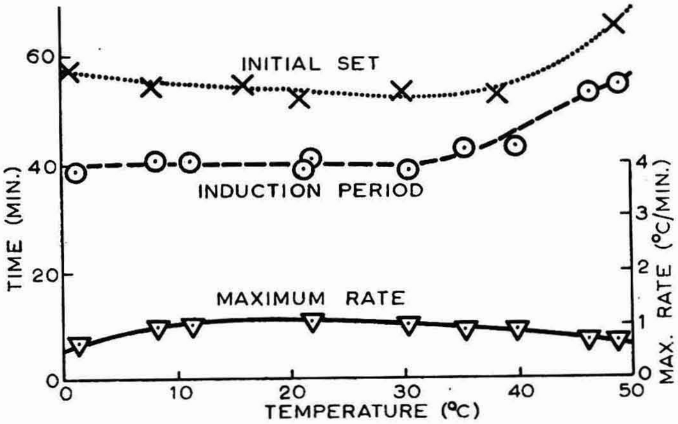
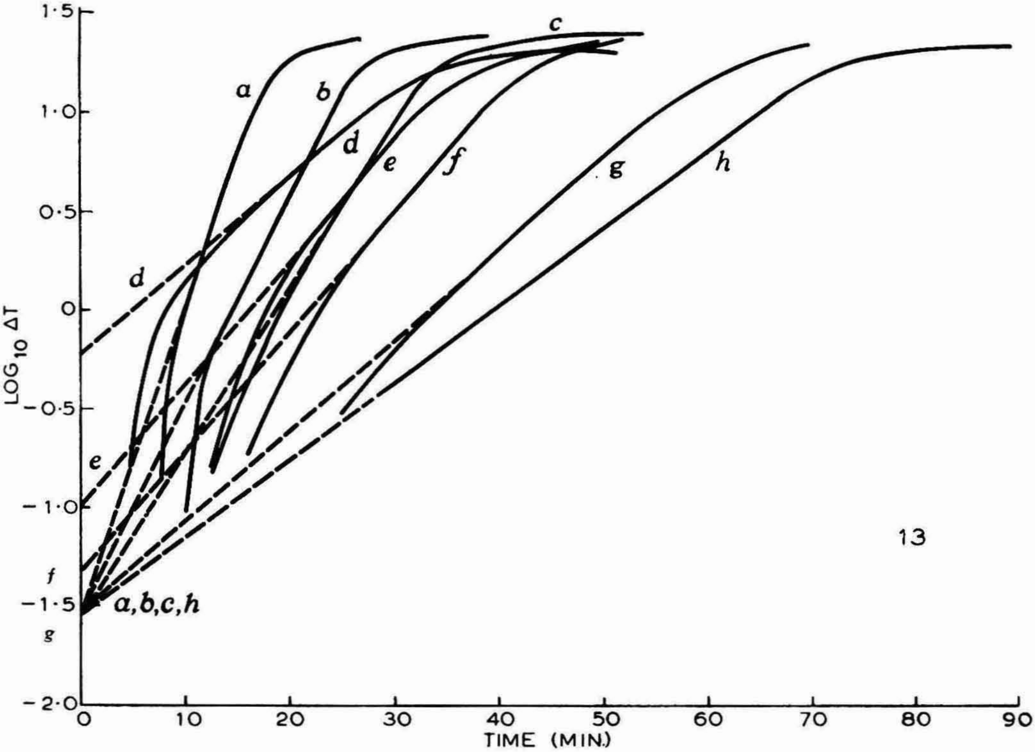


FIG. 12. TIMES FOR THE COMPLETION OF INITIAL SET, INDUCTION PERIOD AND MAXIMUM RATE OF INCREASE OF TEMPERATURE PLOTTED AGAINST INITIAL TEMPERATURE FOR 60 G. WATER AND 100 G. PLASTER
(*After* Ridge[8])

Equation (1) can be tested quite readily. In Fig. 10 ΔT is plotted against time for a sample of plaster accelerated in various ways. If Equation (1) does describe the development of the rate of the reaction, the graph of $\log_{10}\Delta T$ plotted against time should be linear. This is, in fact, plotted in Fig. 13 for the curves shown in Fig. 10, and it is seen to be reasonably linear if it is realised that the approximately straight part represents the time during which 75 per cent of the reactants are converted to products. There are reasons for expecting that the early part of the reaction would not conform with Equation (1)[6] and, as the reaction stops when the reactants are consumed, the curves of $\log_{10}\Delta T$ plotted against time flatten abruptly at a definite value of $\log_{10}\Delta T$.

A plot of ΔT against time is shown in Fig. 14 for a sample of plaster accelerated by the addition of various amounts of "killed" plaster, which consists of small crystals of gypsum. The addition of the small crystals of gypsum accelerates the set, obviously by increasing P in Equation (1). From Equation (1) it would be expected that if $\log_{10}\Delta T$ for these reactions were plotted against time, the approximately straight parts of the curves extrapolated, and the antilog of the intercepts on the ordinate, *i.e.* P, plotted against the mass of "killed" plaster added, a straight line would be obtained. A straight



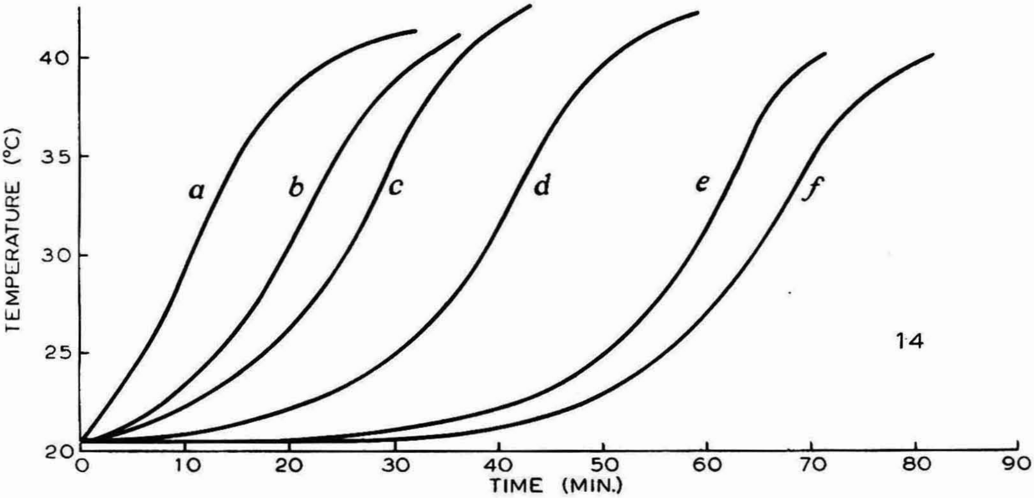


FIG. 13. LOG_{10} ΔT PLOTTED AGAINST TIME FOR THE CURVES SHOWN IN FIG. 10
(*After* Ridge[6])

FIG. 14. CURVES OF TEMPERATURE AGAINST TIME FOR 60 G. WATER AND 100 G. PLASTER ACCELERATED BY VARIOUS ADDITIONS OF KILLED PLASTER
(*a*) 3 g. (*b*) 1 g. (*c*) 0.5 g. (*d*) 0.25 g. (*e*) 0.1 g. (*f*) no additive
(*After* Ridge[6])

line is in fact obtained (Fig. 15), and should, of course, make a positive intercept on the ordinate, but this intercept is comparatively small and the line appears to pass through the origin.

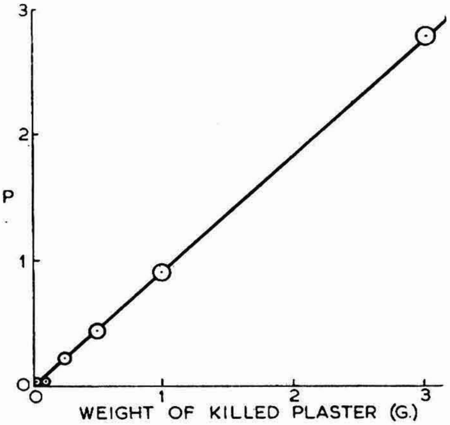


FIG. 15. P IN EQUATION $\Delta T = P_e{}^{k't}$ PLOTTED AGAINST MASS OF "KILLED" PLASTER FOR THE CURVES SHOWN IN FIG. 14
(*After* Ridge[6])

The set of gypsum plaster is accelerated by some neutral salts. Microscopic examination of the accelerated material when set shows that the action is not accompanied by much change in the density of seeding nuclei[6]. If acceleration is not caused by an increase in the density of nuclei, it must be due to an increase in the rate of crystal growth, probably as a result of an enhanced rate of supply to the growing seeds, *i.e.* k' in Equation (1) must be increased. Consequently, $\log_{10}\Delta T$ plotted against time for samples accelerated by salts should yield straight lines, within the limits described above, that converge to a point on the ordinate. In Fig. 13, curves (a), (b) and (c) refer to mixtures accelerated by salts; curve (h) refers to the unaccelerated reaction. The relevant parts of curves (a), (b), (c) and (h) do, in fact, converge with good accuracy to a point on the ordinate.

SOME FEATURES OF THE PHENOMENOLOGY OF GYPSUM PLASTER

Some points of the phenomenology of plaster are of interest to paint manufacturers. As mentioned previously, the rate of setting of gypsum plaster is not very sensitive to temperature, but some interesting variations may be found in the effect of temperature on setting. The phenomena commonly encountered are illustrated in Fig. 12. The induction period, the maximum rate of increase of temperature, the Gillmore initial and final sets are shown plotted against the initial temperature for another plaster in Fig. 16. In this case the induction period and the initial and final sets are somewhat more affected by change in temperature than in the case referred to in Fig. 12. The same parameters plotted against the initial temperature for a plaster with some unusual characteristics is shown in Fig. 17. In this case the induction period and the initial and final sets decrease as the initial temperature falls below about 15°C, *i.e.* the reaction becomes faster as the temperature decreases. These features can be explained theoretically, but such a discussion would be out of place here. These particular phenomena create no practical problems for those who paint plaster and are mentioned only because of their general interest.

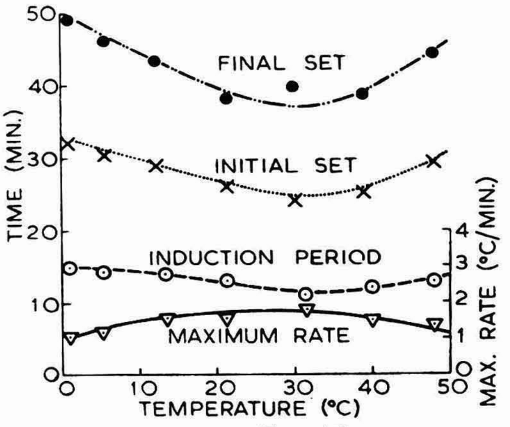


FIG. 16

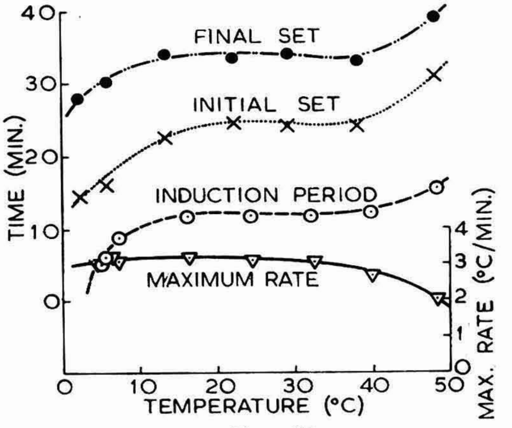


FIG. 17

FIG. 16. TIMES FOR COMPLETION OF INITIAL AND FINAL SET, INDUCTION PERIOD AND MAXIMUM RATE OF INCREASE IN TEMPERATURE PLOTTED AGAINST INITIAL TEMPERATURE FOR 60 G. WATER AND 100 G. PLASTER

FIG. 17. TIMES FOR COMPLETION OF INITIAL AND FINAL SET, INDUCTION PERIOD AND MAXIMUM RATE OF INCREASE IN TEMPERATURE PLOTTED AGAINST INITIAL TEMPERATURE FOR 60 G. WATER AND 100 G. PLASTER (*After* Ridge[7])

Despite the fact that the effect of temperature on the rate of setting is small, the effect of casting temperature on the structure is striking. The microstructures developed by a plaster of the type referred to in Figs. 12 and 16, when cast at various temperatures are shown in Fig. 18. It can be seen that increasing casting temperature is associated with a marked increase in grain size and change in habit[7]. It would be expected that such dramatic changes in microstructure would be reflected in changes in other properties, and work done at the Division

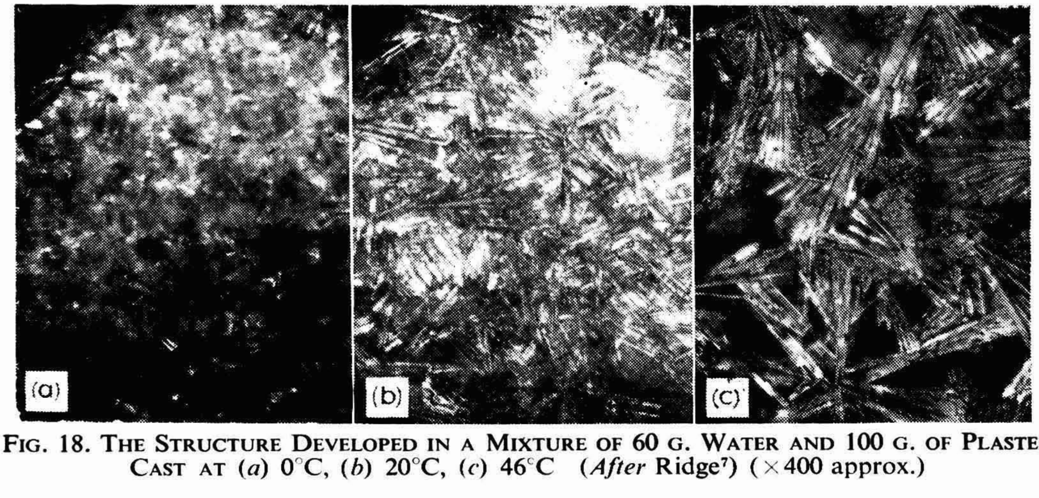


FIG. 18. THE STRUCTURE DEVELOPED IN A MIXTURE OF 60 G. WATER AND 100 G. OF PLASTER CAST AT (*a*) 0°C, (*b*) 20°C, (*c*) 46°C (*After* Ridge[7]) (×400 approx.)

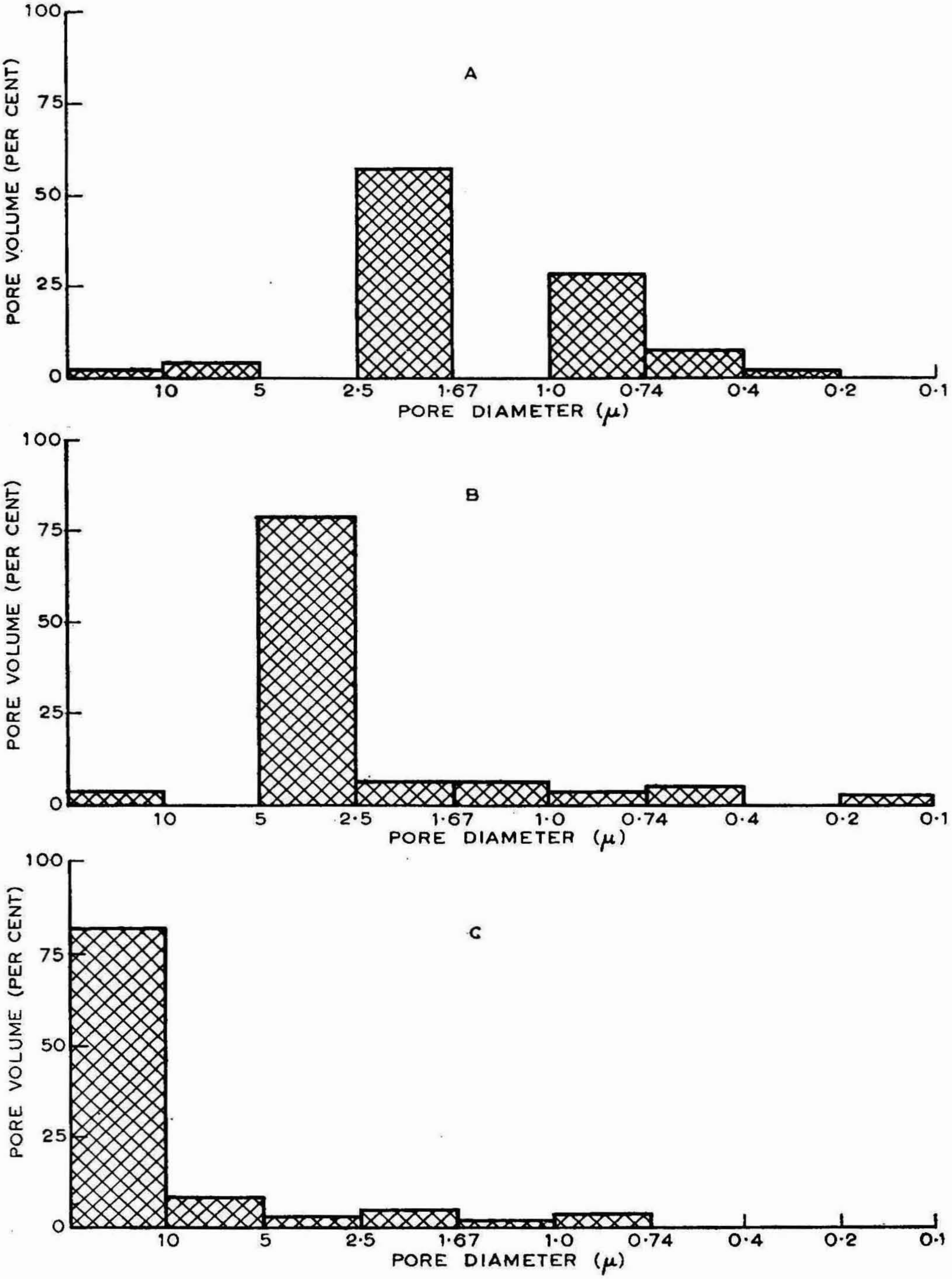


FIG. 19. THE DISTRIBUTION OF PORE SIZE FOR MIXTURES OF 60 G. WATER AND 100 G. OF PLASTER CAST AT (*a*) 0°C, (*b*) 20.5°C, (*c*) 46°C (*After* Ridge and Crook[9])

of Building Research has shown that paint films have an appreciably better adhesion to plaster cast at higher temperatures, although adhesion at lower temperatures is not sufficiently poor to cause trouble. The changes in microstructure are confirmed by measurements of the distribution of pore size, as is shown in Fig. 19. An increase in casting temperature from 0°C to 20.5°C has resulted in a change from a structure in which an appreciable fraction of the pores had a diameter of $>1\mu$ to one in which about 76 per cent of the total pore volume occurred in pores with diameter between 2.5 and 5μ. In a sample cast at 46°C, 75 per cent of the total pore volume occurred in pores greater than 10μ[9].

Another important feature of the setting process is that if the gauge is mechanically worked once thickening has progressed to some extent, the grain size of the product is considerably reduced[10]. This is seen from Fig. 20, which shows the microstructure developed by a sample allowed to set without being further disturbed after the initial mixing, and by a sample that was stirred at the time of initial set. The distributions of pore size in the two cases are of interest to paint chemists and are shown in Fig. 21. From these facts it may be inferred that plaster in flush joints will have a much finer pore size than the adjacent sheets, although it must be realised that the plaster in the sheets has also been subjected to considerable working while setting is in progress, and will be ground finer than the sample represented, for example, by Fig. 20a.

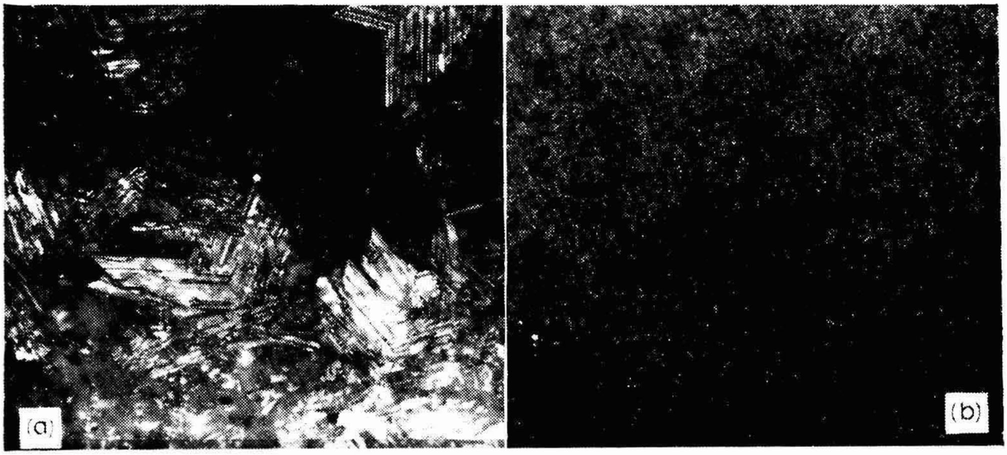


FIG. 20. PHOTOMICROGRAPHS SHOWING THE STRUCTURE OF SET PLASTER
(*a*) Not disturbed after initial mixing
(*b*) Reworked at about the end of the initial set
(*After* Hill and Ridge[10]) (×400 approx.)

At this stage it is appropriate to consider the pore size of surfaces that will be encountered in practice by a painter. The structure of the surface layer of a sheet of fibrous plaster will probably be modified by the release agents used in casting and rendered finer than in the bulk of the sheet. The surfaces of fibrous plaster sheets will of course be rougher than those of specimens made in the laboratory for microscopic examination. The surface of the flush joint will be very fine grained owing to polishing and the modal pore diameter will probably

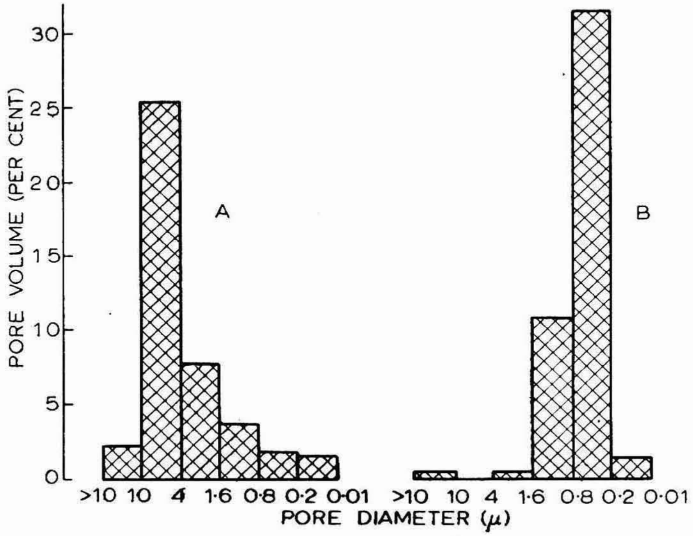


FIG. 21. DISTRIBUTION OF PORE SIZE FOR THE SAMPLES SHOWN IN FIG. 20 (*After* Hill and Ridge[10])

be in the order of 0.1μ or less. The setting coat of solid plaster will resemble the material in the flush joint of fibrous plaster in its texture and will not have very pronounced variations in grain size.

NATURE OF THE NUCLEI IN PLASTER

According to the theory that was discussed earlier, setting plaster may be regarded as a seeded supersaturated solution, and the nature of the nuclei is of importance. This matter is of more interest to plaster millers than to paint chemists, but is still worth considering here.

Plaster is made by the industrial dehydration of gypsum, and it is possible that a little gypsum survives calcination. If this were so it would be expected that heating plaster at 150°C, at which temperature gypsum is rapidly dehydrated, would reduce its reactivity. This is in fact found to be the case, the curves of $\log_{10}\Delta T$ plotted against time for the calcined samples being displaced towards longer time. This reduces the intercept on the ordinate, suggesting destruction of nuclei. This inference is confirmed by microscopic examination, which shows that heat-treated plaster yields coarser grained casts[2]. However, it is found that if the heat-treated plaster is ground, the density of nuclei is increased, which does not accord with the view that the nuclei are residual crystals of gypsum; heating will certainly have destroyed discrete crystals of gypsum. If the recalcined and ground material is again calcined, a product is obtained that is even less reactive than that produced by the first recalcination. It is interesting to note that, although the reactivity of plaster is reduced by recalcination, different plasters respond to very different extents. This is shown in Fig. 22 in which the induction period of hydration is plotted against the period of time, the sample was held at 145°C. The samples settle down fairly rapidly to a value that is characteristic of the sample[2]. The striking variation in susceptibility to the effects of recalcination is also contrary to the idea that the nuclei are residual crystals of gypsum.

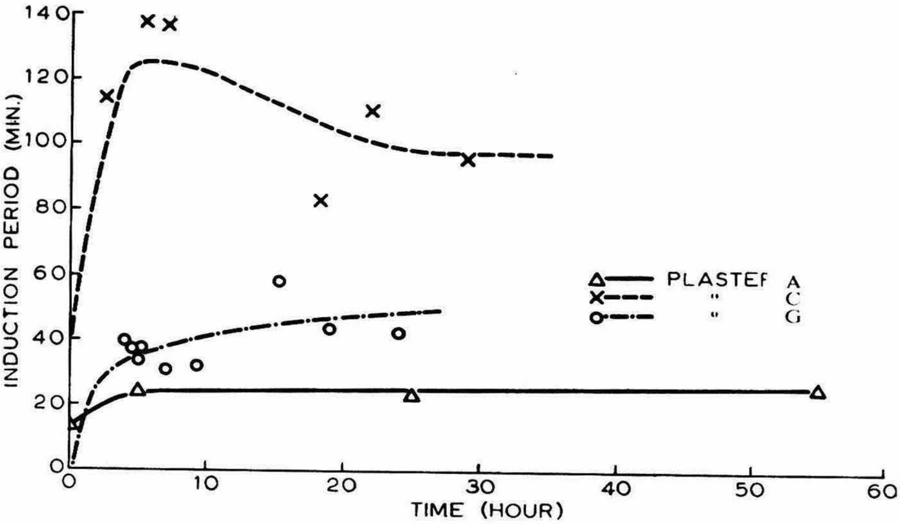


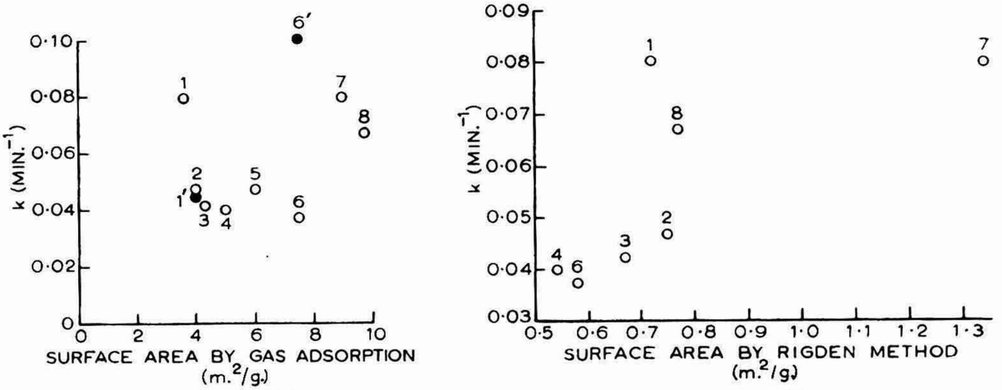
Fig. 22. The Induction Period of Plasters A, C and G (60 g. Water and 100 g. of Plaster) Plotted Against Time of Recalcination at 145°C (*After* Ridge and Hill[2])

The results of a considerable amount of work, too lengthy to discuss here, indicate that the nuclei are sites that are favourable for nucleation and are due to damage and lattice imperfections, including dislocations, in the crystals of the hemihydrate. Judging from the varying reactions of a number of plasters, the population of seeding nuclei contains a range of types of favourable sites[2].

Variations in the Rate of Setting of Plaster

Plaster is a variable material and samples taken from different sources can show much variation in the rate of setting. This is a problem of the plaster industry and is of no practical consequence in the paint industry, but it is of sufficient scientific interest to be discussed in this paper. The theory of setting developed above suggests that variations in the rate of setting will be due to changes in the density of seeding nuclei, or in the rate at which the system can supply the nuclei with fresh material. The latter factor will undoubtedly be affected, to some extent, by the surface area and the particle size of the calcium sulphate hemihydrate. In Fig. 23 the constant k′ in Equation (1) is shown plotted against surface area for a number of samples of commercial plaster. The constant k′ is the parameter that is sensitive to changes in the rate of supply of calcium sulphate to the growing centres. The surface area determined by gas adsorption (Fig. 23a) will include the area of cracks and crevices. There appears to be no appreciable correlation between this surface area and the parameter k. The surface area determined by the permeability of the powder to air (Fig. 23b) is a measure of the external surface area of the grains, and might be expected to be more appropriate in this case than that determined by gas adsorption.

At first sight the correlation in Fig. 23b appears to be better than in Fig. 23a, but if point No. 7 is omitted the correlation is not very striking.



(*a*) (*b*)

Fig. 23. The Constant k in the Equation $\mathrm{Log}_{10}\,\Delta T = \mathrm{Log}_{10}\,P + kT$ Plotted Against Surface Area Obtained by Gas Adsorption, and by the Rigden Method (*After* Ridge and Hill[2])

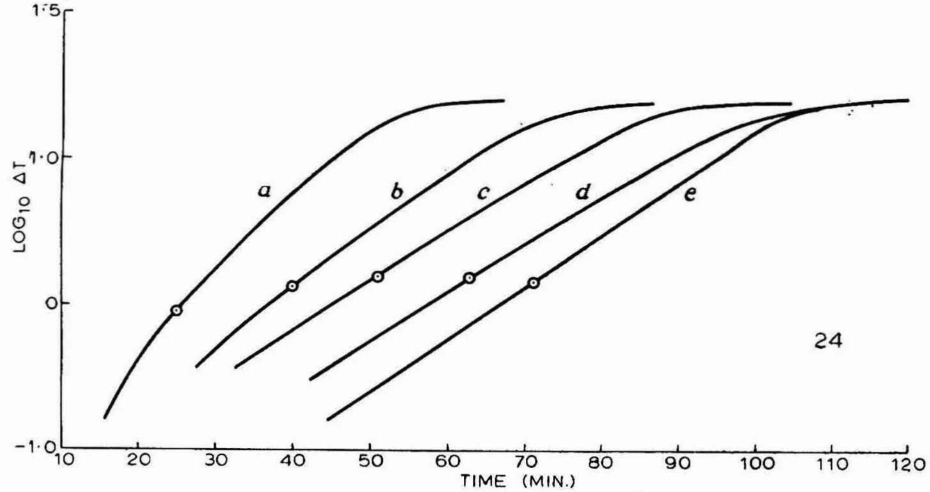
There is little doubt that the surface area of the grains of hemihydrate will affect the rate of hydration. However, the results discussed show that the effect of the surface area is swamped by some other factors, and the conclusion drawn from a large number of experimental results is that these factors are the density and surface area of the population of seeding nuclei.

RETARDATION OF THE SET

The retardation of the set of gypsum plaster is frequently carried out in the plaster industry and is not a matter of indifference to those who apply paint to the plaster. For convenience retarders may be divided into two groups, *viz.* colloidal materials, such as egg albumen, degraded keratin and glue size, and crystalloidal materials, such as sodium citrate and borax.

The effects of some colloidal retarders on the curves of $\log_{10}\Delta T$ against time are shown in Fig. 24. The curves for the retarded reaction retain their original shape and are displaced towards longer time, suggesting that the retarders act merely by poisoning nuclei. Microscopic examination of retarded mixtures when set confirms this view, and correlation of the grain sizes of samples retarded by egg albumen and by recalcination indicates that when retardation is not too pronounced this retarder acts almost entirely by poisoning nuclei.

In Fig. 25 the effect of some crystalloidal retarders on the curve of $\log_{10}\Delta T$ against time is shown, and in this case the effect is more complicated. The curves for calcium borate, calcium citrate and sodium citrate are nowhere good approximations to straight lines, but curve upward. The part of the curves for calcium acetate and sodium hexametaphosphate (c) for the time when most of the reactants are converted to products can be extrapolated to meet that for the blank (a) at the ordinate.



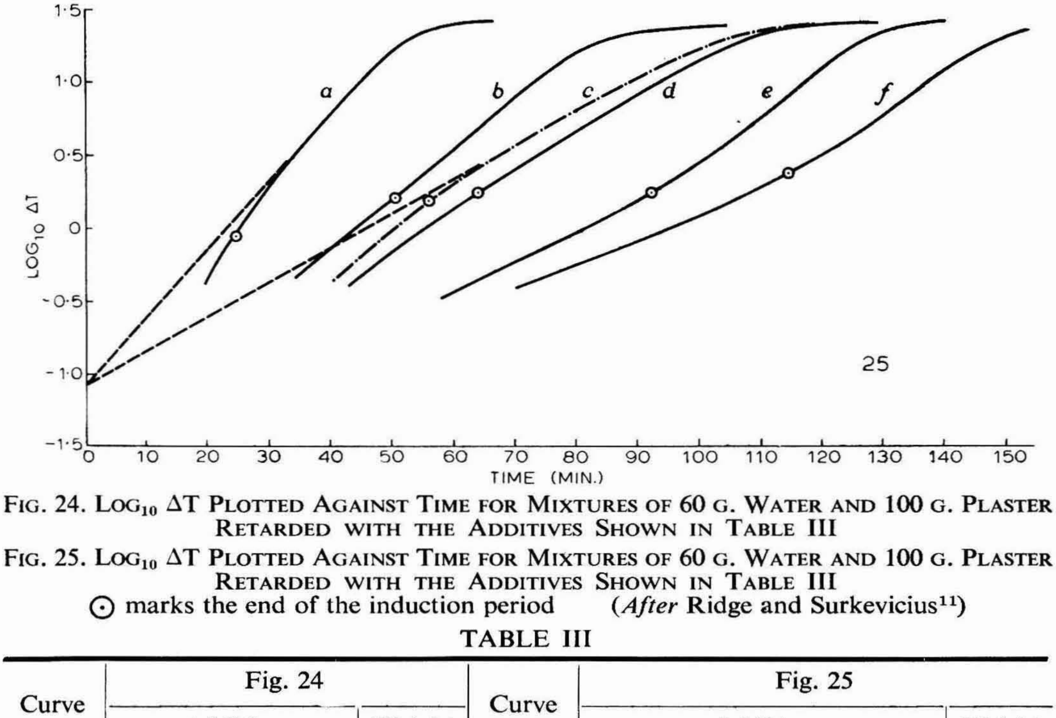


FIG. 24. LOG$_{10}$ ΔT PLOTTED AGAINST TIME FOR MIXTURES OF 60 G. WATER AND 100 G. PLASTER RETARDED WITH THE ADDITIVES SHOWN IN TABLE III

FIG. 25. LOG$_{10}$ ΔT PLOTTED AGAINST TIME FOR MIXTURES OF 60 G. WATER AND 100 G. PLASTER RETARDED WITH THE ADDITIVES SHOWN IN TABLE III

⊙ marks the end of the induction period (*After* Ridge and Surkevicius[11])

TABLE III

Curve	Fig. 24		Curve	Fig. 25	
	Additive	Weight (g.)		Additive	Weight (g.)
a	No additive	—	a	No additive	—
b	Gamma globulin	0.16	b	Calcium borate	0.40
c	Egg albumin	0.20	c	Sodium hexametaphosphate Calcium acetate*	0.10 0.50
d	Gelatin	0.10	d	Calcium salt of ethylene diamine tetra-acetic acid	0.50
e	Degraded keratin	0.10	e	Sodium citrate	0.05
			f	Calcium citrate	0.06

*These curves were coincident.

The microstructure of a number of retarded plasters is shown in Fig. 26: Fig. (b) shows the structure of a sample retarded by egg albumen, and comparison with the structure of the unretarded sample (a) shows that the grain size is coarser but that the habit is not greatly affected. With other retarders effects in the habit are noticeable. The tendency of the retarders, such as calcium citrate, to modify the habit of the crystals of calcium sulphate dihydrate (Fig. 26(c)) provides a clue to the upward curvature of the curves of $\log_{10}\Delta T$ against time for this and some other retarders. Such retarders obviously interfere with the growth and reduce the rate of the reaction at the interface. There is

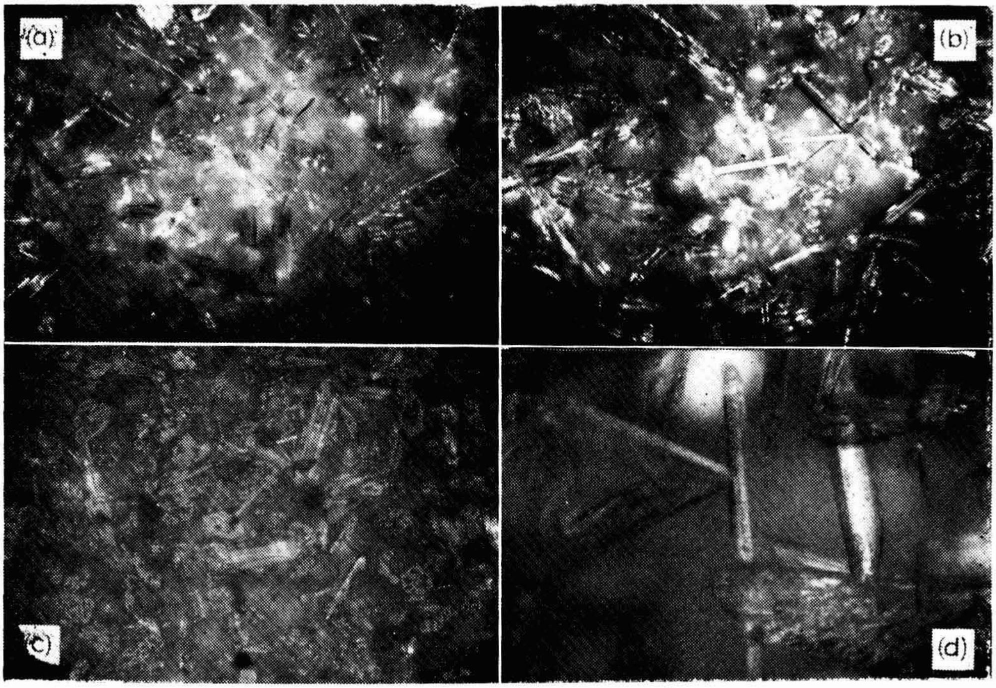


FIG. 26. THE STRUCTURE DEVELOPED BY MIXTURES OF 60 G. WATER AND 100 G. PLASTER RETARDED WITH THE ADDITIVES SHOWN IN TABLE IV

(*After* Ridge and Surkevicius[11])

TABLE IV

	Additive	Weight (g.)	Induction period (min.)
a	No retarder	—	42
b	Egg albumen	0.4	125
c	Calcium citrate	0.1	246
d	Sodium hexametaphosphate	0.1	143

ample evidence that substances which modify the habit of crystals are also incorporated in them during growth. The inference is that some retarders are rendered ineffective during the reaction by being built into the growing dihydrate, so that the reaction becomes less inhibited as it proceeds.

Examination of a number of experimental results leads to the belief that calcium acetate does not appreciably poison nuclei, but acts mainly by inhibiting the rate of growth of the dihydrate. The microscopic evidence for sodium hexametaphosphate (Fig. 26(d)) shows that it is outstandingly effective as a poison for nuclei for the growth of the dihydrate. However, the retardation is not as great as would be expected from the reduction in the density of seeding nuclei, so that this retarder evidently has a complex action.

The fact that some retarders modify the habit of gypsum, and consequently bring about a change in the crystallographic planes that are exposed to the paint, suggests that they may affect the adhesion of paints. However, it has been shown[3] that retarders commonly in use do not adversely affect the adhesion of paints, and in some cases actually improve adhesion.

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Efflorescence Phenomena on Plaster*

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Summary

The types of salt likely to cause efflorescence on gypsum plaster are examined and it is established that sodium sulphate is the salt that causes most trouble. The compounds that could react with the gypsum plaster to give sodium sulphate are discussed, and the manner by which some of the salts could enter the plaster is investigated. Two methods for suppressing efflorescence are suggested and the painting of moist plaster surfaces is considered.

INTRODUCTION

Very few references to efflorescence on gypsum plaster have appeared in literature. Efflorescence on painted plaster and cement was mentioned by Llewellyn[1] and Llewellyn and Eldridge[2], but the salts that could cause the trouble were not identified. More recently Henson and Wise[3] published a paper on this subject, but they considered only the case in which efflorescence arises from the salt content of latex paints. They found that the efflorescence was caused by sodium sulphate, which was present either in the hydrated or anhydrous form according to the atmospheric conditions.

In Australia, gypsum plaster is used very widely as a building material in the form of fibrous plaster, plaster board and set plaster. Efflorescence sometimes occurs on these materials leading to the peeling of any paint film that may be present and consequent expense of redecoration. It became of interest, therefore, to elaborate on those points that previous investigators had not covered, namely, the types of salt that are likely to react with gypsum to form sodium sulphate, which appears to be the salt that causes most damage, and the extent to which other salts cause efflorescence.

There are three types of salt that would be expected to react with gypsum:

(i) Any salt whose anion could give, with calcium, a salt that is less soluble than calcium sulphate, *e.g.* sodium carbonate could react with calcium sulphate to form calcium carbonate and sodium sulphate.

(ii) Sequestering agents like sodium polyphosphate, *Calgon*, and the sodium salts of ethylene diamine tetra-acetic acid, *Sequestrol* or *Perma Clear*. These do not give insoluble compounds with calcium, but form complexes which are very little ionised and liberate the equivalent amount of sodium ions; these are free to form sodium sulphate with the sulphate ions of the calcium sulphate.

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*Portion of a paper read at the Second Australian Convention of the Oil and Colour Chemists' Association at Warburton, Victoria, June, 1960.

(iii) Certain salts whose anions give, with calcium, a salt more soluble than calcium sulphate (see Appendix I).

Plaster may become contaminated with salts in the following ways:

(*a*) If a plaster rendering is in contact with concrete or bricks, salts such as sodium carbonate, sodium sulphate and sodium silicate in these materials could migrate into the plaster during the drying process.

(*b*) Latex paints may contain salts that could diffuse into the plaster.

(*c*) Cleaning compounds for paints may contain materials that could cause efflorescence if they are absorbed by the plaster.

(*d*) The plaster itself could contain salt impurities, the most likely being sodium chloride.

EXPERIMENTAL PROCEDURE

Tests to determine whether various salts would react with gypsum were carried out in the following way. Each salt was added, usually as a 10 per cent aqueous solution, to a cast disc of plaster 2 in. in diameter. In this way, salt concentrations of 1.5 to 3 per cent of the weight of the plaster were produced, *i.e.* from 40 to 80 g./ft.^{2} of surface. The specimens were mounted in such a way that the water could evaporate only through one surface and were stored under conditions of constant temperature and humidity (18°C and 68 per cent R.H.). The surface of each plaster specimen was examined at intervals to observe the appearance of efflorescence. The samples were eventually painted and the adhesion of the paint film determined by the method described in an earlier paper[4].

Salts of the first type that were tested were sodium carbonate, sodium bicarbonate, disodium phosphate, trisodium phosphate, sodium oxalate and sodium silicate. The anions of all these salts give, with calcium, a salt of very low solubility. A copious efflorescence of hydrated sodium sulphate formed within 24 hours, but if the drying was done above 32°C, a temperature that is not uncommon in Australia, the anhydrous salt was formed and it was difficult to see the efflorescence. The importance of the latter fact must be stressed as it means that a substrate could be decorated without the painter being aware that the surface is unsuitable for painting.

The specimens, which at this stage still contained between 30 and 50 per cent of the introduced water, were then painted with a p.v.a. paint or a flat alkyd enamel, and the adhesion was measured when the specimens had fully dried one week later. Although most of the painted samples appeared to be quite normal, the adhesion in all cases was very low, approximately 5 lb./in.^{2}, whereas the adhesion of a p.v.a. or flat alkyd paint to dry plaster is more than 100 lb./in.^{2}

The possible origin of sodium carbonate and sodium silicate has been mentioned previously. Trisodium phosphate is contained in many compounds used for cleaning paint, but as long as these compounds are used to wash an intact paint film there is no danger of the plaster becoming contaminated. However, it is conceivable that contamination could occur if the paint film was not intact, for instance, if the cleaning compound was used to wash off kalsomine. Sequestering agents produced no visible efflorescence, and the adhesion of paint to specimens containing these agents was more than 100 lb./in.^{2}

In the third group of salts, sodium chloride, sodium bromide and sodium iodide were tested. The solubilities in all cases are such that the formation of sodium sulphate would not be expected and this was confirmed by experiment. Plaster containing large amounts of sodium chloride (approximately 3 per cent of the weight of the plaster) showed some efflorescence, and the paint film had a considerably reduced adhesion of the order of 40 lb./in.^{2} With up to 1.5 per cent sodium chloride in the plaster the adhesion was not greatly reduced. It is important to stress that experiments have shown that adhesion troubles are encountered in plaster containing as little as 0.1 per cent sodium sulphate. Sodium bromide and sodium iodide can be added in higher concentrations than sodium chloride without causing any decrease in adhesion or signs of any efflorescence. Apart from the salts mentioned, potassium sulphate, copper sulphate, ammonium chloride, calcium chloride, calcium bromide and ammonium sulphate were examined. These salts were added in 10 per cent aqueous solution in such quantities as to give a salt content of 1 to 3 per cent in the plaster. No efflorescence was visible in the majority of cases and any decrease in adhesion was very small.

The experimental results indicated that the sequestering agents did not cause visible efflorescence, and suggested the interesting possibility that they might suppress efflorescence caused by sodium sulphate. Some experiments were done to investigate this aspect using sodium polyphosphate in preference to E.D.T.A., which preliminary tests had suggested was likely to be less effective.

A 10 per cent aqueous solution of sodium sulphate (5 ml.) was added through the face of 2 in. diameter plaster discs to give a concentration between 0.4 and 1.6 per cent sodium sulphate (5 to 20 g./ft.^{2} of plaster surface). All of the specimens showed efflorescence after drying for twenty-four hours. The efflorescence was removed by rubbing from one-third of the total number; one-third of the specimens were brushed with three coats of distilled water, and the remainder were given three coats of a 15 per cent solution of sodium polyphosphate. The specimens were painted with a latex paint and allowed to dry for three days. The adhesion of the paint was then measured and the results are shown in Table I.

Although the results were very encouraging, these experiments examined only the immediate action of sodium polyphosphate, and it was necessary to ascertain whether this salt would have any long-term effects on plaster. It was known that aqueous solutions of sodium polyphosphate hydrolysed when boiled, but the stability of the salt in the presence of small amounts of water was not known. In order to determine the stability under these conditions two series of experiments were carried out. In the first of these a 15 per cent solution of sodium polyphosphate that had been boiled for fifty hours was added to plaster specimens, which were then kept under observation to see whether any efflorescence occurred. No efflorescence was observed and

TABLE I

TREATMENT OF SURFACES SHOWING SODIUM SULPHATE EFFLORESCENCE

Sodium Sulphate Content of Plaster (%)	Sodium Sulphate per Area of Surface (g./ft.^{2})	Adhesion		
		Wiped Clean	Treated with Water	Treated with Sodium Polyphosphate
1.6	20	100% off at 5 lb./in.^{2}	100% off at 5 lb./in.^{2}	Starts to lift at 80 lb./in.^{2}
0.8	10	100% off at 10 lb./in.^{2}	100% off at 10 lb./in.^{2}	A few pin points at 100 lb./in.^{2}
0.4	6.3	50% off at 40 lb./in.^{2}	50% off at 40 lb./in.^{2}	A few pin points at 100 lb./in.^{2}

some samples that had been painted showed normal adhesion. However, it was found that the decomposition product had lost any power to suppress efflorescence.

In the second series 2 in. plaster specimens were treated with sodium polyphosphate solution and then painted. The amount of the salt introduced in this way was from 4.5 to 7.5 per cent, or 57 to 100 g./ft.^{2} of plaster surface. These amounts are much greater than would normally be introduced. The specimens were then painted, some with a latex paint and the others with a flat alkyd enamel, and 5 ml. of water was introduced from the back, twice per week, and allowed to evaporate through the painted surface. The amount of water added to the plaster specimens and evaporating through the surface each week corresponded to approximately 32 per cent of the weight of the specimen. After twenty weeks no decrease in adhesion was observed. The specimens painted with the flat enamel showed slight efflorescence, but this could be easily wiped off. It was estimated that about twenty times as much water as would be experienced in the worst situation in practice was evaporated through the surface. The results of this experiment were verified by larger scale tests, using fibrous plaster specimens 1 ft. square.

In order to determine the ratio of sodium polyphosphate to sodium sulphate that would suppress efflorescence, aqueous solutions made up of various proportions of 25 per cent solutions of the two salts were applied in 5 ml. amounts to the faces of 2 in. plaster discs. The same amount of sodium sulphate was added to a second group of discs, but without the sequestering agent. After drying, the samples containing only sodium sulphate showed copious efflorescence. The samples were then painted with a latex paint and when this was dry 5 ml. of water was added through the back and allowed to evaporate through the painted front face. The results are summarised in Table II.

The specimens listed in Table II after 16 per cent of the weight of plaster had been evaporated through the painted faces are shown in Fig. 1. Specimens 4 and 5 began to show signs of efflorescence under the paint film when more water was added, but samples 2 and 3 showed no signs of efflorescence even after 200 per cent of water had evaporated through the surface.

TABLE II

ADHESION OF LATEX PAINTS TO PLASTER CONTAINING VARIOUS RATIOS OF SODIUM POLYPHOSPHATE TO SODIUM SULPHATE

Specimen No.	Sodium Polyphosphate on Plaster (%)	Sodium Sulphate on Plaster (%)	Ratio of Sodium Polyphosphate to Sodium Sulphate	Sodium Sulphate (g./ft.^{2})	Adhesion (lb./in.^{2})
1	4	—	—	—	>100
2	3.64	0.36	10.00	15	
3	3.25	0.75	4.34	31	
4	2.56	1.44	1.78	63	
5	1.84	2.16	0.85	95	
6	—	0.36	—	15	10
7	—	0.75	—	31	5
8	—	1.44	—	63	5
9	—	2.16	—	95	5

The experiments described so far were carried out with lime-free plaster, as this is used in the production of fibrous plaster sheet. However, as efflorescence often occurs on set plaster which contains lime, *e.g.* set plaster or fibrous plaster jointing, it is of interest to see whether sodium polyphosphate would be as effective on plaster containing lime. Unfortunately, tests showed that it was not effective in suppressing efflorescence when used in this way.

Calcium chloride and calcium bromide do not cause any efflorescence even at very high concentrations, and sodium chloride and sodium bromide are much less troublesome than sodium sulphate. It follows from these observations that if sodium sulphate could be replaced by sodium chloride or sodium bromide the adhesion of paint films to plaster surfaces containing sodium sulphate could be improved.

In order to investigate this possibility, surfaces of pure plaster and of plaster containing lime were loaded with up to 80 g./ft.^{2} of calcium chloride and calcium bromide. These quantities are twenty to forty times the amounts that would be required to react with the sodium sulphate. Part of each specimen was painted with a latex or a flat enamel paint, and the other part was left unpainted. In no case was there any noticeable efflorescence or peeling of the paint film. However, the high concentrations of the salts decreased the adhesion of the p.v.a. paint to 40 lb./in.^{2}, but there did not seem to be any

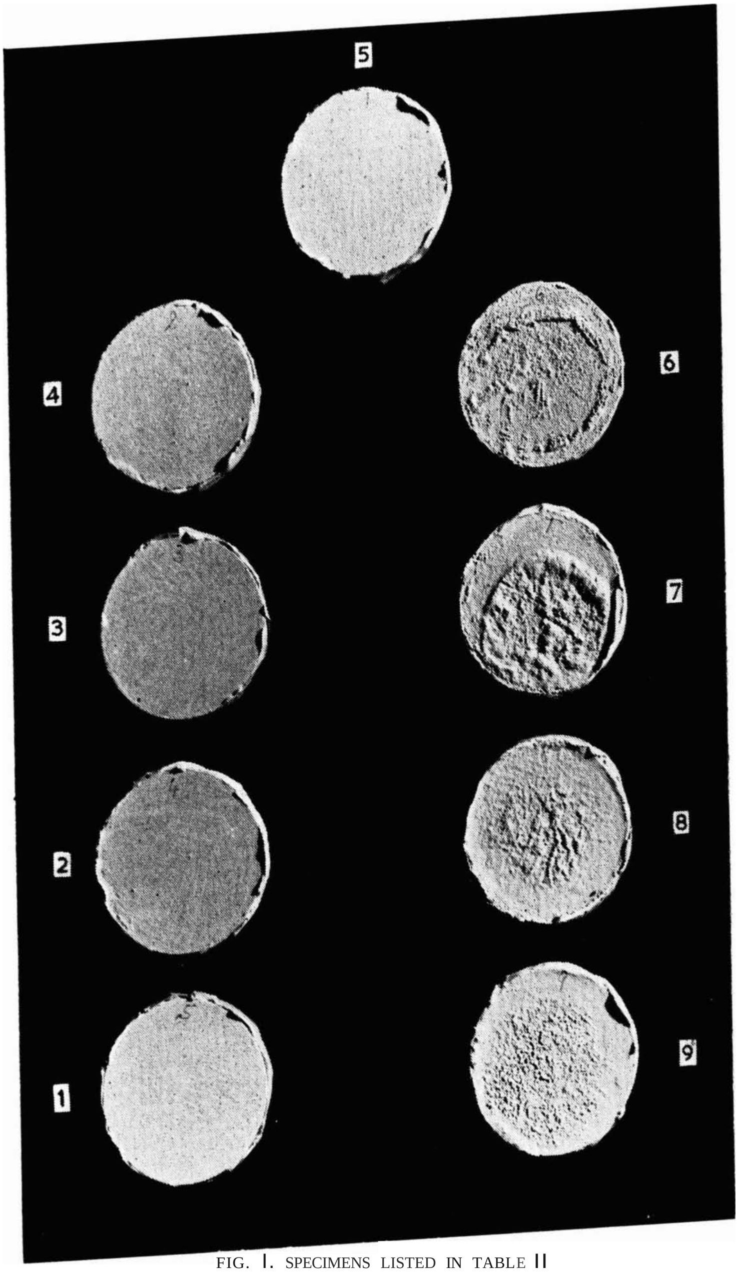
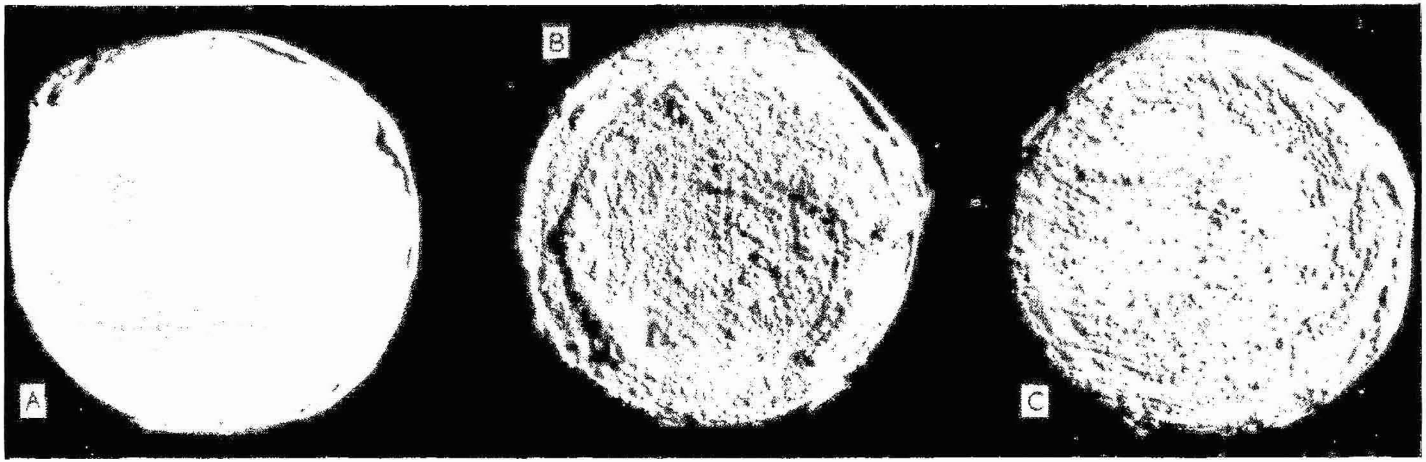


FIG. 1. SPECIMENS LISTED IN TABLE II



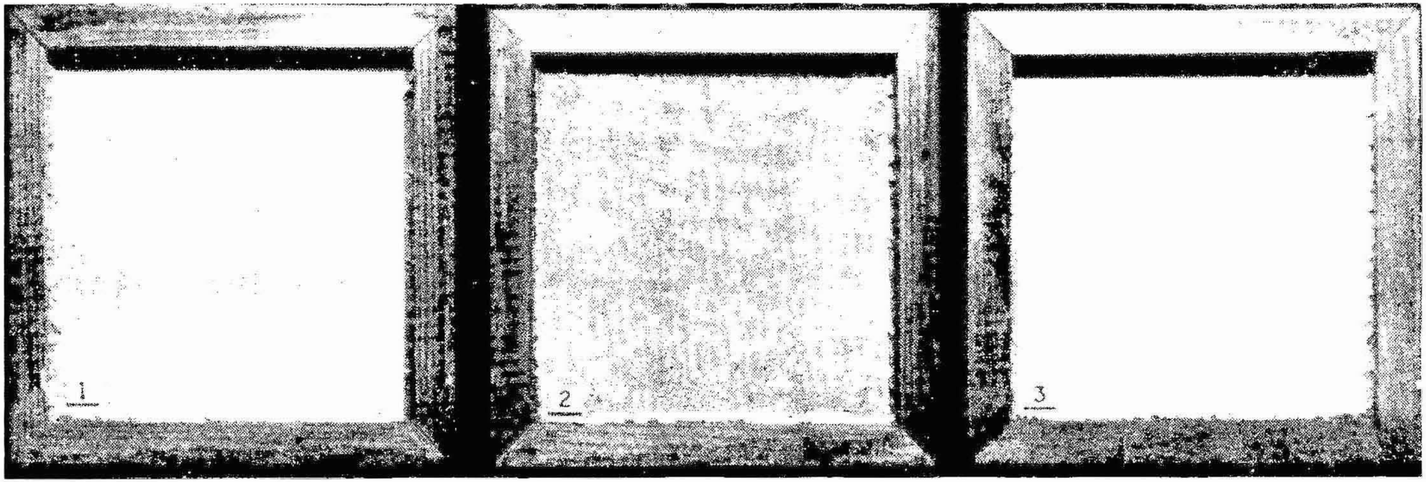


FIG. 2 (*Upper*). A—TREATED WITH CALCIUM CHLORIDE. B—PAINTED WITH LATEX PAINT. C—PAINTED WITH FLAT ALKYD ENAMEL

FIG. 3 (*Lower*). 1—TREATED WITH CALCIUM CHLORIDE. 2—NO TREATMENT TO SUPPRESS EFFLORESCENCE. 3—TREATED WITH CALCIUM BROMIDE decrease in the adhesion of the flat enamel paint. The effect of treatment with calcium chloride can be seen from the photographs in Fig. 2.

The 2 in. plaster specimens were loaded with sodium sulphate to give a concentration of 20 g./ft.^{2} Specimen A was treated with a 15 per cent solution of calcium chloride to give a surface concentration of 35 g./ft.^{2} before applying a latex paint. Specimens B and C were painted with a latex and flat alkyd enamel respectively, without previous treatment with calcium chloride. Water was then added from the back and this was allowed to evaporate through the painted surfaces. Similar results were obtained using calcium bromide instead of calcium chloride.

The small quantity of sodium sulphate needed to cause an almost complete failure is evident from Fig. 3. The plaster specimens are 1 ft. square and to all of them about 2 g./ft.^{2} of sodium sulphate was added to the surface. Specimen No. 1 was then treated with calcium chloride (approximately 50 per cent excess) and specimen No. 3 was treated with calcium bromide. The specimens were then painted with a p.v.a. paint and 200 ml. of water was added through the backs, which were then sealed with polythene sheeting. The photograph shows the appearance after storage for four days at 18°C and 68 per cent R.H. After one month another 200 ml. water was added from the back without producing any change in appearance. No further change was visible six months later.

A series of experiments was carried out to simulate what happens when an apparently dry surface of plaster is painted. Specimens 1 ft. square were painted with a latex or with a flat alkyd enamel. One specimen of each pair was treated with a solution of calcium chloride before painting. After the paint film had dried, 200 ml. of a solution of sodium sulphate was introduced from the back, which was then sealed. The amount of sodium sulphate introduced in this way was 8, 4, 2, 1 and 0.5 g./ft.^{2} As little as 2 g./ft.^{2} of sodium sulphate could cause almost 100 per cent failure. On an untreated surface, 1 g. sodium sulphate caused a failure over about 30 per cent of the surface. The effect of 8 g./ft.^{2} of sodium sulphate is illustrated in Fig. 4.

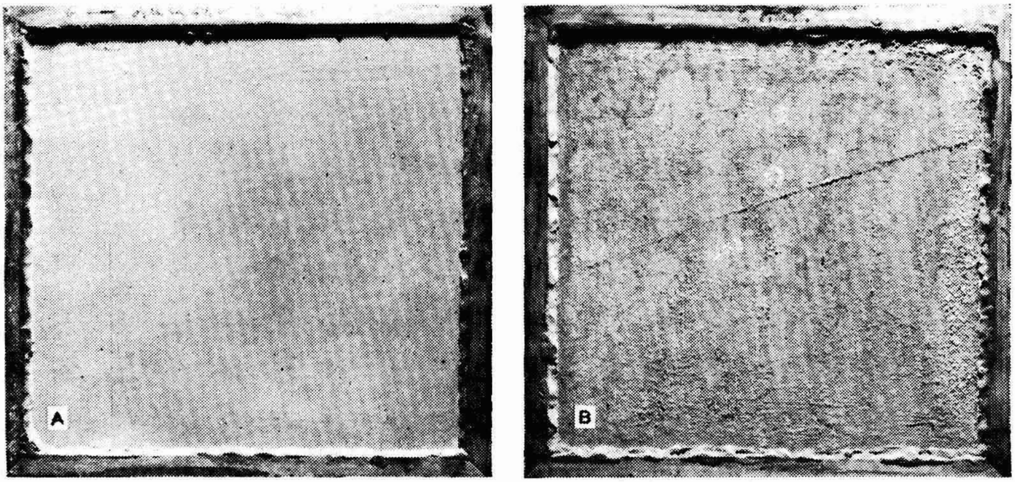


FIG. 4. A—TREATED WITH CALCIUM CHLORIDE. B—EFFLORESCENCE OF SODIUM SULPHATE

Experiments carried out with plaster containing 21 per cent of lime showed that treatment with calcium chloride to suppress efflorescence is also effective in the presence of lime.

DISCUSSION AND CONCLUSIONS

The efflorescence phenomena discussed here have a bearing on the painting of wet plaster surfaces. It is well known that such surfaces occasionally show adhesion failures due to efflorescence, which usually lead to considerable expenditure for redecorating or repairing. The tendency, therefore, is to defer painting operations until the walls are considered to be dry, but there is no reliable method by which the painter may determine when the amount of free water in the plaster is low enough for painting to be safely undertaken.

The work described in this paper has shown that sodium sulphate contained in plaster causes the most damage to the paint film, and its process of formation has been explained. A simple and rapid analytical method has been devised to determine whether the wall material contains sodium sulphate or salts that could react with calcium sulphate to form sodium sulphate. A sample of about 0.5 in.^{2} of the rendering (plaster and concrete) is crushed, extracted with water, and the extract is analysed for sulphate and soluble chlorides. Experiments have shown that 0.5 g./ft.^{2} of sodium sulphate does not cause harm. If more is present the wall could be allowed more time for drying or the sodium sulphate could be rendered inactive by applying calcium chloride or calcium bromide solution.

The need to remove a sample of the material from the wall can be avoided by making an analysis of the cement, plaster, sand and brick used in the construction. This would also give a good indication as to whether or not it is necessary for the surfaces to be completely dry before painting. It must, of course, be realised that the procedure recommended here for judging whether it is reasonably safe to paint a new wall and the remedy proposed in case of efflorescence is far from ideal.

In this paper it has been proposed that calcium chloride or calcium bromide be used to inactivate the sodium sulphate. It is clear that other soluble calcium salts could be used for this purpose, and some of them may be better than those proposed here. It will be recalled that vinegar was formerly applied to fresh plaster and cement surfaces to "kill" the alkalinity. Although the amount of vinegar applied was probably insufficient to neutralise the comparatively large amounts of lime present in set plaster, the acetic acid of the vinegar could react either with free lime or with calcium carbonate to give soluble calcium acetate which, in turn, could react with any soluble sulphates present. Other acids that have sometimes been used could do the same.

One phenomenon of efflorescence that is rather puzzling and appears to warrant further investigation is the fact that sodium sulphate, even if present in very small amounts, appears as a deposit on the surface, whereas other salts, *e.g.*, sodium chloride, do so to a much lesser degree. A detailed study of the mechanism of drying of wet plaster and of the way in which salt solutions diffuse in plaster may form the basis for a better method of avoiding troubles from efflorescence.

It is evident from this investigation that some of the compounds recommended for cleaning and removing old paint could cause efflorescence if the cleaning solution was absorbed by the plaster.

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APPENDIX I

Formation of Sodium Sulphate

If the anion of the salt introduced gives a highly soluble salt with the calcium ion, there is a possibility of the formation of sodium sulphate. The likelihood that this will occur can be estimated from the solubility of the salts concerned.

It may be best to exemplify the procedure by an example. Suppose that the wet plaster contains sodium chloride; in what form will the sodium ions be precipitated when the plaster dries?

The solubility of sodium sulphate at 20°C	3.1 mole/litre
The solubility product of sodium sulphate	$(3.1)^3=29.8$
The solubility of sodium chloride at 20°C	6.1 mole/litre
The solubility product of sodium chloride	$(6.1)^2=37.2$

Sodium sulphate will be precipitated when its solubility product, that is the value of the product $(Na^+)^2\ (SO_4'')=29.8$, *i.e.* precipitation will occur when the Na^+ concentration equals $\sqrt{\dfrac{29.8}{1.5\times10^{-2}}}=44.6$. Sodium chloride on the other hand will be precipitated when $(Na^+)\ (Cl')=37.2$, or $(Na^+)=6.1$. It will be seen that under these conditions the sodium ion will appear as sodium chloride. However, if the plaster should contain soluble sulphates such as magnesium sulphate or ammonium sulphate so that a sulphate anion concentration of, say, 1.5 mole/litre could be built up, then the 1.5×10^{-2} in the above equation is replaced by 1.5 and sodium sulphate will be precipitated when the sodium ion concentration reaches a value of 4.8 mole/litre.

In the above calculations it has been assumed that the electrolytes are completely dissociated. This, of course, is not correct, but such calculations are quite valuable for preliminary orientation in spite of the approximation that is involved.

The Design of Unsaturated Polyester Resins For Surface Coatings*

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Summary

The design of unsaturated polyester systems is discussed in relation to the problems of "air-inhibition" and gel-time. It is shown that air-inhibition may be overcome either by building allyl ether groups into the polyester chain, or by using a low molecular weight polyallyl ether as the monomer. The latter method is preferred because there is then no loss of monomer by evaporation. The use of certain unsaturated polyesters with melting points above 90°C is also shown to avoid the formation of tacky surfaces, but brittle films result. The lower aliphatic oximes are found to extend the gel-times without appreciably increasing the drying times. They function most successfully in wax-free polyester systems based on monomers of low volatility.

INTRODUCTION

Unsaturated polyesters have now become well-established as structural plastics, but are making slower progress in the surface coating field. The simplest form of polyester is obtained by esterifying maleic anhydride with ethylene glycol to form a low polymer with a molecular weight usually between 1,000 and 3,000, of the type illustrated below:

$$\mathrm{HOOC-CH{=}CH-COO-CH_2-CH_2-OOC-CH{=}CH-COO-CH_2}$$
$$\mathrm{-CH_2-OOC-CH{=}CH-COO-CH_2-CH_2-OH}$$

Other glycols, especially propylene glycol, are frequently used, and part of the maleic acid is usually replaced by some other modifying acid, such as phthalic. Fumaric acid may replace maleic anhydride or acid. Solutions of such polyesters in styrene will form three-dimensional networks if treated with free radical liberating compounds. Since styrene, which initially acts as a solvent, takes part in the polymerisation reaction, polyester systems have been described as solventless coatings.

The use of polyester systems in surface coatings involves some difficulties. Their polymerisation is usually initiated by the addition of an organic hydroperoxide and an activator, but once the peroxide has been added the useful life of the material is limited. This means that polyesters must be "two-component" systems and that the peroxide should not be added until just before the polyester is applied. Several mechanical arrangements are available to ensure that the polyester and the peroxide do not come into contact until just before application. A further disadvantage of normal polyester systems is their failure to cure satisfactorily at surfaces in contact with air. This phenomenon is usually referred to as air-inhibition. This paper describes work carried out to produce polyesters which will cure in the presence of air and

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*Read before the London Section on 28 September, 1960.

which possess gel-times long enough for them to be used with conventional spraying equipment.

POLYESTER SYSTEMS WHICH CURE IN THE PRESENCE OF AIR

Ordinary polyester systems will not cure satisfactorily in thin films if air is present. The under-surface of the film is strong and tough, but the surface exposed to air is tacky and easily attacked by solvents. Polyester films will cure satisfactorily in an atmosphere of nitrogen so that the principal factor in air-inhibition must be the presence of oxygen, which is known to interfere with free radical polymerisation[1]. Thus, Kolthoff and Bovey[2] showed that the presence of oxygen during the emulsion polymerisation of styrene greatly decreased both the rate and degree of polymerisation. Localised depletion of styrene at the surface caused by evaporation could account for the phenomenon, but since systems based on monomers of very low volatility are still air-inhibited it is presumably not the main factor.

Air may be excluded by mechanical methods, such as covering the surface with a layer of plastic during curing, but by far the simplest method is by adding a small quantity of a high melting point wax to the polyester. The wax functions by being more soluble in the uncured polyester than in the cured material, so that as the applied film of polyester polymerises the wax is precipitated and squeezed out to the surface, where it behaves as a barrier to oxygen. The amount of wax used varies from 0.001 to 1 per cent[3, 4, 5]. The wax barrier also impedes the evaporation of styrene, so that evaporation losses, which may be as high as 60 per cent of the total styrene in the absence of wax, fall to approximately 10 per cent or less.

The use of wax, however, has its disadvantages. Adhesion between the polyester and the substrate may be weaker, since the wax also migrates to this interface. The surface is turbid where the wax has exuded, and even after removal by sanding and polishing it may continue to exude, particularly if the polyester film is heated. Sanding is also made somewhat more difficult since the wax tends to clog the cutting paper. The authors have found that the loss of adhesion may be overcome by first spraying with a wax free polyester and then overspraying the surface with a solution of wax after a short interval, thus ensuring that the wax film lies only at the upper surface. The other disadvantages of the use of wax still, however, remain. The work described in this section was carried out in an attempt to avoid the use of wax.

POLYESTERS WITH HIGH MELTING POINTS

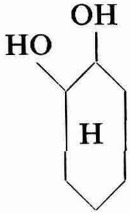
This approach accepts the inhibiting effect of air but aims at producing a polyester which is so hard that even the uncured surface of a film derived from it can be polished without clogging. In the experience of the authors the hardness is related to melting point, and the best results were obtained by using resins having melting points above 90°C. The melting point of a polyester may be increased in several ways. Propylene glycol may be replaced by cyclic diols, which presumably reduce the flexibility of the polymer chains, or amide linkages may be incorporated into the polymer chain, thus increasing the amount of hydrogen bonding. In addition, replacement of *o*-phthalic acid by isophthalic acid gives a small increase in melting point as is apparent from Table

TABLE I

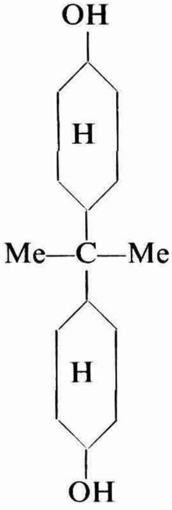
EFFECT OF REPLACING *o*-PHTHALIC BY ISOPHTHALIC ACID ON THE MELTING POINT OF POLYESTERS

Molar ratio isophthalic/*o*-phthalic	M. pt. (Ball and Ring) (°C)
0	66-69
1.0	80-82
3.0	84-88
∝	78-81

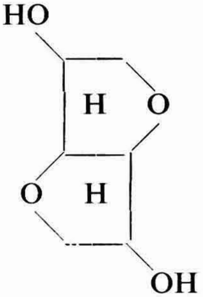
Several cyclic diols have been suggested for this application[6, 7, 8, 9, 10, 11], and the authors have investigated the use of cyclohexane 1.2 diol hydrogenated bisphenol A and 1.4, 3.6 dianhydro sorbitol (isosorbide) which are shown below.



Cyclohexane 1.2 diol



Hydrogenated bisphenol A



Isosorbide

Polyesters prepared from these compounds in combination with isophthalic acid and maleic acid gave resins with melting points greater than 100°C, as shown in Table II.

TABLE II

EFFECT OF 50% MOLAR REPLACEMENT OF PROPYLENE GLYCOL BY CYCLIC DIOLS

Glycols	Acids (in equimolar proportions)	M. pt. (Ball and Ring) (°C)
Propylene glycol only	isophthalic maleic	78-87
Cyclohexane 1.2 diol	isophthalic maleic	115-117
isosorbide	isophthalic maleic	115-118
Hydrogenated bisphenol A.	*o*-phthalic maleic	99-101

Amide linkages were introduced into the polyester by using ethanolamine or ethylene diamine, and when these were used *o*-phthalic acid was replaced by isophthalic acid to avoid imide formation. The results are shown in Table III. When compared on a molar basis, ethylene diamine had more effect on the melting point than monoethanolamine, which indicated that the increase in melting point depended upon the number of amide groups present.

TABLE III

EFFECT OF REPLACING PROPYLENE GLYCOL BY ETHYLENE DIAMINE AND MONOETHANOLAMINE

Polyester based on equimolar proportions of iso-phthalic and maleic acid.

Amine	Molar replacement of propylene glycol (%)	M. pt. (Ball and Ring) (°C)
Ethylene diamine	0	78-87
,, ,,	5	88-93
,, ,,	10	96-99
,, ,,	15	95-97
Monoethanolamine	18	88-91
,,	25	92-95

Resins produced by this method were yellow-brown, the colour increasing with increasing amounts of amine. The colour could be reduced by esterifying at lower temperatures, and completely eliminated if the unsaturated acid was omitted altogether, but mere reduction in the proportion of this component had little effect. It is a fairly simple matter to produce high melting point polyesters, but they are not really satisfactory since, although the cured film is hard, it is solvent-sensitive and distinctly brittle, with a tendency to "craze" when submitted to temperature variations. In addition, because wax is absent, the evaporation losses of styrene are rather high.

POLYESTERS CONTAINING AIR-DRYING GROUPS

It is unlikely that the inhibiting effect of oxygen on free radical polymerisation can be avoided. For this reason much research has been directed towards producing polyester systems which can be polymerised in the presence of oxygen by some alternative mechanism. The simplest solution to the problem would seem to be the addition of alkyds based on air-drying long-chain acids, but this is not possible because they are incompatible with unsaturated polyesters. The incompatibility might be expected to disappear if they both formed part of the same molecule, and a simple method of achieving this would be the co-esterification of a monoglyceride, *e.g.* glyceryl monolinoleate, with maleic anhydride to give a linear polyester with linoleate side-chains. Unfortunately, the esterification reaction is accompanied by a *Diels-Alder* reaction between the linoleate and the maleate groups to form molecules which are polyfunctional with respect to the acid groups, with the result that premature gelation occurs long before esterification approaches completion.

A somewhat similar approach, but one which is claimed to avoid difficulties from gelation, involves the reaction between an acid-terminated polyester and a partially epoxidised alkyd based on a drying oil acid[12]. This reaction, which is essentially an esterification, can be conducted at temperatures low enough to avoid the *Diels-Alder* reaction, and so the molecules may be linked together without gelation occurring. Other methods of linking the two types of molecules are conceivable, such as by the reaction between a diglyceride, a hydroxyl-terminated polyester and a di-isocyanate.

The drying-oil acid grouping, even when successfully introduced into the polyester still possesses four serious disadvantages. Its molecular weight is high and a large proportion is necessary to provide the required air-drying properties. It does not copolymerise readily with styrene or with the maleate group; it tends to yellow on ageing and also have a plasticising effect on the film.

Another air-drying group was therefore sought with as many as possible of the following properties.

(i) Short chain-length.

(ii) Satisfactory copolymerisation characteristics with either styrene or the maleate or fumarate group.

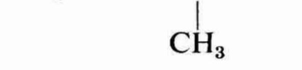
(iii) Low reactivity towards maleic anhydride and its derivatives in the *Diels-Alder* reaction.

The allyl ether grouping ($CH_2{=}CH{-}CH_2O{-}$) conforms with these requirements. It has a chain length of about one-fifth of the linoleate group, and it was found that it could be incorporated into the polyester chain without excessive difficulty from premature gelation. In addition, compounds which are polyfunctional with respect to allyl ethers are usually compatible with polyesters, and will copolymerise with them with ease even though the preparation of homopolymers is difficult[13].

The air-drying properties of polyallyl ethers seem to have been discovered in the nineteen forties. The preparation and properties of a number of polyallyl ethers of various polyhydric alcohols have been described[14, 15, 16, 17], probably the best known being the so-called "allyl-starch"[18] and the allyl ethers of polymethylol melamine[19, 21]. Other related groups have similar properties *e.g.*

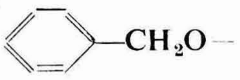
$CH_3{-}CH{=}CH{-}CH_2O{-}$

Crotyloxy[20]



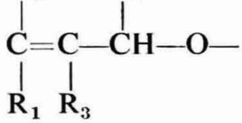
$$CH_2{=}CH{-}\underset{\substack{|\\ CH_3}}{CH}{-}O{-}$$

Methallyloxy



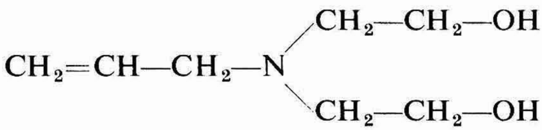
Benzyloxy

This appears to indicate that the structure essential for air-drying is

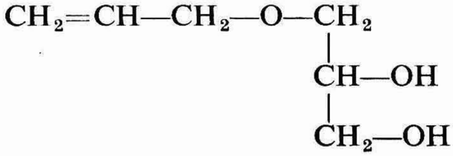


$$\overset{\substack{R_2\\ |}}{\underset{\substack{|\\ R_1}}{C}}{=}\underset{\substack{|\\ R_3}}{C}{-}\overset{\substack{R_4\\ |}}{CH}{-}O{-}$$

Diallyl phthalate and similar compounds in which the allyl group is not linked to ether oxygen do not have air-drying properties; neither apparently do N-allyl compounds, since work in the laboratory showed that polyesters prepared from N-allyl diethanolamine and phthalic anhydride did not share the air-drying properties possessed by a polyester prepared from glycerol monoallyl ether and phthalic anhydride.



N-allyl diethanolamine



$$\begin{array}{l} CH_2{=}CH{-}CH_2{-}O{-}CH_2 \\ \qquad\qquad\qquad\qquad\quad | \\ \qquad\qquad\qquad\qquad\quad CH{-}OH \\ \qquad\qquad\qquad\qquad\quad | \\ \qquad\qquad\qquad\qquad\quad CH_2{-}OH \end{array}$$

Glycerol monoallyl ether

The air-drying properties of polyallyl ethers are similar to those of the linoleates in that their film-forming properties are much improved by the presence of heavy metal soaps, especially cobalt, and their viscosity may be increased by air-blowing at elevated temperatures[16]. Allyl ether groups may be introduced into polyesters either by simple addition of a polyallyl ether to a polyester, or by the preparation of modified polyesters containing chemically bound allyl ether groups. Both of these methods have been investigated and will be described separately.

POLYESTERS CONTAINING ALLYL ETHER SIDE CHAINS

In order to prepare polyesters containing allyl ether side chains, compounds should be used which contain allyloxy groups in addition to the carboxyl or hydroxyl groups which take part in the polyesterification reaction. Some of the most accessible compounds of this class are the partial allyl ethers of polyhydric alcohols which may be readily prepared by the classical *Williamson* ether synthesis, and the work described below was limited to the use of the monoallyl and diallyl ethers of glycerol. Recently, the use of partial allyl and benzyl ethers of other polyhydric alcohols has been described[22].

When a polyester containing allyl ether groups is prepared, the two most important factors affecting its air-drying characteristics are the number of allyl ether groups per mole, *i.e.* the functionality, and the proportion by weight of allyl ether groups. The functionality of the resin, which should be at least 3 and preferably as high as possible, steadily increases with increase in molecular weight in any particular recipe. Table IV shows how the allyl ether functionality is related to molecular weight in the polyesterification of an equimolar mixture of propylene glycol and glycerol monoallyl ether with maleic anhydride.

TABLE IV

RELATION BETWEEN ALLYL ETHER FUNCTIONALITY AND MOLECULAR WEIGHT

Molecular weight of polyester	Allyl ether groups (per mole)
375	1
750	2
1,500	4

The proportion by weight of allyl ether groups required for satisfactory air-drying was determined empirically. This figure will obviously be affected by the functionality, *e.g.* allyl ethyl ether and the triallyl ether of trimethylol propane both contain roughly the same proportion of allyl ether groups, but only the latter has air-drying properties (*cf.* methyl linoleate with glycerol trilinoleate).

In practice the introduction of allyl ether side-chains into an unsaturated polyester reduces the maximum molecular weight which can be obtained without gelation, thus limiting the allyl ether functionality. Table V shows that when polyesters were prepared from propylene glycol, glycerol monoallyl ether and equimolar amounts of phthalic anhydride and maleic anhydride, the maximum allyl ether functionality which could be obtained without gelation was approximately 4.

TABLE V

RELATION BETWEEN GEL POINT AND ALLYL ETHER FUNCTIONALITY

Molar proportion of mono allyl glycerol in total diols (%)	Allyl ether groups in resin (g. mole/100g.)	Max. molecular weight possible without gelation (End group analysis)	Allyl ether functionality of resin
0	0	3,000	0
25	0.12	3,000	3.6
50	0.24	1,800	4.4
80	0.33	1,000	3.3
100	0.40	800	3.2

Complete omission of maleic anhydride overcame these difficulties and much higher molecular weights could be obtained, though the polyester would no longer copolymerise with styrene. Mere reduction of the proportion of maleic anhydride used to 25 molar per cent of the total acids had little or no effect on the tendency to "gel". In view of this, work on glycerol monoallyl ether was limited to the effect of varying the molar proportion of the allyl ether in the diol mixture, each polyester having an allyl ether functionality of 3-4.

The film properties of these polyesters dissolved in styrene showed that the hardness and general resistance of the surface increased as the proportion of allyl ether groups was raised. Replacement of 30 molar per cent of the propylene glycol gave surfaces which could be cut without clogging the cutting paper, whilst an 80 per cent replacement gave a tough, hard surface, which was almost completely resistant to solvents and scratching with the finger-nail. From these and similar results it was estimated that for satisfactory polishability, the polyester should contain approximately 0.15 g. mole of allyl ether per 100g. and 0.33g. mole/100g. for mar-resistance.

When glycerol mono allyl ether is replaced by glycerol diallyl ether, the allyl ether functionality of the polymer is automatically limited to 4, because the glycerol diallyl ether is a monofunctional alcohol which acts as a "chain-stopper" giving polyesters with two allyl ether groups at each end. Preparation of a number of polyesters with varying amounts of the di-ether showed that for satisfactory polishability, such polyesters should contain approximately 0.2g. moles of allyl ether per 100g., whilst for mar-resistance this figure should be increased to about 0.45g. mole/100g.

The molecular weight of a polyester from glycerol diallyl ether containing 0.45g. mole of allyl ether groups per 100g., is limited to about 800. Comparison with a polyester of similar molecular weight to monoallyl glycerol, which contained only 0.33g. mole of allyl ether groups per 100g., showed that the latter, in spite of its lower allyl ether content, had better air-drying properties. Since the functionalities of the two polyesters are respectively 4 and 3 the difference must be ascribed to differences in the mode of distribution of the allyl ether groups along the chain. These results are summarised in Table VI.

TABLE VI

COMPARISON OF AIR-DRYING PROPERTIES OF POLYESTERS PREPARED FROM GLYCEROL MONO- AND DIALLYL ETHER

Ether	Allyl ether groups in resin required for (g. mole/100g.)	
	Polishability	Mar-resistance
Glycerol mono allyl ether	0.18	0.33
Glycerol diallyl ether.	0.20	0.45

The final polyesters produced from either of these systems were completely cured as was shown by the excellent solvent resistance of films made from them. The films were tough, clear and mar-resistant, and their adhesion was good. However, they had a serious disadvantage in that losses of styrene by evaporation might amount to 40 per cent of the monomer present.

POLYALLYL ETHERS AS COPOLYMERISING SOLVENTS FOR POLYESTERS

The molecular weights of unsaturated polyesters containing allyl ether side chains are limited by their tendency to gelation if the polyesterification is carried too far. The heavy losses of styrene which occur from thin films of these materials is a more serious disadvantage, which is aggravated if the drying time of the polyester is prolonged. These defects would be overcome by the development of a system consisting of a conventional polyester dissolved in a monomer of low volatility with the additional properties of good copolymerisation characteristics with unsaturated polyesters, good air-drying properties and low viscosity. Such a material would give air-drying polyesters which were truly solventless.

Derivatives of linoleic acid, as we have already seen, must be rejected on grounds of incompatibility and poor copolymerisation characteristics. Polyallyl ethers on the other hand are compatible with polyesters, possess fairly low viscosities and have good air-drying characteristics. Their reactivity towards unsaturated polyesters is high as can be seen from Table VII, which shows the peak exotherms obtained when the same unsaturated polyester is dissolved in a number of different polyallyl ethers and styrene. This high reactivity contrasts with the extreme reluctance of these materials to homopolymerise.

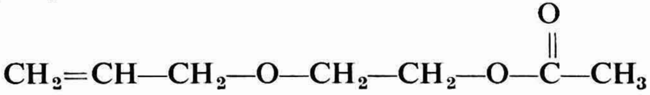
TABLE VII

PEAK EXOTHERMS OF AN UNSATURATED POLYESTER IN VARIOUS MONOMERS

60% solutions, 20g. samples. The polyester was prepared from 4 moles propylene glycol, 3 moles fumaric acid and 1 mole phthalic acid.

Monomer	Peak exotherm (°C)
Styrene	189
Acetate of glycerol diallyl ether	175
Adipate of glycerol diallyl ether	169
Glycerol diallyl ether	140

Some indication of the general behaviour of the allyl ether group in copolymerisation can be obtained by the use of a simple monofunctional substance such as β-allyloxyethyl acetate.



$$CH_2{=}CH{-}CH_2{-}O{-}CH_2{-}CH_2{-}O{-}\overset{\overset{\displaystyle O}{\|}}{C}{-}CH_3$$

β-allyloxyethyl acetate

This substance copolymerised with diethyl fumarate over a wide range of molar ratios even though neither monomer will homopolymerise. On the other hand, attempted copolymerisation of this allyl ether with styrene yielded almost pure polystyrene, even from an initial monomer mixture containing 75 molar per cent of the allyl ether. Such results imply that when a mixture of an allyl ether and styrene is used as the monomer for a polyester, the allyl ether will copolymerise almost exclusively with the fumarate groups of the polyester, any unused allyl ether remaining unpolymerised.

Choice of Monomer

The high reactivity of the allyl ether group with the fumarate groups in polyesters thus being established, it remained to determine the most suitable materials in terms of air-drying properties, volatility and viscosity. Polyallyl ethers, such as those derived from melamine derivatives and those derived from esterification of glycerol monoallyl ether with phthalic anhydride, were rejected because of their high viscosities. Since the viscosity is to a large extent dependent upon the molecular weight, the polyallyl ether chosen should have the lowest molecular weight consistent with a reasonably high functionality.

The air-drying characteristics of the polyallyl ether would be expected to improve with increase in allyl ether functionality. Table VIII summarises the film properties obtained when three parts of an unsaturated polyester are dissolved in two parts of various polyallyl ethers of different functionalities.

TABLE VIII

EFFECT OF ALLYL ETHER FUNCTIONALITY ON AIR-DRYING PROPERTIES

Monomer		Functionality	Allyl ether groups in resin (g. mole/100g.)	Order of mar-resistance after 18 hours	Touch dry time (hr.)	Viscosity of monomer at 25°C (c/poises)
Glycerol diallyl	ether ..	2	1.18	4 (worst)	14	—
	acetate ..	2	0.95	3	14	Approx. 7
	adipate (GDA)	4	0.87	2	6	29
	pyromellitate	8	0.93	1 (best)	1½	2000

The use of the glycerol diallyl ether adipate gave a reasonable compromise between acceptable viscosity and good air-drying properties and G.D.A. was in fact chosen for the work described below. This material has only a slight odour and is non-volatile at ordinary temperatures. In contrast to many other monomers it is remarkably stable even at temperatures of 180°-200°C, providing that air is absent, and shows little tendency to polymerise alone, even at elevated temperatures.

Concentration of allyl ether

The concentration of G.D.A. used in the final lacquer composition should be chosen having regard to both the air-drying properties required at the surface and the bulk properties of the film. Experiments have shown that the composition must contain at least 40 per cent of the monomer if a hard mar-resistant surface is to be obtained. It is interesting that this concentration corresponds to 0.35g. mole of allyl ether groups in 100g. of the film, which is similar to the figure obtained for polyesters with built-in allyl ether groups. The best concentration of the monomer from the point of view of general film properties was also found to be about 40 per cent. The effect of changes in the composition of the film on the hardness is shown in Table IX. The maximum hardness is attained in all cases at a level of 40 per cent monomer, which also corresponds roughly with the maximum heat of polymerisation shown in Fig. 1.

TABLE IX

EFFECT OF COMPOSITION OF UNSATURATED POLYESTER ON FILM-HARDNESS

Polyester composition		Sward hardness of film (Glass=100)		
		G.D.A. concentration		
Unsaturated acid	Molar ratio unsaturated acid/phthalic acid	30%	40%	50%
Fumaric ..	3	26	28	23
Maleic ..	2	13	15	13
Fumaric ..	1	15	18	15
Fumaric ..	0·5	15	17	12

Choice of Polyester

Some consideration was also given to the performance of different types of polyester when used with G.D.A. Examination of the reactivity ratios of fumarates and maleates when copolymerised with a number of other monomers shows that in general fumarates are more reactive than maleates. This also appears to be true for copolymerisations involving allyl ethers, since the gel times and film drying times of polyallyl ethers are shorter when copolymerised with diethyl fumarate than with diethyl maleate (see Table X).

TABLE X

COPOLYMERISATION OF DIETHYL FUMARATE AND DIETHYL MALEATE WITH POLYALLYL ETHERS

Type of polyallyl ether	Gel time of solutions in		Drying time of same solutions* (when cast as films 0·015 in thick.)	
	Diethyl fumarate	Diethyl maleate	Diethyl fumarate	Diethyl maleate
Adipate of glycerol diallyl ether ..	16 hr.	72 hr.	16 hr.	72 hr.
Polyester from glycerol monoallyl ether and phthalic acid	70 min.	120 min.	18 hr.	5 days

*Catalysed with 0.1% cobalt as metal and 2.4% M.E.K. peroxide.

The differences between maleate and fumarate polyesters are less striking, probably because isomerisation occurs during the polyesterification reaction[25], but the advantage usually seems to lie with the fumarate polyester as the peak exotherms in Fig. 1 show. The ratio of unsaturated acid to saturated acid should for preference be greater than one. Ratios below this value give low peak exotherms (see Fig. 1) and rather weak films.

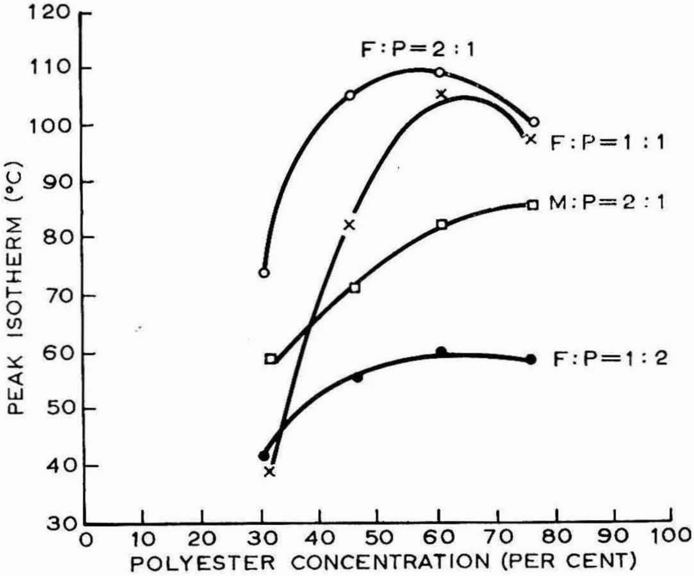


Fig. 1. Variation of Peak Isotherm with Type of Polyester.
F=Fumaric Acid M=Maleic Anhydride P=Phthalic Anhydride

Viscosity

In a system of this kind the amount of monomer to be used must be chosen not only to give the required film properties, but also so that the viscosity of the composition is suitable for the particular application method to be used. The viscosity will, of course, depend on the molecular weight as well as on the concentration of the polyester used. Table XI shows the viscosity of two polyesters of the same composition, but of different molecular weights, when dissolved in G.D.A. and styrene.

TABLE XI

Viscosity of Polyesters in Styrene and G.D.A.

M.wt of polyester (based on acid value)	Polyester concentration	Viscosity of solution (c/poises)	
		G.D.A.	Styrene
1300	40	395	16
,,	50	937	34
,,	60	3530	112
,,	70	9050	295
2300	40	3120	34
,,	50	11900	117
,,	60	34000	730
,,	70		9430

It has already been shown that the optimum film properties are obtained at a concentration of 40 per cent G.D.A. In general, suitable spraying viscosities are not obtained at this concentration and the viscosity must, therefore, be reduced either by further addition of the polyallyl ether, styrene, or a suitable volatile solvent.

Rate of drying

Satisfactory air-drying of these systems is not obtained when dimethylaniline and benzoyl peroxide are used for initiation. On the other hand, cobalt-hydroperoxide systems give satisfactory results, although the cobalt concentration affects the drying rate and must, therefore, be chosen with care. The effect of cobalt concentration on the touch-dry time is illustrated in Table XII.

TABLE XII

EFFECT OF COBALT CONCENTRATION ON DRYING TIME AT 23°C

Cobalt concentration (%)	Touch-dry time (hr.)
0.012	18
0.024	18
0.05	10
0.1	8½
0.2	7½

These results show that 0.1 per cent cobalt is the optimum level required for a satisfactory rate of air-drying. Further increases in the cobalt level had little effect on the drying time and merely resulted in darker films. The drying time is not much affected by the type of unsaturated polyester used or by the film thickness. A series of 60 per cent solutions of unsaturated polyesters with fumarate: phthalate molar ratios varying from 3 to 0.5 all gave drying times of 5-6 hours at 24°C, independent of film thickness over the range 0.001-0.008 in.

Film Properties

The final film obtained from a mixture of two parts G.D.A. and three parts polyester (based on 3 moles of propylene glycol, 2 moles of fumaric acid, and 1 mole of phthalic acid) was examined for hardness, and resistance to alcohol and water. For comparison the results from a conventional polyester dissolved in styrene containing wax are included in Table XIII.

TABLE XIII

COMPARISON OF THE FILM PROPERTIES OF A CONVENTIONAL POLYESTER COMPARED WITH ONE BASED ON G.D.A.

Film thickness = 0.008 in.

Test	Type of polyester	
	Conventional	G.D.A.
Sward Hardness (glass = 100) after four days	20	27
Water absorption (24 hr. immersion at room temp.) (%) ..	0.4	0.6
Alcohol absorption (24 hr. immersion at room temp.) (%) ..	1.5	6

Despite the relatively high alcohol absorption of the films from G.D.A., the gloss is almost unaffected by immersion in alcohol.

THE FUNCTION OF ALLYL ETHERS IN OVERCOMING AIR-INHIBITION

It has been shown that the introduction of allyl ethers into polyesters improves the mar-resistance and solvent-resistance of surfaces exposed to air; further improvement can be obtained if wax is added. This is true even when non-volatile monomers, such as G.D.A., are used, in which case the wax presumably acts solely as a barrier to atmospheric oxygen. These results imply that when surfaces of polyesters based on allyl ethers are exposed to air, they dry by a different mechanism from the bulk of the film. This conclusion is supported by the observation that when cured films of polyesters based on G.D.A. are removed and immersed in alcohol, they curl up in such a way that the surfaces previously exposed to air are on the outside of the roll. This points to differential swelling and suggests that the upper surface of the cured film is chemically different from the bulk of the film. These results can be explained by assuming that whilst the bulk of the film cures by a normal free radical co-polymerisation between allyl ether and fumarate groups, the surface exposed to air cures by some kind of oxidative polymerisation similar to that involved in the case of allyl starch.

THE USE OF OXIMES FOR INCREASING POT-LIFE

Any surface coating which cures at room temperature by the addition of an external catalyst system has a limited pot-life once the catalyst has been added; the shorter the pot-life, the shorter the drying time. It has been shown that it is possible to design polyester systems which cure satisfactorily in the presence of air and lose little or no monomer by evaporation. These materials would clearly be more attractive if their pot-lives could be extended to the length of a working day.

The pot-life of a polyester may be increased by adding inhibitors, such as quinones, but the drying time is also increased. However, if volatile inhibitors could be found which would evaporate quickly from the polyester in film form, the pot-life could be increased with little increase in the drying time of the film. The lower aliphatic oximes, which are volatile liquids or low melting point solids, have been found to inhibit the polymerisation of polyester systems when initiated by cobalt salts and organic hydroperoxides. The boiling points of three oximes are shown in Table XIV and these are clearly low enough for the materials to be examined as volatile inhibitors.

TABLE XIV

BOILING POINTS OF ALIPHATIC OXIMES

Oxime	B. pt. (°C)
Acetoxime	136
Acetaldoxime	115
Methyl ethyl ketoxime	152

These substances act as inhibitors only when initiator systems of the cobalt hydroperoxide type are used, and they have little effect with dimethyl aniline and benzoyl peroxide. Their action appears to be due principally to their capacity to form complexes with cobaltic cobalt, thus lowering its effective concentration as a promoter, and thereby reducing the rate of decomposition of the hydroperoxide (see Table XV).

TABLE XV

DECOMPOSITION OF 4% HYDROXYCYCLOHEXYL HYDROPEROXIDE IN THE PRESENCE OF 0.03% COBALT NAPHTHENATE AND VARYING AMOUNTS OF ACETOXIME. (SOLUTION IN 74 O/P METHYLATED SPIRIT.)

Oxime concentration (%)	Half-life of peroxide at 25°C
0	40 min.
0.05	1 hr.
0.12	2 ,,
0.20	4½ ,,
0.40	24 ,,

The addition of oxime to a polyester initiated with cobalt and hydroperoxide intensifies the green colour of the cobalt. The intensity of this colour depends on the concentration of oxime and cobalt. Since reduction in the cobalt concentration would seriously affect the drying properties of the film, ways must be sought of keeping the oxime concentration to a minimum. The gel time of a polyester will depend, other things being equal, on the cobalt concentration, the concentration of oxime, and the concentration and kind of peroxide employed. If the cobalt concentration is fixed, the amount of oxime required to give a gel time of eight hours will depend upon the ease of decomposition of the peroxide used, the most active peroxides requiring the largest amount of oxime. Table XVI shows that cumene hydroperoxide requires less oxime than either methyl ethyl ketone peroxide or cyclohexanone peroxide and is, therefore, to be preferred on these grounds.

TABLE XVI

RELATIONSHIP BETWEEN OXIME CONCENTRATION AND GEL TIME FOR VARIOUS CATALYST SYSTEMS AT 23°C

All concentrations calculated on 100 parts of resin. Peroxide concentrations were chosen to give the same concentration of peroxide oxygen as determined iodiometrically.

Peroxide type	Peroxide concentration (%)	Cobalt concentration (%)	Acetoxime concentration for gel time of 8 hours (%)
Methyl ethyl ketone peroxide	2.5	0.06	1.0
Cyclohexanone peroxide	4	0.06	0.7
Cumene hydroperoxide	5.6	0.06	0.3

There are two additional requirements which must be met if a volatile inhibitor is to be effective. It must be able to evaporate without impediment from the film, and it must be considerably less volatile than the monomer used. Attempts to use the oximes in conventional polyesters containing added wax failed in both these respects, for the wax impedes evaporation of the oxime, and the styrene used as monomer (B. pt. 145°C) is as volatile as the inhibitor. The use of oximes in systems consisting of a polyester with built in air-drying properties dissolved in styrene was still not entirely satisfactory, because the oxime increased the drying time sufficiently to cause increased losses of monomer by evaporation.

From these results it might be expected that a polyester system which contains no wax, and which uses monomers of low or negligible volatility, would be more suitable for use with oximes. This has been confirmed, and the application of oximes to polyester systems based on G.D.A. gives gel times of eight hours without either a serious increase in the drying time or any loss of monomer by evaporation.

EXPERIMENTAL

With the exception of those described below the materials used were commercial products.

Glycerol monoallyl ether

This was prepared by the reaction of glycerol monochlorhydrin with allyl alcohol in the presence of potassium hydroxide by the method of Fairbourne and Gibson[26]: $n_D^{20}=1.4615$, $d_4^{20}=1.070$, -OH value=856 (theory=849); Dannenberg[27] reports $n_D^{20}=1.4627$, and $d_4^{20}=1.068$.

Glycerol diallyl ether

This was prepared by the reaction of epichlorhydrin and allyl alcohol in the presence of potassium hydroxide[28, 29]. It was purified by distillation: $n_D^{20}=1.4522$, $d_4^{20}=0.979$, -OH value=345 (theory=326).

Esters of glycerol diallyl ether

The acetate was prepared by esterification with acetic anhydride: B. pt. 92°C (1mm.), $n_D^{20}=1.4434$, saponification value 261 (theory 262). The crotonate was prepared by direct esterification at 180°C; B. pt. 130°C (5mm.), $n_D^{20}=1.4625$, saponification value 253 (theory=233).

The adipate had too low a vapour pressure to be distilled and the reaction product from the following experiment was used without further purification. Glycerol diallyl ether (2747g., 16.6 moles), adipic acid (1021g., 7 moles) and 300 mls. *Aromosol H*, a high-boiling aromatic hydrocarbon, were replaced in a three necked flask fitted with a stirrer, and a *Dean and Stark* water separator. The esterification was carried out under reflux at 210°-220°C for eleven hours under an atmosphere of nitrogen until 225 ml. (12.5 moles) of water was removed; the acid value of the product was 32. After treatment with carbon black followed by filtration, the excess glyceryl diallyl ether was removed by vacuum distillation to give 2860g. of product. Viscosity=29 c/poises at 23°C, $n_{20}^{D}=1.471$. The pyromellitate was prepared similarly.

A highly purified adipate of glycerol diallyl ether was also prepared, but since this gave similar results to the product obtained by the method described above, all the work in the paper was carried out on the less pure product.

Isosorbide

Isosorbide, 1.4, 2.6 dianhydro sorbitol, was prepared by the dehydration of sorbitol with 0.5 per cent H_2SO_4 catalyst, using toluene as entraining agent[30]. After distillation and recrystallisation from ethyl acetate, the material was obtained as a solid, m. pt. 61-62°C.

Transcyclohexane 1.2 *diol*

This was prepared by the reaction of cyclohexene with performic acid[31].

PHYSICAL TESTS

The films were applied on glass by means of film applicators (Sheen Instruments Limited). The hardness was measured by means of a *Sward Hardness Rocker Tester*, and scratch and mar-resistance were judged subjectively. Peak exotherms were determined on 20g. samples of the polyester, initiated with 0.1 per cent cobalt naphthenate (calculated as cobalt) and 2.4 per cent of methyl ethyl ketone peroxide. The samples were placed in an insulated glass tube 15 cm. × 2.5 cm. diameter, and the peak temperature measured by means of a thermometer placed centrally in the tube. The highest temperature reached, less the room temperature, was recorded as the peak exotherm.

ACKNOWLEDGMENTS

The authors thank the Directors of Howards of Ilford Limited for granting permission to publish this paper.

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[*Received* 6 *October*, 1960

DISCUSSION

MR. C. G. STRAIN asked whether the lecturer would give a more precise definition of the term "drying" as used in the lecture.

DR. R. J. WICKER replied that the term implied touch-dry as used by the paint chemist.

MR. R. W. HALL inquired if the use of acetoxime was effective in increasing the pot-life of systems in which the allyl ether group was built into the polyester.

DR. WICKER replied that it would be as effective as those systems in which the allyl ether group was present in the monomer.

MR. M. R. HOLT asked whether experiments had been carried out using combinations of glycerol diallyl ether adipate and styrene as the monomer.

MR. V. F. JENKINS replied that since allyl ethers and styrene will not co-polymerise, they tended to compete for the fumarate groups of the polyester. This resulted in either over-plasticisation by unpolymerised allyl ether or loss by evaporation of styrene. A carefully balanced system was desirable.

MR. C. H. MORRIS asked the authors to comment on the relative reaction rates of formation of the low-molecular weight oxygenated polystyrene believed to be responsible for air-inhibition and oxidation of the unsaturation of the allyl ether. He thought that the former reaction occurred almost instantaneously whilst the latter took a much longer time.

DR. WICKER stated that with a built-in system containing styrene, the presence of styrene peroxide on the surface would not prevent the surface drying of the allyl ether group as there would be an unlimited air supply. The allyl ether group drying reaction was a slower reaction than the reaction of oxygen with styrene but was continuing all the time and after five hours the film was dry.

DR. E. M. WILKINSON inquired how efficiently the allyl groups were used in cross-linking with glycerol diallyl ether adipate, and whether, if there was residual unsaturation, this was detrimental to the ageing of the polyester.

DR. WICKER replied that he did not know how efficiently the allyl ether groups were used, but accelerated weathering tests had shown no difference between the allyl ether type of polyester and the conventional styrene type.

MR. K. W. GREEN said that a drying time of seven to eight-and-a-half hours was most impractical from the point of view of the furniture manufacturer and suggested that the pyromellitate esters would have been worth further investigation despite the higher cost.

DR. WICKER explained that the object of the work was to produce a system which was not subject to air-inhibition and without increased drying time.

MR. A. MOTT suggested that laboratory and factory conditions were very different, as force-drying systems were probably used in a factory. By suitably increasing the concentration, it was probably possible to cut the air-drying time to one hour but not, as yet, to fifteen minutes.

MR. GREEN said that the pyromellitate with the faster drying time had been rejected on the grounds of price, but this might be inconsiderable compared with the cost to the smaller furniture manufacturer of installing force-drying equipment or the necessity for isolating large storage areas for air-drying purposes with the inevitable problem of dirt pick-up on the wet film. He thought that a quicker drying material with gloss from the gun without the need to face down the top surface should justify the extra cost per gallon.

DR. WICKER agreed that this was an interesting point of view and would be investigated.

Dr. L. A. O'Neill asked how much of the drying of the film was due to the copolymerisation reaction and how much to oxidation, and what would result if a saturated polyester was used in combination with an allyl ether.

Dr. Wicker replied that although a dry, hard film would be produced it would not be solvent-resistant.

Mr. Jenkins added that saturated polyester/allyl ether combinations gave films with a drying time of about twenty-four hours whereas the use of unsaturated polyesters reduced this to five to six hours. It therefore seemed that the unsaturated groups in the polyester also played some part in the air-drying mechanism at the surface.

Mr. Holt asked whether there was any information as to the shelf-life without promoter or catalyst.

Mr. Jenkins replied that there was not much experience of this point as yet, but samples had shown little change in two months.

Mr. G. E. J. Reynolds inquired as to the possible cost of producing glycerol diallyl ether adipate.

Dr. Wicker suggested that although allyl compounds were fairly expensive at present, owing to small demand, they could presumably become much cheaper since certain basic allyl compounds occurred as intermediates in the synthetic glycerol process.

Mr. N. R. Fisk asked whether isosorbide would be available in quantities for evaluation.

Dr. Wicker said that it was intended to make this product available very shortly.

Mr. K. H. Arbuckle inquired whether dimeric or trimeric styrene, which would have a slower evaporation rate than styrene, would be expected to show advantages also with regard to inhibition of drying.

Dr. Wicker replied that such compounds would be fairly expensive and would not be expected to have air-drying properties as all vinyl monomers so far known were subject to air-inhibition.

Mr. J. D. Lewis asked whether there was any information as to film inflammability on wood, and whether the materials were in any way toxic.

Dr. Wicker said that there was no knowledge on either of these points, but that he would not expect any difficulties from the toxicity angle.

Reviews

CHEMICAL PROCESSING NOMOGRAPHS

Edited by D. S. DAVIS. New York: Chemical Publishing Company, 1960. Pp. 255. Price $12.00.

The nomograph, aptly described as the poor man's computer, is well known as a valuable tool, but it is not as widely used in processing calculations as might be expected. If this new collection of 165 nomographs contributes to a greater use in industry then this book serves a valuable purpose.

Dale S. Davis, lecturer in chemical engineering in the University of Alabama, who is an authority on the subject, has produced this book in collaboration with the editor of *Chemical Processing* and a team of experienced engineers. The book is aimed at more aspects of the practice of chemistry and engineering than a previous collection and achieves a nice balance of helping many branches of these sciences, yet gives good value to the specialist.

A detailed knowledge of the theory of nomographs is not expected, though the explanatory first chapter will stimulate the future enthusiast to read further. An adequate list of references assists this purpose. The lazy and impatient will skip this chapter, but they will still get value from those nomographs presented in subsequent chapters. The nomographs vary from the simple, such as vapour pressure against temperature, through reciprocal, multiple and sum relationships, to several stage calculations, such as insulation requirements with seven or more variables.

For those with an engineering leaning there are chapters covering heat transfer, fluid flow, vapour pressure and pressure-volume temperature variables. Chapters on solubility, specific gravity, viscosity and humidity will more concern the chemist. Costing data and problems such as optimum sizing of equipment are considered. A chapter covering a field of particular interest to the editor, pulp mill calculations, illustrates a special field. The paint specialist is not overlooked in a miscellaneous chapter with a nomograph for calculating alkyd resin formulations, though many of the other graphs are also of use in this field. Mathematical problems such as computing standard deviations and solving equations are included.

The book is well set out in readable print. The size, 6 in. × 9 in., permits the necessary accuracy, but at some expense in time in keeping the surface of the book flat. Again, the wide range of problems covered will mean that many different people will wish to retain the copy available in their library. However, the real purpose of this book is to persuade and encourage those interested to make their own nomographs. This book should go a long way to achieving this worthwhile object.

D. P. DODGSON.

METALLIZING OF PLASTICS

By H. NARCUS. New York: Reinhold Publishing Corporation, 1960. Pp. x+208. Price 44s.

This is the latest addition to the range of the Reinhold Plastics Applications series, being the sixteenth and the first to be devoted solely to production methods rather than to materials. In his preface, Dr. Narcus claims that the purpose of the book is to present the several methods for metallising of which he has had personal experience, and which have actually been used in production at his factory. It is suggested that the book offers a practical and complete summary of the methods for metallising non-conductors, without being in any way a mere bibliography. The author deals with his subject from the angle of the practical user rather than from the theoretical basis of the processes described. Chemical deposition by reduction of suitable metallic salt solutions includes the *Electroless* copper process developed by the author. The vacuum-metallising process, in accordance with its importance, accounts for approximately 30 per cent of the book and the practical detail given, including the application methods for the base coat and protective lacquer, is comprehensive. Unfortunately, the author does not suggest the types of surface coatings suitable for these purposes, but states that the choice of solvent system and resin combination is more critical than when formulating conventional coatings. A list of (American) suppliers of suitable coatings is included.

The fundamental differences in principle of the cathode sputtering and vacuum-metallising methods are discussed and the special commercial application of the former is emphasised in Chapter 4. The silver spray method using a two-nozzle spray gun is fully described in Chapter 5, and here the author stresses the necessity for cleaning, "wetting out" and sensitising the base lacquer coat. Electro-deposition is considered in Chapter 6 and particularised in the production of printed circuitry. Amongst the miscellaneous methods discussed in Chapter 7, painting methods are reputed to have given excellent results, although they would appear to be crude and even obsolete when compared with the methods discussed previously. Conductive paints based on copper and silver powders are described and some basic formulations suitable for application to various surfaces are suggested. Metal spraying and "gas-plating", a process dependent on the pyrolysis of such materials as metal carbonyls, are also discussed in this chapter. There is also a chapter devoted to the characteristics and testing of metallic deposits, which includes details of tests for adhesion, thickness, porosity, conductivity and corrosion resistance. The need for special treatment of such plastics as polyethylene and polypropylene is stressed in the last chapter, which is entitled "Recent developments and future potential uses for metallized plastics", and reference is made to the Kreidl and Kritchever patents as well as to the less popular methods, such as electron bombardment or chemical treatment.

The book is well illustrated both by photographs of equipment and excellent line drawings, and all the processes described are fully discussed in relation to their advantages and disadvantages. References in the text are to a bibliography placed at the end of the book rather than in the individual chapters. The comprehensive index which closes the book leaves a little to be desired for, although elaborate cross-referencing has been attempted, there are some bad references, *i.e.* "Primer coatings, formulations for" refers to page 36, a page partially devoted to a photograph of a ball-burnishing process and partially to deposited films of gold, platinum, *etc.*, instead of to page 143. Similarly, printed circuitry in the index refers to a single mention of the copper *Electroless* process on page 27, whilst pages 126-9 deal much more fully with the subject and there is an earlier illustration on page 9. Printing and general lay-out of the book are good, although the appearance of table 1-1 on pages 2 and 3 is a little surprising when reference to it is delayed until page 5.

These slight criticisms apart, it can be said that the book fulfils the claims made by the author and can be recommended as a critical, detailed survey of the practical methods involved in the coating of plastics with metallic films for either decorative or industrial uses.

C. R. PYE.

AMINO RESINS

By J. F. BLAIS, Chemical Consultant, New York: Reinhold Publishing Corporation; London: Chapman & Hall, 1959. Pp. 215 and Index. Price 40s.

The book is one of a series of twenty-four books prepared to cover various plastic materials and, in conformity with the theme of the series, this present volume is concerned mainly with the applications of amino resins. It is not of an advanced technical character and can be understood by personnel other than research chemists. It is, however, limited to American development and usage, and only to amino resins which have reached commercial importance. The main types considered are urea resins and butylated modifications, melamine resins and butylated modifications, ethylene urea condensates and the butylated guanamine resins. The chemistry of the formation of the resins, with special reference to the relation between the formulation of the special types required and their particular applications, is reviewed in general terms, giving the main reactions which are involved in producing the resins concerned. No particular attempt is made to discuss the fundamentals of the reactions involved, the main reaction mechanism only being referred to, and the principal resins for particular types of application described. These applications are in the following main categories, which are dealt with subsequently in individual chapters:

Compression-moulding applications.
Adhesive applications.
Laminating applications.
Textile applications.
Paper-treatment applications.
Surface-coating applications.

The book only devotes seventeen pages out of a total of 215 pages to the subject of the use of amino resins in surface-coating applications, and in this description reference is only made essentially to the use of alkylated urea, melamine and benzoguanamine resins and particularly their butylated derivatives in stoving enamels.

No reference is made to the newer applications of the water-soluble amino resins in water-thinned stoving enamels. Nor is any reference made to the considerable use being made now of both urea- and melamine-type resins in catalysed lacquers of both clear and pigmented types, for use particularly in wood finishing.

In this way, this book is not up to date in this particular field as commercial application in these other ways, in this country, is now well established and there is the probability of their further growth by comparison with other wood-finishing lacquers. To the reviewer it seems a pity that the book, in this particular field, which interests technologists in the surface-coating field, should be deficient in this respect, and one is led to conjecture whether in the other technological applications, which are described in other chapters, there may be similar lack of attention to the latest developments in the particular fields of application described.

It may be that the deficiency mentioned arises from too much dependence by the author on technical information available from manufacturers of the resins concerned, and thereby has missed some of the more recent applications in the surface-coating field which these manufacturers might still regard as in an advanced development stage. It is significant that no reference to published work is given for this section on surface coatings. On the other hand, the author has made it clear, and it is the intention of the book as one of the series of volumes involved, to deal principally with the applications of proved commercial resins.

The major part of the book deals with the application of amino resins in the fields detailed above, other than surface-coating applications. In this respect the book presents a readily readable review of the applications of the various modifications of amino resins suitable for the fields of technology described. There are quite a number of illustrations, both reproductions of photographs and diagrams, to illustrate some of the plant used in the various processes referred to in the text, and some illustrations of finished products, particularly in the moulding applications section, which do not appear to serve any very particular purpose other than general information. It also refers to quite a number of the problems involved in the production of certain finished materials, and the particular manufacturing techniques and choice of resins to give special results.

References to original work are only given at the ends of the sections on
"Chemistry of Amino Resins" (latest reference 1957),
"Textile Applications" (latest reference 1958),
"Paper Treatment Applications" (most recent reference 1959).

The index, which is clearly set out, was found to be accurate with the text for the items which were tested. The book is printed on a good quality paper, the type used makes it easy to read, and the text is singularly free of type-setting errors. Being an American book, there are some differences at times of expression and spelling, such as "thermic stable" for "thermally stable" or "heat stable" on p. 148 and "salable" for "saleable" in lines 8 and 31 on p. 137.

The book represents a worth-while purchase to anyone wishing to have a readily readable review of the applications of amino resins in American technology, but would only act as an introduction to anyone wishing to specialise in, or seriously study, any one of the particular applications described. It would also be useful as a ready reference book for technical libraries.

F. C. RUZICKA.

PHYSICAL CHEMISTRY OF SURFACES

By A. W. ADAMSON. London: Interscience Publishers Ltd., 1960. Pp. 629. Price 96s.

This book, from its title and contents, invites comparison with Professor Adam's *The Physics and Chemistry of Surfaces.* The field is similar, and the book is also written as a graduate-level text. It is indeed a worthy successor to Adam.

The topics covered include capillary phenomena, surface films on liquids, wetting and detergency, emulsions, electrical phenomena at interfaces, and a large section is concerned with the surfaces of solids, including adsorption of gases and vapours, chemisorption, and adsorption from solution. Chapters of particular interest deal with liquid-gas interfaces from a thermodynamic aspect, and long-range interaction forces between molecules. Rather surprisingly, surface agents are dismissed in two pages; there are, however, several existing texts on this vast subject.

A particular feature of the book is the presentation, in some detail, of the theoretical derivation of the topics under discussion. This, together with the worked examples and the problems at the ends of some of the earlier chapters, makes for a greater appreciation of the quantitative aspects of surface chemistry.

The literature is well covered on the particular subjects with which the reviewer is most familiar, some references up to 1959 being included in the text. One gains an overall impression of thoroughness and care in the selection and presentation of material, and the book will be of considerable value to all those in the paint and pigment industries who are concerned with surface activity.

The book is well produced, although perhaps the subject index could be improved, and it is unfortunate that its price will restrict its purchase, in the main, to institutions and libraries. It is, however, a valuable book, as a source both of published information and of inspiration.

V. T. CROWL.

POLYSTYRENE

By W. C. TEACH and G. C. KIESSLING. Reinhold Publishing Corporation, 1960. Pp. ix+176, cr 8vo. Price 40s.

This is an introduction to the chemistry, properties, manufacture and uses of that versatile material, polystyrene, and ranges from the early days of storax distillation to the ubiquitous stiletto heel. The basic physico-chemistry of the polymerisation processes and the dependence of physical properties on molecular structure are clearly outlined. Short chapters on commercial production of moulding powders and on fabrication by injection, extrusion, sheet forming *etc*., with descriptions of the plant and principles involved, are followed by a review of the myriad uses in the world around us. Polystyrene foam, the newest star in the galaxy, is discussed in some detail. A chapter on specialities, including a mention of surface-coating applications, and an assessment of future prospects complete this most readable book.

It is an attractive volume. The paper is of good quality, the binding is handsome, and illustrations and line drawings are frequent. Occasional references to original work and a short bibliography provide the reader with fuller discussions of the work. The index is adequate. Only one error was detected—"perfluoroethane and ethylene" are not solvents for the polymer, as stated (p. 153). Presumably perchloroethane and perchloroethylene are the solvents in question.

The intention was to provide an introductory book for the practical man, the engineer, designer and student, in order to show what may and, every bit as important, what may not, be done with this polymer. It is also intended to broaden the outlook of the chemist, on the one hand, and the fabricator on the other. It will, no doubt, be successful. It is clear, concise and eminently readable. However, the reviewer cannot help feeling that it is rather superficial, and that one pays rather dearly for it. Most of the information is available in manufacturers' data booklets, in *Modern Plastics Encyclopaedia* and in other books which should already be in the library of the fabricator or technician. He would prefer to see it at a more workaday price, though doubtless in more workaday garb. Chacun à son goût.

W. HOPKIN.

HISTORY OF SEED CRUSHING IN GREAT BRITAIN

By HAROLD W. BRACE. London: Land Books Limited, 1960. Pp. 172. Price 30s.

The revolutionary changes which have taken place in the structure of industry and its processes since the beginning of the twentieth century are proceeding with an ever-increasing momentum, and there is a real danger that much information of historical interest may soon be lost to us. Those who have witnessed the more spectacular developments are now approaching the age of retirement, and they alone can paint a reliable picture of the industrial scene during this period. Any historical records now being compiled are, therefore, worthy of our attention.

The seed crushing industry is, of course, a very ancient one that has been practised in this country for at least 600 years. Very little has been published about it, however, and the present book is of particular interest since it contains a wealth of information that would be extremely difficult to find elsewhere, written by one with the requisite practical experience. Slightly more than half is devoted to a narrative describing the development of the industry from its beginnings up to the present time, and the remainder consists of three appendices giving historical details of all the known oil mills in the country, a list of the relevant patents and a short bibliography.

Those who are particularly interested in seed crushing, or in industrial history generally, will find the book extremely useful. Users of vegetable oils wishing to be informed of the processes of seed crushing will find them described in greater detail than is usual elsewhere.

The same cannot be said of solvent extraction processes, however, since many of the continuous systems in use outside the United Kingdom are not described.

M. R. MILLS.

Correspondence

O.C.C.A. TECHNICAL EXHIBITION

Mr. Fisk's letter in your October issue makes a point which is perfectly valid if his premise is right. The premise is that the exhibition is an exhibition of new products and new research; in this, of course, he has the support of the officially declared objective of the Exhibition Committee.

But we should be deluding ourselves if we were to imagine that eighty odd exhibiting firms have ever, in the past, turned out such original work each year. They certainly make a very good appearance of having done so, but the unwritten agreement to accept this appearance of doing so is not conducive to steady progress. It means for one thing that long-term systematic research in the supplier industries runs the risk of being squeezed out by the need to "do something for the O.C.C.A. Exhibition". It encourages that curse of the paint industry, the fleeting fashion in formulation. It puts at a disadvantage those suppliers whose contributions are likely to be large ones developed over long periods. It grossly embarrasses those suppliers who are concerned with staple and stable raw materials. It encourages the flashy, get-rich-quick supplier. It provides the younger technologists with an entirely illusory impression that the industry is in a state of rapid progress. It exalts, in fact, the transitory over the permanent, the trivial over the important, and the trite over the profound.

It will be best to acknowledge that the exhibition performs most useful functions apart from its declared ones. It enables technologists to meet one another and to meet their suppliers or possible suppliers in a relaxed atmosphere. It enables technologists to obtain, even if cursorily, a visual impression of the range of products available to them. Most important of all, it enables suppliers to meet technologists directly; it is perhaps not sufficiently realised that in many of the less progressive firms the buyer determines the technical and manufacturing policy of the company by restricting his purchases to a favoured circle, and preventing the technologists from having access to possible new materials.

All this can be achieved without laying upon the exhibitors a burden greater than they can honestly bear. So long as the exhibition retains its technical flavour then so long will it form a satisfactory technological meeting ground. So, it might as well be held every year; it might as well be allowed to become as big as it wishes. If this means that unprogressive companies start to exhibit, this will be shown up by comparison with competitors much more clearly than if there is a spurious atmosphere of technical progress.

Bowmans Chemicals Ltd.,
Moss Bank,
Widnes, Lancs.

M. H. M. ARNOLD.
1 November, 1960.

CO-OPERATIVE RESEARCH PROJECT BY THE NEWCASTLE SECTION

On page 810 of the November issue of the *Journal* under Table I, the description of the driers should read:—

$$\left.\begin{array}{l}\text{0.05 per cent cobalt}\\ \text{0.5 per cent lead}\end{array}\right\}\text{metal (as nepthanate) on binder}$$

EDITORIAL

—11,000 Miles Apart—

A hundred years ago the Maori wars were starting and the government of New Zealand was still in Auckland. A lot of Maori gum has been run since then and a lot of useful things have been made out of the cases in which the gum used to come to this country. We wonder if those same cases are still used? That shows how long it is since we had anything to do with gum-running. These thoughts have been prompted by the news of the growth of this Association in New Zealand to the stage where we welcome two sections from the original one. The New Zealand Section has now become the Auckland and the Wellington Sections of the Association. Does anyone make paint on the South Island?

While welcoming new sections we do not wish to pass unnoticed an event which in the context may have some significance in showing that the Association in the United Kingdom can gain from experience in the Commonwealth. Elsewhere in this *Journal* is mentioned the formation, for the first time in this country, of a branch of a local section—the Southern Branch of the London Section, in the Southampton and Portsmouth area. It is farther from Auckland to Wellington or from Perth to Adelaide than from Southampton to London, but there are plenty of common problems, and we would like to think that in the recognition of this lies future strength for the Association.

COMMENT

We have heard (*J.O.C.C.A.*, 43, 2, 124) from Dr. Stead of some thoughts on future trends in organic pigments, and it is strange to think retrospectively of the effect on technology of the large-scale production of phthalocyanine blue. That one pigment has possibly done as much as anything to change the attitude of a formulator to cost per pound of a raw material, and to encourage the use of pigments which at one time would have been regarded as prohibitively expensive but which are now fully accepted for technical reasons. We feel sure that developments of this kind will have been accelerated by the recent opening of a new laboratory block at JAMES ANDERSON & CO. (COLOURS) LTD. at Paisley. The laboratories include provision for research and development and we admired the blend of modern and traditional in the materials and methods of construction. The exterior facing of Westmorland slate provides a pleasant and practical relief from the surfeit of curtain walling in modern buildings.

The use of films for advertising and educational purposes is so well known as to need no comment. We have seen recently, however, a film concerned with the design and manufacture of mixing machinery, produced for STOCKDALE ENGINEERING LTD. under their new guise as LIGHTNIN MIXERS LTD. The film, "Fluid Mixing", considers process design, power consumption and mechanical design, and probably contains both interest and information for anyone of the increasing numbers of people who think about high-speed mixing.

When presenting the awards for the research essay competition organised by BUTTERWORTHS PUBLICATIONS LTD., Viscount Slim commented on the increase in recent years in the rather uneasy awe with which the average citizen regards scientists and things scientific. This could contribute to the danger of intellectual arrogance which is sometimes met with in scientific circles, but not, Viscount Slim rightly added, among leading scientists who usually have the humanity and simplicity which goes with true learning. Scientific advances made it progressively more difficult to explain to the humanist exactly what was happening and why, and this was aggravated by the natural incapability of scientists to speak and write clear and comprehensive English. Viscount Slim expressed his faith in men as individuals, not as professionals, and emphasised the fact that no profession has the complete answer to governing. There is, or should be, no division of learning.

Our attention has been drawn to the publication by the INTERNATIONAL COUNCIL OF SCIENTIFIC UNIONS (I.C.S.U.) of a quarterly journal. The Council comprises forty-eight national members from scientific academy or national research council and thirteen members from various International Scientific Unions, including that for Pure and Applied Chemistry. Apart from presenting papers of international scientific interest, the I.C.S.U. *Review* covers the work of the various Unions and is published by the Elsevier Publishing Co.

We have received from FATOILS LTD. a reprint of a paper published in Sweden on "Developments Within the Tall Oil Industry", by W. Krohnstad. The paper deals with the availability, extraction and refining of the oil and its fractional distillation into fatty acid, resin and residues. We assume that copies may be obtained from FATOILS LTD.

We have recently seen mention of an American process whereby vinyl plastisol is applied to primed metal sheet or strip in a continuous process. We regard this as an important potential advance over the now conventional systems of laminating vinyl sheet to metal and look forward to hearing more.

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.)*

It has been announced that British Celanese Limited will produce nitration grades of benzene and toluene, and 3° xylene at a new Aromatics Condensate Separation Unit at its chemical works at Spondon, Derbyshire. The unit is being designed and will be erected by the Chemical Engineering Division of the A.P.V. Company, and will incorporate a cyclopentadiene recovery unit operating according to British Celanese process design.

F. W. Berk & Co. Limited has sent a copy of *Synthese Guide No. 10*, which describes *Setanon 600*, a cyclohexanone resin for use in flexographic and rotogravure printing inks and nitrocellulose lacquers. The company states that *Setanon 600* is a water-white resin having a softening point of 105°-110°C (ring and ball), with considerably better colour fastness.

Marchon Products Limited has announced the construction at its Whitehaven factory of a plant to manufacture sulphamic acid (amido sulphonic acid) with a capacity more than sufficient to meet the whole of the United Kingdom's known requirements. The company is also considering the manufacture of ammonium sulphamate.

The ABCO Chemical Company announces the availability of 1,2,3,4 tetracarboxybutane (T.C.B.), which is a polyfunctional acid with a melting point of 187°-191°C, soluble in water and most polar solvents, but insoluble in hydrocarbons. T.C.B. is a highly reactive acid which is useful for the preparation of esters having potential applications as plasticisers and high-temperature lubricants. The potential applications of T.C.B. in the coating industry include preparation of alkyd resins, polyesters which may be employed in resin manufacture and in polyurethane formulations. The acid or its anhydride may be important as an epoxy-curing agent.

The Dyestuffs Committee of the Association of British Chemical Manufacturers has appointed Mr. Harold Blackshaw, who retired after forty years' service with Imperial Chemical Industries Ltd. (Dyestuffs Division) and its predecessors, to succeed Mr. F. Scholefield as Technical Dyestuffs Adviser. Mr. Blackshaw is well known for his work in connection with the Second Colour Index, which was acknowledged with the award of the Gold Medal of the Society of Dyers and Colourists.

Short & Mason Ltd. are the sole agents in this country for the *Rheomat 15* rotational viscometer manufactured by Contraves A.G. of Zurich. Instead of the usual single-point measurement obtained with many types of viscometers, *Rheomat 15* is claimed to yield data on the flow properties of Newtonian, plastic, thixotropic, dilatant and pseudoplastic materials.

Owing to lack of space to carry out their expansion programme, VINYL PRODUCTS LTD. and VINATEX LTD., members of the REICHHOLD CHEMICALS LTD. group of companies, will be moving in two to three years' time from the site they share at Carshalton, Surrey. The new works, complete with extensive offices and research and development laboratories, will be built on the 100-acre site at Brockhampton, near Havant, Hants.

The following publications have been received: *Resinotes* from CRAY VALLEY PRODUCTS; *Maydown Works* from DUPONT; *Plastics Today* and *Silicones* from I.C.I.; *Catalyst 5* from SHELL, and *Synthese Guide 10* from KUNTHARSFABRICK SYNTHESE, Holland, and *Beetle Bulletin* from B.I.P. Ltd.

AMALGAMATED OXIDES (1939) LTD. has formed a joint subsidiary with STOLBERGER ZINK A.G. of Aachen, having the title STOLBERGER ZINCOLI G.m.b.H. für Zinkstaub. The new company will produce *Zincoli* Superfine Zinc dust, which was developed by AMALGAMATED OXIDES some six years ago and has found increasing use with zinc rich paints and inorganic zinc dust paints. Plant has been erected at the Munsterbusch works of the German company, and production has begun.

It has been announced that COWAN BROTHERS (STRATFORD) LTD. have joined the JOHNSON MATTHEY group of companies. The company, which specialises in the production of inorganic colours, pigment dyestuffs and lakes, will continue its manufacturing and trading as before, and will retain all its staff.

A new plant, with an annual capacity of 11,000 tons, for the bulk manufacture of *Propathene* (polypropylene), is now on stream at I.C.I. LTD's Wilton Works. It is stated that *Propathene* is the lightest in weight of all commercially available plastics, with a density of only 9.0 g./c.c.; it has a melting point of about 165°C and possesses a high degree of rigidity at room temperature, together with retention of useful strength up to 140°-150°C. Polypropylene offers a high resistance to oils, fats and greases.

Bristol Section

THE PHYSICS OF PAINT FILMS

The annual paint meeting of the Section was held jointly with the Birmingham Paint, Varnish and Lacquer Club at the Hawthorn's Hotel, Bristol, on 28 October, 1960. Unfortunately, owing to a clash of dates, only six members of the Club were able to be present, but after dining the company of thirty-six assembled to hear Mr. G. Phillips lecture on "The Physics of Paint Films".

In his paper, which was interspersed with some much appreciated dry humour, the lecturer discussed the problems of adhesion, tensile strength and moisture permeability as they applied to surface coatings. He described a method for determining the adhesion of paint films using the Hounsfield Tensometer, and showed how this property of a film was related to its degree of cure and moisture content. Similarly, with a small tensile testing machine, he demonstrated graphically the effect of rate of cure, moisture content and pigment volume concentration on this property of a film.

Finally, dealing with permeability and water absorption, Mr. Phillips suggested that the determination of the absorption isotherm of a paint film gave a better indication of its performance than did either of the two previously mentioned properties.

The discussion was opened by Mr. I. H. Phillips (Bristol) and a lively discussion followed in which Mr. J. Hitchin (Birmingham), Mr. D. Newton (Bristol), Mr. R. Woodbridge (Bristol) and Mr. K. Reiser participated. A vote of thanks was proposed by Mr. H. Clarke (Birmingham) and was unanimously endorsed.

D. N.

London Section

WATER-THINNABLE STOVING FINISHES

The third technical meeting of the session took place on 10 November, 1960, at Manson House, when, to a meeting to which members of the Plastics and Polymer Group of the Society of Chemical Industry had been invited, Mr. A. G. North, of Cray Valley Products Ltd., delivered a paper entitled "Water-Thinnable Stoving Finishes". The paper, which was an extension of that delivered to the Hull Section during the 1959-60 session, will be published in a later issue of the *Journal*. The paper was followed by a lively discussion, in which the following members took part: Mr. R. N. Wheeler, Dr. O. Liebermann, Mr. E. S. J. Fry, Mr. A. G. Robinson, Mr. W. J. Smith, Mr. G. E. C. Mercer, Dr. M. L. Ellinger, Dr. H. G. Rains, Mr. R. S. Colborne, Mr. E. Hammond, Mr. J. A. Handscomb and Mr. A. Gellman.

The discussion was terminated by a vote of thanks to the lecturer proposed by Dr. J. C. Hudson, who commented on the advantages to be gained by the use of water as the solvent for paints to be applied in enclosed areas. He suggested that if the types of resins described in the paper could be made air-drying, it would be a great step forward to solving the problem of the painting of, for example, the double bottom of ships. The importance of the subject of this paper and the interest of the industry in it was amply illustrated by the attendance, which taxed the capacity of the hall, and by the way in which the discussion maintained its continuity to a later hour than is usual at such meetings.

C. R. P.

Midlands Section

RECENT DEVELOPMENTS IN POLYESTER SURFACE COATINGS

The second meeting of the session was held in Wolverhampton on 21 October at Mander Bros. Ltd. As is customary at these meetings, the Chairman, Mr. N. H. Seymour, asked Mr. W. A. Edward, a past Midlands Chairman from Wolverhampton, to take the chair for the paper by Dr. Patheiger on "Recent Developments in Polyester Surface Coatings".

Dr. Patheiger's approach to the subject was essentially a practical one, the polyester lacquer system being discussed in detail. The factors considered in formulation were the advantages and disadvantages of wax and wax-free coatings, the constitution of the polyester and the importance of the purity of the styrene. Stability was improved by the addition of hydroquinone, or tertiary butyl catechol, and silicone oils helped the flow properties and pigment-wetting characteristics.

Consideration was also given to the peroxide catalysts, such as methyl ethyl ketone peroxide and cyclohexanone peroxide, and also to cobalt-type accelerators.

The surface to which the polyester coating is to be applied was important, and Dr. Patheiger referred especially to the effect of the water content of the wood and the inhibitive effect of phenolic-type dyes present in teak and rosewood. The sealing of wood surfaces was normally satisfactory when a polyurethane coating was applied.

Application methods were also discussed, such as spraying and curtain coating, together with the final treatments of sanding and polishing. Dr. Patheiger concluded his lecture with a colour film illustrating very clearly the points raised during the paper. The vote of thanks was proposed by Mr. W. A. Edwards.

R. D. C.

Newcastle Section

POLYSULPHIDE COATINGS AND SEALANTS

The second meeting of the present session was held in the Royal Turks Head Hotel, Newcastle upon Tyne, on 3 November, when Mr. A. D. Hibberd was in the chair. There was a large attendance of members and visitors to hear Mr. A. A. Duell, erstwhile Secretary of the Section, read a paper entitled "Polysulphide Coatings and Sealants".

Mr. Duell reviewed the history of the polysulphide polymers culminating in the production of the first liquid polymer in 1943. The preparation and physical properties of typical polymers were discussed, and the mechanism of their cure with oxidising agents, such as lead peroxide, was illustrated. Indication was made of the marked effect of temperature and humidity on the rate of cure, and methods of adjusting cure rates by means of retarders were discussed. Compounding of the polymers for specific properties was reviewed, and the effect of various fillers at different concentrations on the final properties of the cured polymers was shown. For various performance requirements, plasticisers and adhesion promotion agents were added, the choice of these being influenced by the end use envisaged for the compounded polymer.

The ability of the compounded materials to be applied in liquid or paste form and afterwards cured to a rubber *in situ*, the rubbers having excellent solvent and weather resistance, was utilised in a variety of industries. An important use of compounded liquid polymers was in the sealing of integral fuel tanks and pressure cabins in aircraft, and typical methods of sealing aircraft were discussed and applications illustrated by slides. The properties of sealants designed for aircraft use were shown, and a comparison was given with the properties of lead peroxide-cured sealants and sealants cured with soluble dichromates. Examples of the use of the compounds in the electronics, marine and constructional industries were illustrated by specimens. Finally, a film was shown which illustrated many of the applications mentioned in the course of the lecture.

In the discussion which followed, Mr. R. Wilson, Mr. E. L. Farrow and Dr. A. I. Escolme expressed interest in the mechanism of curing, particularly with epoxide resins, Mr. N. Ellis asked about possible uses as contact resins and Mr. G. W. Loosemore and the Chairman commented on the effect of fillers on the extensibility of the cured resin. Others participating in the discussion were Mr. W. P. Jenkins, Mr. J. G. N. Smith and Mr. J. A. Willey. Dr. Escolme, in proposing a vote of thanks to Mr. Duell, mentioned his personal criterion for the maximum duration of a lecture; this will probably be followed with interest by members at future meetings.

G. W. D.

New South Wales Section

Pigments for High-Grade Industrial Finishes

The October meeting of the Section was attended by eighty-five persons who were present at the M.L.C. Building Theatrette to hear Mr. D. M. Varley, of Imperial Chemical Industries, Manchester, England, deliver a paper entitled "Pigments for High-Grade Industrial Finishes". During his lecture Mr. Varley showed slides illustrating the excellent light fastness of the newer vat pigment types anthrapyrimidide, *CI 68420 Vat Yellow 20*, and flavanthrone, *CI 70600 Vat Yellow No. 1*. Both these pigments, he indicated, were suitable for high-quality stoving enamels and nitrocellulose lacquers as used in automobile finishes, and also for high-grade industrial stoving and decorative paints.

Mr. Varley also discussed and illustrated by slides the *Supra Chromes*. This range of chrome colours, he said, represented an important advance in the development of chrome pigments of improved durability. Whereas lead chromes were not deemed suitable for high-grade industrial work prior to these recent advances, owing to their darkening characteristics on exposure to light, the *Supra Chromes* were regarded as being suitable for such finishes, and were actually now finding a use in automotive lacquers and enamels.

Question time was lively and indicated the general interest shown in the lecture. Particularly active at question time was Mr. L. Williamson, himself a "colour man". The vote of thanks was proposed by Mr. A. Wilson.

L. R. R.

Scottish Section

Paint Industry in Other Countries

In the presence of a large and representative gathering of the Section, the new session was inaugurated on 13 October, 1960, at More's Hotel, Glasgow. The Chairman, Mr. E. A. Bullions, welcomed the members and friends present and expressed the hope that the Section would have a successful and happy year. It is now an established feature of the Scottish Section that the new Chairman has the honour and privilege of delivering the first lecture, and for this purpose Dr. D. M. Stead, immediate past Chairman, assumed the chair in order to allow Mr. Bullions to deliver his paper, an event eagerly anticipated by the members.

The lecturer commenced by stating that the paper about to be delivered was of a general nature and could be briefly enumerated under four well-known broad sections: raw materials, methods of manufacture, types of paint and laboratory control, as used and practised in the following countries: Canada, France, India, Norway, Pakistan, Spain and the United States. He then dealt with each country individually, making reference to the locality in which the industry was concentrated and gave details of the lay-out of factory, laboratory and office buildings. On the engrossing and ever topical subject of raw materials for the paint industry, the lecturer produced a wealth of well-tabulated information and data. He reminded his audience of the fortunate position of some of the aforementioned countries in having access to native raw materials. Processing of materials to finished paints, types of mill employed, types of paint produced and laboratory control methods were all treated at length and with discernment. It appeared that in India the paint industry was now well established and that steady progress had been made since the formation of the Indian Paint Manufacturers' Association some eighteen or nineteen years ago. The industry in Pakistan was established as recently as 1949, but in the short period of a decade tremendous advances have been made and today paints were produced which can and do satisfy the highest specification standards.

Referring to Spain, the lecturer stated that paint manufacturers there had been severely handicapped by a government ban on the import of pigments and resins. Paint produced was of a poor quality and laboratory control was virtually unknown. In recent months these restrictions had been removed and the government was now taking steps to ensure that modern equipment would be installed; but it is doubtful whether any great improvement could be made until the industry has at its disposal an adequate number of trained technical personnel. France had 413 companies engaged in the manufacture of paints and varnishes. The total annual output of these materials, he said, was approximately 431,000 tons. White pigments, umbers, ochres *etc.*, synthetic resins and rosin were produced internally, but iron oxides, both natural and synthetic, were imported. The manufacturing techniques and the equipment employed were very similar to the British pattern. There appeared to be some confusion in the industry regarding the standardisation of methods of laboratory control and the maintenance of specification standards, but these points were at present being investigated.

Mr. Bullions next made reference to Norway, which has fourteen paint manufacturing companies producing approximately 30,000 tons of paint per annum. The production capacity exceeded the total current demand and those firms were seeking export markets for their products. It was interesting to note that dissolvers and ball mills were the main items of equipment in the production lines. It seemed that no steps had been taken to standardise shades on a national basis. Rutile titanium dioxide, zinc oxide and native white extenders were extensively used, but most coloured pigments, both organic and inorganic, had to be imported. Laboratory equipment was modern and a high standard of production control was maintained at all times. He then informed the audience that the United States had 1,500 companies manufacturing paints and varnishes, with total annual sales valued at some 1,600 million dollars.

Regarding raw materials, the pigments, extenders, resins, solvents, etc., used were comparable to those used by our own paint industry. Manufacturing processes followed a similar pattern, single-roll, twin-roll and ball mills being standard equipment. As would be expected in such a large self-sufficient economic unit, the range of paints manufactured was very extensive and all-embracing and appeared to be sufficient to meet all domestic demands. Laboratory control was adequate, but generally speaking raw materials were not subjected to the stringent tests that were accepted as normal routine in the United Kingdom.

Finally, the lecturer discussed the paint industry in Canada. There were 125 units producing a wide range of paints and varnishes. Raw materials, manufacturing techniques and laboratory control methods were similar to those employed in the United States.

At the request of Dr. Stead, acting Chairman, Mr. A. McLean proposed the vote of thanks. He complimented the lecturer on delivering such a comprehensive and informative paper in the short time at his disposal.

H. G.

West Riding Section

PLASTIC-COATED STEEL STRIP

The second technical meeting of the Section was held on 11 October, 1960, when Mr. W. Bullough, from B.I.S.R.A., gave a lecture on "Plastic-Coated Steel Strip".

Mr. Bullough gave a general review of the development of p.v.c. coated steel, mentioning several processes at home and abroad, but with particular reference to his own research at B.I.S.R.A. on *Plasteel.* The main features, he said, of the laminate were that it was a prefinished coated steel, which eliminated all need for painting after fabrication. It could be deep-drawn, roller-formed or lock-seamed without damage to the coating. The thermal insulation of the film, which could be obtained in a wide range of decorative finishes, including embossing, gave the surface a warm feel. The surface had good scratch abrasion and chemical resistance, together with the advantage of being easily washed.

The lecturer gave a detailed discussion on the method of manufacture of laminate steel strip. This, he said, was a continuous process and started with a pretreatment by phosphating to protect the back if only one side was to be laminated. Adhesive was applied by a roller, and the solvent dried off and then "cured" in an electrically heated oven. The cured strip covered with adhesive was coated with a plastic film in a nip roll and then coiled. By careful control of the laminating temperature quite heavily embossed films could be applied to the steel.

Although this material had several advantages over painted steel, development has been rather slow due to high initial overhead charges caused by small production runs, especially where orders were for small lots covering a wide range of colours, emboss finishes and film thicknesses, and the necessity for maintaining stocks disproportionately high in relation to the production runs. Losses in scrap too could be considerable in cutting laminate sheets to customers' ordered size.

After the lecture there was a lively discussion time, when Mr. Bullough answered many questions from the members. Mr. Griffin enquired what the maximum depth of draw of these p.v.c. coatings was in relation to the thickness of the steel strip. The lecturer replied that p.v.c. coatings might be drawn as far as the steel could be drawn providing that the adhesive used in the bonding process had a satisfactory bond strength. Mr. Nicholls then asked what the abrasion resistance was of those p.v.c. laminates and whether they were suitable for use on desk tops. Mr. Bullough stated that the abrasion resistance of the laminate would depend upon the hardness of the plastic selected; in general, the resistance to wear and abrasion could be regarded as being excellent. In reply to Dr. Hargreaves, who asked in what industrial application p.v.c. laminates were likely to be employed, the speaker said that p.v.c. laminates had a wide variety of colours and finish. Since the laminates might be formed, welded or bonded, the coated sheet might be used in the manufacture of household as well as industrial equipment. Where a metal to metal bond was required, the p.v.c. coating had to be cut away or removed in some way from these areas, and some novel ideas were being used for this purpose. Hence p.v.c. laminates had a wide field of application in both chemical and corrosion-resisting plant; they also had a decorative appeal. Office and home furniture might therefore be fabricated from p.v.c. laminates.

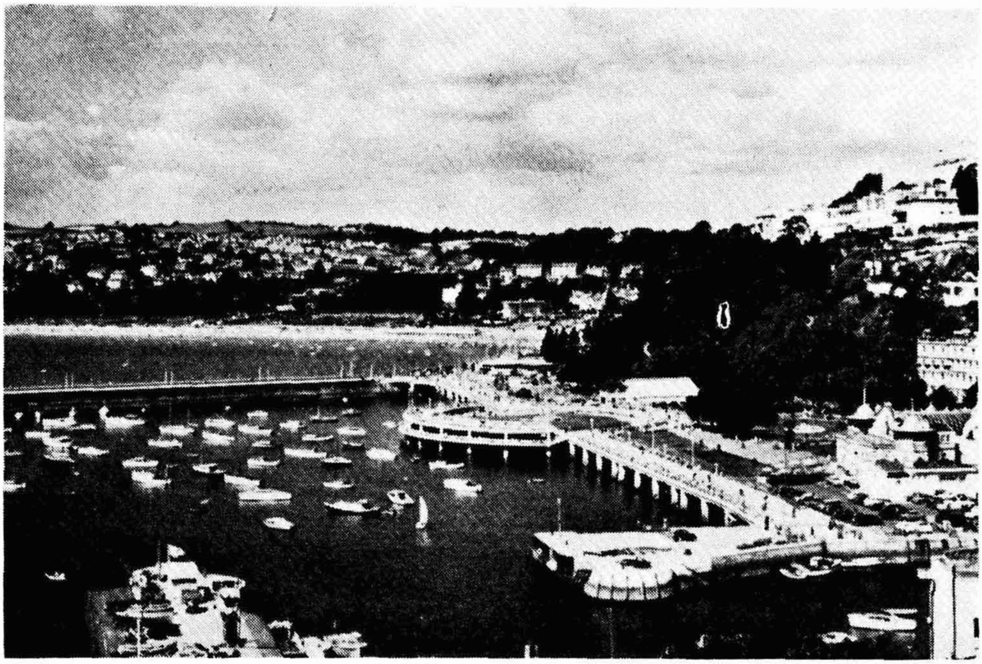
Mr. du Rieu remarked that one of the limiting factors of p.v.c., rather than p.v.c. coated steel, was "dirt retention", and asked whether anything had been done to eliminate this hindrance. Mr. Bullough considered that a number of embosses could be selected which were easy to clean. Other plastics were being evaluated—polyester resins were being used in the United States and polyethylene was being evaluated mainly for its chemical resistance. These materials were internally plasticised and dirt retention would be substantially reduced by the use of these resins. Mr. Anderson then continued the questioning by enquiring if it was possible to emboss on calender after the application of the p.v.c. sheeting. The speaker felt that this could be done if a plastisol were used, but generally speaking this would not offer any advantages over the p.v.c. sheeting owing to the necessity for two heating stages involved in curing the adhesive and gelling the plastisol. If a suitable adhesive was found which would enable both adhesive and plastisol to cure and gel at the same time, then this would be a more attractive proposition than the method which had been described.

Mr. Cockran said that it was common knowledge that one large steel company in Great Britain is building a plant for the application of a "one-coat" p.v.c. finish which would be applied to steel sheet, and asked whether a one-stage application of p.v.c. direct to metal had been attempted by B.I.S.R.A. The lecturer had not heard of a p.v.c. plastic which could be applied in this way. B.I.S.R.A. had had some success by using a plastisol without an adhesive, but this material could not be considered as having a satisfactory bond. Possibly by substituting a copolymer of p.v.c./p.v.a./ maleic acid for p.v.c. a better adhesion could be obtained. Vinyl lacquers for metal were prepared from p.v.c./p.v.a./maleic acid copolymers and these adhered quite well to steel. He was of the opinion that this was the basis for such one-coat p.v.c. coatings being used.

Mr. Cass wondered whether any tests on the resistance of p.v.c./adhesive/metal laminates to thermal shock had been attempted, to check whether any surface defects occurred. Mr. Bullough reported that such tests had been carried out at elevated temperatures, and so far no failures had appeared, nor were they expected to occur, if a thermosetting adhesive was used for bonding the laminate. Mr. Young, in conclusion, enquired what the general solvent, chemical and flame resistance was of p.v.c. laminates. The speaker replied that strong solvents, such as ketones and esters, would attack p.v.c. and dissolve out the plasticiser; p.v.c. was slightly swollen by alcohol. The flame resistance of p.v.c. laminates depended on the film thickness of the plastic sheet. If a thin film was considered, the heat would soon be conducted away through the metal from the area of contact. If a thick film were to be used, local overheating could cause decomposition of the p.v.c. at this centre.

L. J. W.

ASSOCIATION CONFERENCE, 1961



THE HARBOUR, TORQUAY

PHYSICS IN SURFACE COATINGS

The 1961 Conference of the Association will take place at Torquay from Tuesday, 30 May, to Saturday, 3 June. The majority of members will be accommodated in the Palace Hotel and the overflow will be housed in the Babbacombe Cliff, Osborne, Queens and Victoria Hotels. The Technical Sessions, under the general title *Physics in Surface Coatings*, will be held in the Palace Hotel. The papers are being arranged by the Honorary Research and Development Officer of the Association, Dr. J. B. Harrison, who has reported to the Council that the following is the programme for the presentation of the papers:

Wednesday, 31 May, 1961

In the chair: Dr. J. B. Harrison (Honorary Research and Development Officer).

Mr. G. Phillips (British Resin Products Ltd.), *The Physical Behaviour of Paint Films*.

Mr. C. C. Mill (P.A.T.R.A.), *The Behaviour of Printing Ink on Rollers*.

Mr. N. D. P. Smith, Mr. S. E. Orchard and Dr. A. J. Rhind Tutt (I.C.I. Ltd., Paints Division), *The Physics of Brush Marks*.

Thursday, 1 June, 1961

In the chair: Dr. L. Valentine (Director, Paint Research Station).

Dr. R. N. C. Strain (National Gas Turbine Establishment, Farnborough), *Solar Reflectivity of Paints*.

Mr. H. Williams (British Resin Products Ltd.), *Some Aspects of the Assessment of Emulsion Paint Films*.

Mr. F. G. Dunkley and Dr. D. P. Earp (British Railways, Research Department), *The Correlation of Service Behaviour with Observed Physical Characteristics of Air-Drying Paints for Structures*.

Friday, 2 June, 1961

In the chair: Dr. V. G. W. Harrison (Director, P.A.T.R.A.).

Mr. G. W. Mack (Building Research Station), *Painting Porous Building Materials.*

Dr. V. R. Gray (Timber Development Association), *The Wetting, Adhesion and Penetration of Surface Coatings on Wood.*

Mr. T. R. Bullett and Mr. A. T. S. Rudram (Paint Research Station), *The Coating and the Substrate.*

The Council has fixed the registration fee at £4 for members, £5 for non-members and £3 for wives, and the leaflet setting out details of the lectures and social programmes, together with registration forms, will be dispatched in January to members resident in the United Kingdom and General Overseas Sections. The relevant documents will also be sent to the Honorary Secretaries of the Commonwealth Sections so that any member of these Sections who wishes to come to the Conference should apply to his local Section Honorary Secretary.

The Mayor and Mayoress of Torquay have invited those attending the Conference to a Civic Reception at the Palace Hotel on the evening of Wednesday, 30 May, and the Association's Conference Dinner and Dance will take place on the final evening, Friday, 2 June, also at the Palace Hotel.

Arrangements have been made for several coach trips as well as a coastal and river cruise, two visits to the I.C.I. Limited's Marine Station at Brixham, and a mannequin parade for the ladies will be held in the Palace Hotel. Facilities are also available for members to play golf, tennis and table tennis.

Non-members of the Association wishing to obtain registration forms and any other information should write to the General Secretary, R. H. Hamblin, M.A., F.C.C.S., Oil & Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London, E.C.2.

Thirteenth Technical Exhibition, 1961

One hundred stands (covering over 13,000 square feet) have been allocated by the Exhibition Committee for the Thirteenth Technical Exhibition, which will take place in both the Old and New Halls of the Royal Horticultural Society, London, S.W.1, on 6, 7, 8, 9 March, 1961.

This is the first occasion on which the Exhibition has been open on four days and the increase of 25 per cent in applicants and 33 per cent in stand floor area over the 1960 Exhibition testifies to the popularity of this Exhibition, which provides a focus for the technical display of advances in raw materials, plant and equipment for use in the paint, printing ink and allied industries. Of the exhibitors at the 1961 Exhibition, 10 have not shown at previous exhibitions and a further 20 did not show at the 1960 Exhibition. Exhibitors from countries in the European Free Trade Association (Norway and Sweden), the European Common Market (Germany, Holland and Italy), and the Commonwealth (India), will be showing at the Thirteenth Technical Exhibition, besides companies from other countries who will be showing through associated companies in the United Kingdom. It is confidently expected, therefore, that the Exhibition will be the most comprehensive one yet held.

A central section in the Old Hall will be devoted to a display by research associations and will include a Technical Education Stand, on lines similar to that at the Twelfth Technical Exhibition. Parties of sixth form science students will once again be invited on two mornings only, when they will be given short introductory talks in a separate Lecture Hall, before visiting the Exhibition. The Technical Education Stand will be organised by technical colleges who will show the courses available in the technology of the paint, printing ink and allied industries and it is felt that this will be of interest not only to new entrants, but also to those who are concerned with the training of junior technical personnel.

A pleasing feature of recent Exhibitions has been the growing attendance of overseas visitors and interpreters will be present to help both overseas visitors and exhibitors.

The Exhibition Luncheon will once again be held at the Criterion Restaurant, Piccadilly, London, W.1, on 6 March and principal officers of other scientific bodies, industrial research associations, and organisations representing both suppliers and consumers will be invited to attend. The principal guest will be Sir Cyril Hinshelwood, O.M., D.SC., F.R.S., President of the Royal Society 1955-60, who will perform the opening ceremony at the entrance to the Old Hall at 3 p.m.

The hours of opening will be as follows:

Monday, 6 March	.. 3 p.m. to 7 p.m.
Tuesday, 7 March	.. 10 a.m. to 7 p.m.
Wednesday, 8 March	.. 10 a.m. to 7 p.m.
Thursday, 9 March	.. 10 a.m. to 7 p.m.

There will be no charge for admission or for the *Official Guide*, which will be available from the Information Bureaux, or from the Association's offices prior to the Exhibition.

Buffet facilities will be available in both Halls for the convenience of visitors to the Exhibition and a full restaurant service will be available from 12.30 p.m. to 2.30 p.m. on the second, third and fourth days.

Further information can be obtained from the General Secretary, R. H. Hamblin, M.A., F.C.C.S., Oil and Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London, E.C.2. Tel.: MONarch 1439.

Alphabetical List of Exhibitors

Albro-Fillers & Engineering Co. Ltd.
Alchemy Ltd.
Allied Colloids Ltd.
Amalgamated Oxides (1939) Ltd.
A.P.V. Co. Ltd., The.
Associated Lead Manufacturers Ltd.
Bakelite Ltd.
Baldwin Industrial Controls.
Beck, Koller & Co. (England) Ltd.
Berk, F. W., & Co. Ltd.
B.I.P. Chemicals Ltd.
Beake, A., Roberts & Co. Ltd.
Boehm, Frederick, Ltd.
Boulton, William, Ltd.
British Celanese Ltd.
British Oil & Cake Mills Ltd.
British Oxygen Co. Ltd.
British Resin Products Ltd.
British Titan Products Co. Ltd.
BX Plastics Ltd.
Campbell, Rex, & Co. Ltd.
Carless, Capel & Leonard Ltd.
Ciba (A.R.L.) Ltd.
*Ciba Clayton Ltd.
Cole, R. H., & Co. Ltd.
Cornbrook Chemical Co. Ltd.
Cornelius Chemical Co. Ltd.
Cray Valley Products Ltd.
*Crosfield, Joseph, & Sons Ltd.
Croxton & Garry Ltd.
Distillers Co. Ltd.
Durham Raw Materials Ltd.
Elliott, H. J., Ltd.
Esso Petroleum Co. Ltd.
Evans Electroselenium Ltd.
Farbenfabriken Bayer Aktiengesellschaft
*Fatoils Ltd.
*Ferranti Ltd.
Frenkel, Leon, Ltd.
Geigy Co. Ltd.
Gilbarco Ltd.
Goodyear Tyre & Rubber Co. (G.B.) Ltd.
Hardman & Holden Ltd.
Hercules Powder Co. Ltd.
*Hoechst Chemicals Ltd.
Hygrotherm Engineering Ltd.
Imperial Chemical Industries Ltd.
Isopad Ltd.
Johns Manville & Co. Ltd.
Johnson, Matthey & Co. Ltd.
Kek Ltd.
*Kunstharsfabriek Synthese, N/V
Remmert-Holland, N/V
Laporte Titanium Ltd., and
Laporte Chemicals Ltd.
*Lehmann Maschinenfabrik, G.m.b.H.
Marchant Bros. Ltd.
Metal Propellers Ltd.
Micafine Ltd.
Mill Room Accessories & Chemicals Ltd.

Mitchell, L. A., & Co. Ltd.
Mitchell, W. A., & Smith Ltd.
*Montecatini-Soc. Generale per l'Industria Mineraria e Chimica
National Adhesives Ltd.
National Coal Board
Norwegian Talc, A/S
Novadel Ltd.
**Paint Journal, The*
Paint Manufacture
Paint, Oil & Colour Journal
Paint Research Station
Paint Technology
Pascall Engineering Co. Ltd.
Premier Colloid Mills Ltd.
Price's (Bromborough) Ltd.
Research Council of the British Whiting Federation
Research Equipment (London) Ltd.
*Schenectady-Midland Ltd.
Sheen Instruments (Sales) Ltd.
Shell Chemical Co. Ltd.
Shellac Export Promotion Council
Silverson Machines (Sales) Ltd.
Spelthorne Metals Ltd.
Steele & Cowlishaw Ltd.
Styrene Co-Polymers Ltd.
Surface Coating Synthetics Ltd.
Svenska Oljeslageri Aktiebolaget
Swada (London) Ltd.
Tin Research Institute
Tintometer Ltd.
Torrance & Sons Ltd., & Holmes Bros. (Paint Machinery) Ltd.
Union Carbide Ltd.
United Coke & Chemicals Co. Ltd.
Vickers-Armstrong (Engineers) Ltd.
Vinyl Products Ltd.
Watford Chemical Co. Ltd.
Winkworth Machinery Ltd.
Wolf, Victor, Ltd.
Younghusband Stephens & Co. Ltd.
Technical Education Stand
O.C.C.A. Information Bureaux

* *Denotes exhibitors who have not shown at previous Exhibitions.*

London Section

FORMATION OF SOUTHERN BRANCH

At its meeting on 18 November the Council gave formal approval to the formation of a Southern Branch of the London Section, which will meet in the area Southampton/Portsmouth, and the Honorary Officers for the time being will be:

Chairman:	Mr. R. A. Brown
Honorary Secretary:	Mr. F. W. Davies
Honorary Treasurer:	Mr. J. C. Kingcombe

The London Section Chairman, Mr. J. A. L. Hawkey, will be *ex officio* a member of the Branch Committee and Mr. F. W. Davies will represent the Branch on the London Section Committee.

It is hoped to hold a programme of technical meetings throughout the remainder of this Session and there will be a first Annual General Meeting when the election of Officers and Committee will take place.

Members or non-members wishing to learn more of the activities of the Branch should write to the Honorary Secretary at the following address:

5 Charlesbury Avenue,
Alverstoke,
Gosport, Hants.

LADIES' NIGHT

Once again the feet of London Section members and their guests were borne by the varied array of public and private transport to that mecca of all travellers, the hub of the Commonwealth, when on Friday, 18 November, the Annual Ladies' Night was held at the Criterion Restaurant in Piccadilly. This year the attendance was recorded as 249, which was rather smaller than usual, owing perhaps to the nearness in date of other paint industry functions of a similar nature.

After reception by the Chairman, Mr. J. A. L. Hawkey, and Mrs. Hawkey and the President, Mr. P. J. Gay, and Mrs. Gay, the company partook of a meal of the high standard which has come to be associated with the event. The guest of

The President, Mr. P. J. Gay, and Mrs. Gay, with the Chairman of the London Section, Mr. J. A. L. Hawkey, and Mrs. Hawkey, at the London Section Ladies' Night.

honour this year was Dr. L. Valentine, the newly-appointed Director of the Paint Research Station, who proposed the toast to the London Section. Dr. Valentine confessed that he had been informed that the inner circles of the London Section Committee, having laid down the qualifications necessary for the proposer of the toast as being a knowledge that colour is measured in N.B.G. units, that a long oil alkyd is made from C_{18} acids and a short oil one from C_{12} acids, and that the proposer should never have attended a London Section meeting, had decided that he was the only candidate who could fulfil all those conditions. He continued by reference to his earlier experiences in the fields of oil and colour by admitting to having written in his University days an essay on "Colour and Chemical Constitution". This had been so well spotted as an examination question by the examinees that the examiners had decided to leave it out of their consideration in the award of degrees. He referred to the first public function of the London Section some twenty years ago at the Criterion Restaurant and commented that tickets then cost no more than five shillings. He continued by saying that whilst London Section was not perhaps the oldest in age as a Section, it was by no means the least distinguished, and referred to the many activities which had emanated from it, citing in particular the post-graduate lectures and the Technical Exhibition, which had now grown into such a healthy offspring as to warrant national status.

The Chairman responded to the toast and said that it was pleasant to know that the Sectional activities could be taken as a mirror of the Association and, without any disrespect to the other Sections, he felt that the praise lavished by Dr. Valentine was fully merited. He paid tribute to the work carried out by the Committee and especially the Hon. Secretary, Mr. M. R. Mills, in whose hands the arrangements for the Ladies' Night had been completely vested. He referred to the effects of the lecture given by Mrs. Arnold last year and the ways in which members had taken her advice so much to heart in their personal activities. Mr. Hawkey reported that the advice given by Mr. Arnold at an earlier date, that there should be a division of London Section, had now been partially taken and the formation of a Southern Branch of London Section had been approved by Council at its meeting earlier in the day. He asked Mr. F. W. Davies, who was present, who had been the most prominent member in this new activity, to accept the acclamation of the company.

The Chairman then greeted the guests of the Section, Mr. and Mrs. Geary from Hull, Mr. and Mrs. Hibberd from Newcastle, Mr. and Mrs. Seymour from the Midlands, Mr. Smith from Manchester and Mr. Butler from the West Riding, and also two past Presidents, Mr. Mundy and Mr. Gosling. He said that the ladies needed no introduction to oil and colour as they were only too familiar with them in their daily lives in such places as the kitchen, and concluded by proposing the toast to the guests and ladies. Mrs. S. R. W. Martin, wife of the former Hon. Treasurer of the Section, responded to the toast on behalf of the guests and ladies in a brief but delightfully phrased speech, in which she confessed to knowing the answer to what the members did at their meetings by having been present at one of them at Scarborough.

Following the formal toast list, which was concluded in accordance with the wishes of the Committee at a comparatively early hour, the remainder of the evening was given over to the delights of the dance, which ranged from the exotic Latin American rhythms to the athleticisms of the Gay Gordons, all being most ably accompanied by the music of George Fierstone and his Orchestra, who have fulfilled this role for so many years to become an almost inseparable part of the London Section Ladies' Night. The colourful displays of the terpsichorean arts continued with only a slight pause for refreshment with "the cup that cheers" until 1 a.m. After the traditional musical honours had been accorded to the Chairman, the President and the guest of honour and their respective ladies, another successful London Section Ladies' Night was over, and the party unwillingly broke up to wend their various ways through the murky fog of a November night.

C. R. P.

Manchester Section

DINNER-DANCE

The Section's Annual Dinner and Dance, held at the Midland Hotel, Manchester, on 28 October, was attended by some 350 people. After the President, Mr. P. J. Gay, and Mrs. Gay had been welcomed by the Chairman, Mr. H. Smith, and Mrs. Smith, they jointly received the guests and members. This year's principal guests were Dr. H. Samuels, a director of the Dyestuffs Division of Imperial Chemical Industries Limited, and Mrs. Samuels, Dr. L. Valentine, Director of the Paint Research Station, Mr. R. H. Hamblin, General Secretary of the Association, and Mrs. Hamblin, Mr. E. A. Bullions, Chairman of the Scottish Section, and Mrs. Bullions, and Mr. J. S. Geary, Chairman of the

The President, Mr. P. J. Gay, and Mrs. Gay, chatting with the Section's Chairman, Mr. H. Smith, and Mrs. Smith at the Manchester Section's Annual Dinner and Dance on 28 October.

Hull Section, and Mrs. Geary. In addition, the top table was graced by Dr. H. A. Hampton, President Designate of the Association, and Mrs. Hampton, and Dr. J. B. Harrison, Association Research and Development Officer, and Mrs. Harrison.

After cocktails, the company received their Chairman and guests at table with acclamation and sat down to an excellent meal, most efficiently served. Dr. Samuels, proposing a toast to the Manchester Section, recalled its formation as the first Section to be constituted, and its contribution to the Association's well-being from those of its members who had held the office of President in the past and those who had served as Officers or as Council members.

Mr. H. Smith, responding, spoke of the continuance of tradition within the Section, particularly in recapturing the atmosphere of that first meeting when the Section was founded in one of Manchester's well-known public houses. He went on to propose the toast to "Our Guests", in which he expressed the assembled company's pleasure at having with them the President and his lady, and the several distinguished guests at this, the Section's principal social occasion. Mr. Smith made special reference to the ladies who, he said, brought such charm to the occasion, and, like a good wine, kept in obscurity for another year, seemed always to come out improved in both quality and appearance.

Dr. Valentine, in response, on behalf of the guests, expressed appreciation for the warm welcome which was being extended to them. In his new position, he said, he planned to renew many of his past associations with members in the North-West, and to get to know the interests represented by members of the Manchester Section.

Dancing began after a short interlude, music being provided by the Midland Hotel Orchestra, and the remainder of the evening was spent in a most convivial and enjoyable atmosphere. This occasion was a considerable success, and Mr. Hudson, the Social Secretary, and Mr. McDowell, the Secretary, are to be warmly congratulated.

I. S. M.

Register of Members

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

Ordinary Members

AIKEN, ALEXANDER RONALD, 10 Rodney Street, Moorabbin, S.20, Victoria, Australia. (*Victorian*)

ATKINS, ERIC GEORGE, Highwood, Runwell Road, Wickford, Essex. (*London*)

BRIGHT, JOHN EDWARD, 4 Susan Court, Cheltenham, S.22, Victoria, Australia. (*Victorian*)

CONSTANT, BRENDON SMITH, 42 Clarendon Gardens, Stone, Dartford, Kent. (*London*)

COOPER, ERIC LESLIE, 58 Northaw Road, Cuffley, Herts. (*London*)

DALTON, FRANK, Fishburns Printing Ink Co., St. Albans Road, Watford, Herts. (*London*)

DELORETTE, GUSTAV OTTO HANS-JURGEN, Smith & Walton S.A. (Pty.) Ltd., 317 Chamberlain Road, Jacobs, Natal, South Africa. (*South African*)

HANSON, ROBERT PHILIP, 15 Grange Road, Ryton-on-Tyne, Co. Durham. (*Newcastle*)

HOFFERTH, BURT F., Armstrong Cork Co., Research and Development Centre, 2500 Columbia Pike, Lancaster, Pa., U.S.A. (*Overseas*)

JUBB, ANTHONY WILLIAM, Associated Chemical Co. Ltd., Central Research Laboratories, Kirkstall Lane, Leeds, 5. (*Manchester*)

LEVER, COLIN, Transparent Paper Ltd., Bridge Hall Mills, Bridge Hall Lane, Bury, Lancs. (*Manchester*)

LONG, DENIS TERENCE, Harringtons & Goodlass Wall Ltd., Commons Road, Cork. (*Manchester*)

MATTHEWS, JOHN WILLIAM ALEXANDER, 185 Victoria Square, c/o Icianz, Adelaide, South Australia. (*South Australian*)

MIEBACH, FRIEDRICK AUGUST WERNER, Gelson Industries, 15 Church Street, Hawthorn, Victoria, Australia. (*Victorian*)

MORRIS, ARTHUR, 63 Beckett Street, Bolton, Lancs. (*Manchester*)

MOSS, LESLIE, Russell Kirby Ltd., Birchill Road, Kirkby Industrial Estate, Liverpool. (*Manchester*)

PARTINGTON, JOHN LIONEL, 13 Sidmouth Avenue, Flixton, Nr. Manchester. (*Manchester*)

SELF, WILLIAM GOODWIN, 5 Hovea Crescent, Wundowie, Western Australia. (*South Australian*)

SHETH, LALITCHANDRA TRIBHOUANDAS, C-7 Sarvonnati Society, Kirol Road, Ghatkopar, Bombay, 77, India. (*Overseas*)

SHNEIER, LIONEL BERTRAND, 1 Waugh Avenue, Northcliff, Johannesburg, South Africa. (*South African*)

SPENCER, CYRIL, 19 Beechcroft Road, Ipswich, Suffolk. (*London*)

TRUKSA, LESLAW KAZIMIERZ, 18 Kings Avenue, Woodford Green, Essex. (*London*)

WAHLIN, ERIK, Korsvagen 15, Nacka, Sweden. (*Overseas*)

WALLACE, DONALD ARTHUR, 27 Brooklands Avenue, Wimbledon Park, London, S.W.19. (*London*)

WOOD, GERALD, 29 Leveson Crescent, Balsall Common, Nr. Coventry, Warwicks. (*Midlands*)

Associate Members

Bartrum, Robert Paul, Walmley, Layton Road, Rawdon, Leeds. (*West Riding*)
Millan, Robert B., 85 Palm Bay, St. George's Street, Durban, South Africa. (*South African*)
Watson, Peter Wainwright, Associated Lead Manufacturers Ltd., 63 Albion Street, Leeds. (*West Riding*)
Willis, Robert John, Colours & Chemicals Pty. Ltd., P.O. Box 5, Mentone, Victoria, Australia. (*Victorian*)
Wood, Gerald, 12 Nelson Avenue, Monton, Manchester. (*Manchester*)

Junior Members

Allan, Robert, 47 Sandringham Avenue, Willenhall, Staffs. (*Midlands*)
Ekermans, B., Herbert Evans & Co. Ltd., P.O. Box 1386, Durban, South Africa. (*South African*)
Fowler, Philip John, 14 Ashville Road, Ashton Gate, Bristol, 3. (*Bristol*)
Greenland, Roger, Permoglaze Ltd., James Road, Tyseley, Birmingham, 11. (*Midlands*)
Hatfield, Hugh Roger, 204 Birmingham Road, Sutton Coldfield, Warwicks. (*Midlands*)
Hosking, Ronald Barry, 26 Tular Avenue, South Oakleigh, Victoria, Australia. (*Victorian*)
Lane, Arthur Edwin, 19 Richford Road, Portway, West Ham, London, E.15. (*London*)
Laybourn, Peter, 123 Hedgeley Road, Hebburn, Co. Durham. (*Newcastle*)
Lowrey, Kenneth William, "The Greyhound Inn", Hedworth, Jarrow upon Tyne. (*Newcastle*)
Macdonald, Donald, c/o Spartan Paints Pty. Ltd., Box 43, West Footscray, Victoria, Australia. (*Victorian*)
Masik, Andrey, c/o Spartan Paints Pty. Ltd., Box 43, West Footscray, Victoria, Australia. (*Victorian*)
Seja, Viesturs, 68 Bindi Street, Glenroy, Victoria, Australia. (*Victorian*)
Watson, Kenneth Alan, 14 Colmore Avenue, Kings Heath, Birmingham, 4. (*Midlands*)
Williams, Warwick Harriden, c/o Robert Bryce Co. Ltd., 526 Little Bourke Street, Melbourne, Victoria, Australia. (*Victorian*)
Windsor, Frederick Barry, 26 Ventnor Street, Salford, 6. (*Manchester*)

Dr. H. W. Keenan in Norway

The Association of Norwegian Paint and Varnish Chemists held its annual meeting in Bergen on 14 and 15 October, under the Presidency of Mr. E. Lund. Mr. K. G. Fleischer was the Host of the Conference.

The technical programme included a lecture on "Colour Conditioning" delivered by Mr. H. Songstad, and a lecture by Dr. H. W. Keenan on "The Use of Specific Additives in Polyester Resins for Surface Coatings". The afternoon of Friday was devoted to a visit to International-Farvefabrik A/S and on Saturday the party visited Norwegian Talc A/S. On Friday evening, Members of the Association foregathered at the Restaurant Bellevue, most aptly named, for dinner. After the dinner the President welcomed Dr. Keenan as Guest of Honour and reminded him that it was ten years since his last visit to Norway and for this reason the occasion was something of a jubilee.

In reply, Dr. Keenan thanked the President and Mr. Fleischer for the honour they had accorded to him on behalf of the Association. Ten years ago, said Dr. Keenan, he was presented at Sandefjord

THE TOP TABLE AT BELLEVUE, BERGEN

with a bottle of Aquavit from which only Norwegian friends, and an occasional Danish friend, had partaken until Haavard Langseth (Halv Langseth's son) drained the bottle on his twenty-first birthday. Dr. Keenan then took from his pocket a piece of translucent polyester laminate in which the Aquavit label was embedded and returned it to Mr. H. Langseth as a memento of two happy occasions, 1950 and 1960.

H. W. K.

Obituary

ROY B. WATERS

Dr. Roy B. Waters, a member of the Manchester Section for many years, died recently at the age of 52 after a brief illness. He gained his B.Sc. in 1928 at the East London College and the following year was awarded an M.Sc. and a Research Studentship. In 1931 he received his Ph.D. and remained at the college to continue research until entering industry two years later.

After a brief spell with a paint company, Dr. Waters joined the Resins Service Department of I.C.I. and was thereafter actively engaged in development and technical service work up to the time of his illness, when he held the position of Assistant Manager in the Polymer and Chemicals Service Department under Dr. H. A. Hampton. During these years he travelled widely to many European countries and to India and was able to pursue his interest in photography during his spare time abroad. He was also a keen bird-watcher and for many years was actively engaged in local amateur dramatics.

Roy Waters was well known to many people in the resin-consuming industries, who will remember him particularly for his dry sense of humour, utter frankness and total dislike of any form of hypocrisy or ostentation. He leaves a wife and daughter.

NEWS OF MEMBERS

Mr. J. McPherson, an Ordinary Member attached to the Midlands Section, has recently been appointed as the Technical Director of Harrison S. Walton & Son Ltd.

Mr. J. L. Fletcher, an Ordinary Member attached to the Manchester Section, has been appointed manager of the General Chemicals Division, Cyanamid of Great Britain Limited. Mr. Fletcher, who was previously connected with the paint and plastics industries, will be responsible for the bulk sales of Cyanamid's chemical products in the United Kingdom which will include the sales direction for melamine crystal production from the company's new Gosport plant.

Mr. W. F. Williams, an Ordinary Member attached to the Scottish Section, has been appointed Manager of the Scottish Sales Region of the Industrial Chemicals Division of the Shell Chemical Company.

MR. W. A. WOOD

Mr. W. A. Wood, an Ordinary Member attached to the West Riding Section, is now sales representative for London and the South of England for *Epok* surface-coating resins, manufactured by British Resin Products Ltd. Mr. Wood was formerly their Northern representative.

CHANGE OF ADDRESS

Since membership of the Association is on a personal basis, members are asked to note that, although the General Secretary's office may be notified of the change of address of companies, this does not mean that such changes are automatically noted on the record cards of members employed by those companies.

Where a member uses his company's address for communication purposes, it is necessary for him to inform the General Secretary's office of any change in the address and, at the same time, to quote his personal number, if possible.

APPLICATIONS FOR ORDINARY MEMBERSHIP

At the Council meeting held on 15 February, 1955, the Council passed a resolution in accordance with Association Rule 5 (ii), reaffirming the standard of competence for the election of candidates to Ordinary Membership of the Association, and it is felt that members would like to be aware of this information when sponsoring candidates for election.

The Council's resolution reads as follows:

THAT the qualifications for the granting of Ordinary Membership shall be—

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

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

BINDING OF THE ASSOCIATION'S JOURNAL

Members will be pleased to know that W. Heffer & Sons Ltd., Hills Road, Cambridge, will undertake the binding of back volumes of the Association's *Journal*, sent in by individual members, at a cost of 16s. 6d. per volume.

Members wishing to avail themselves of this facility should send the parts direct to Heffer & Sons Ltd., enclosing a remittance of 16s. 6d. and ensuring that notes bearing their names and addresses are enclosed with the parcels.

THE PERKIN CENTENARY TRUST

The Celebrations marking the discovery of Mauvine, the first important synthetic dye, by William Henry Perkin, in 1856, have led to the formation of the Perkin Centenary Trust. This has been established as a lasting memorial of this occasion and to promote technical education in all aspects of the manufacture and application of colouring matters. Application for the following awards should be made on forms available from the Secretary of the Trust, c/o The Chemical Society, Burlington House, London, W.1, and must be received not later than 1 May, 1961:

The Perkin Centenary Fellowship

This award is offered for one or two years for the purpose of higher study of any subject approved by the Trustees. Candidates will be required to show *either* that they have had industrial experience relating to the manufacture or application of colouring matters, *or* that their intended field of study has a direct bearing on these subjects.

The Perkin Centenary Scholarship

Two such awards are offered, each for two years, renewable at the discretion of the Trustees for one further year, to enable candidates employed in an industrial firm or other institution concerned with the manufacture or application of colouring matters to receive an education at a university or a technical college.

The Perkin Centenary Trust Travel Grants

Applications are invited from teachers concerned with the study of any aspect of the manufacture or application of colouring matters at a university, technical college or other institution.

THE BRITISH COLOUR MAKERS' ASSOCIATION

As a result of the Annual General Meeting of the British Colour Makers' Association held on 9 November, 1960, the following will be the Officers and Council for the ensuing year: *Chairman*, Mr. A. K. Ames (Imperial Chemical Industries Ltd.); *Vice-Chairman*, Mr. G. E. Hillier (J. W. & T. A. Smith Ltd.); *Honorary Treasurer*, Sir Christopher Cowan (Cowan Bros. (Stratford) Ltd.); *Council:* Mr. A. K. Ames (Imperial Chemical Industries Ltd.), Sir Christopher Cowan (Cowan Bros. (Stratford) Ltd.), Mr. G. E. Hillier (J. W. & T. A. Smith Ltd.), Mr. H. Gosling (Cornbrook Chemical Co. Ltd.), Mr. J. H. Grimshaw (Horace Cory & Co. Ltd.), Mr. C. L. Lewis (Joseph Storey & Co. Ltd.), Mr. H. Pike (Hull & Liverpool Red Oxide Co. Ltd.), Mr. J. Smethurst (James Anderson & Co. (Colours) Ltd.) and Mr. V. Watson (Cromford Colour Co. Ltd.).

LECTURES ON SOLVENTS

A course of six lectures will be given by specialist lecturers on "New Trends in the Use of Solvents" at the Borough Polytechnic. The course is intended for graduates and others engaged in industrial and paint laboratories and the fee for this course will be 10s. Application to attend this course should be made to the Secretary, Borough Polytechnic, Borough Road, London, S.E.1.

The programme for the series is given below:

12 January, 1961
"General Introduction and Some New Solvents", Dr. L. M. Barakan (Distillers Company Ltd.).

19 January, 1961
"The Use of Hydrocarbon Solvents in Surface Coatings", Mr. J. E. Loible and Mr. R. J. Turner, B.SC., A.R.I.C. (Shell Chemical Co. Ltd.).

26 January, 1961
"The Scientific Selection of Paint Solvents—The Solubility Parameter", Mr. A. R. H. Tawn, F.R.I.C. (Coates Brothers Ltd.).

9 February, 1961
"Water as a Paint Solvent—Stoving Finishes", Mr. A. G. North, B.SC. (Cray Valley Products Ltd.).

16 February, 1961
"Water as a Paint Solvent—Air-drying Finishes", Mr. F. Armitage (Berger, Jenson & Nicholson Ltd.).

All lectures will commence at 6.30 p.m.

PHYSICS OF POLYMERS

The Institute of Physics and the Physical Society announce that a Conference entitled "Physics of Polymers" will be held at the H. H. Wills Physics Laboratory at the University of Bristol on 10, 11, 12 January, 1961. The sessions are as follows:

Chain statistics and solution properties.
Molecular motions.
Crystallinity: experiment, theory.
Irradiation effects.

The introductory lecture will be given by Professor Hermann Mark, and an evening lecture will be given by Sir Gordon Sutherland on infra-red spectroscopy of polymers.

Limited hostel accommodation is available. Registration is necessary and programmes and application forms are obtainable from the Administration Assistant, The Institute of Physics and the Physical Society, 47 Belgrave Square, London, S.W.1.

VITH FATIPEC CONGRESS

The VIth FATIPEC Congress will be held at Wiesbaden in the Federal Republic of Germany from 21 to 25 May, 1962. The subject of the Congress will be "The Modern Scientific Basis for the Production, Use and Examination of Pigments, Media, Surface Coatings and Printing Inks". Information on the Congress will be given in three bulletins to be published in 1961, and in the meantime if any member wishes to present a paper, or to receive copies of the bulletin, he should write to the General Secretary at the address shown on the front cover.

Forthcoming Events

(Note: Details are given of meetings arranged in the U.K. up to the 15th of the month following publication, and in the Commonwealth up to the 15th of the second month after publication.)

Thursday, 5 January
Newcastle Section. "The Corrosion of Iron and Steel" by Dr. J. E. O. Mayne, at the Royal Turks Head Hotel, Newcastle upon Tyne, at 6.30 p.m.

Monday, 9 January
Hull Section. Film Show. Programme to be announced later. Members' wives and friends will be welcome. To be held at the Royal Station Hotel, Hull, at 7.15 p.m.

Tuesday, 10 January
West Riding Section. "A Review of Synthetic Resins" by S. R. W. Martin, PH.D., at The Metropole Hotel, Leeds, at 7.30 p.m.

Friday, 13 January
Scottish Section. Annual Dinner-Dance in the Central Hotel, Glasgow.

Manchester Section. "The Blooming of Organic Pigments in Stoving Lacquers" by G. G. Kositzke, at Bleachers' Association Ltd., Blackfriars House, Manchester, 3, at 6.30 p.m.

Wednesday, 18 January
London Section. Joint Meeting with Surface Activity Group, Society of Chemical Industry—"The Use of Surface Agents in the Paint and Printing Industries" by W. Carr, B.SC., PH.D. (The Geigy Co. Ltd.).

Thursday, 19 January
Scottish Section. "Automobile Painting Processes" by I. Walker, at More's Hotel, India Street, Glasgow, C.2, at 7.30 p.m.

Friday, 20 January

Midlands Section. "Problems Encountered with Air-Drying Soya Bean Alkyds" by P. D. Cox, A.R.I.C., and G. T. Williams, at Regent House, St. Philips Place, Colmore Row, Birmingham, at 6.30 p.m.

Saturday, 21 January

Scottish Section Student Group. "Colour and Art" by H. O. Hellier, A.T.D., at More's Hotel, 18 India Street, Glasgow, C.2, at 10.0 a.m.

Friday, 27 January

Bristol Section. "A Method of Hiding Power Determination as an Aid to Paint Formulation" by P. I. Adams, M.A. (Imperial Smelting Corporation Ltd.) at the Royal Hotel, College Green, Bristol, 1, at 7.15 p.m.

Thursday, 2 February

Newcastle Section. "The Growth of the Paint Industry in the North-East" by J. C. Hanna, at the Royal Turks Head Hotel, Newcastle upon Tyne, at 6.30 p.m.

Monday, 6 February

Hull Section. "Some Resin Problems of an Electrical Component Manufacturer" by Dr. W. Marshall (Technical Manager, Morganite Resistors Limited), at the Royal Station Hotel, Hull, at 7.15 p.m.

Thursday, 9 February

Scottish Section. "Surface Coatings in Atomic Energy Installations" by H. Wells, at More's Hotel, India Street, Glasgow, C.2, at 7.30 p.m.

Friday, 10 February

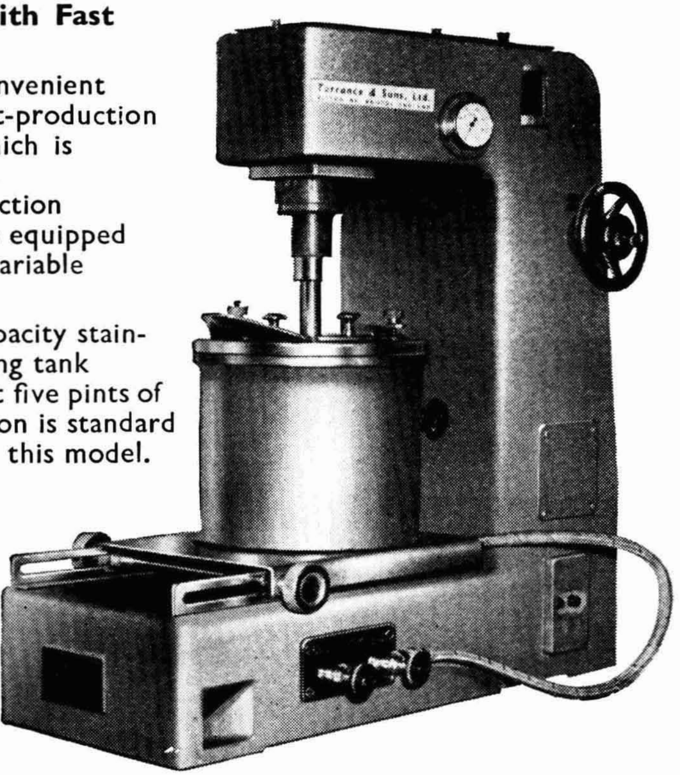
Manchester Section. "Plastisols" by A. E. Meazey, B.SC., at Bleachers' Association Ltd., Blackfriars House, Manchester, 3, at 6.30 p.m.

Saturday, 11 February

Scottish Section Student Group. "Microbiology in Paints" by D. J. Cowley, B.SC., at More's Hotel, 18 India Street, Glasgow, C.2, at 10.0 a.m.

Tuesday, 14 February

West Riding Section. "Problems Encountered with Air-Drying Soya Bean Alkyds" by P. C. Cox, A.R.I.C., and G. T. Williams, at The Metropole Hotel, Leeds, at 7.30 p.m.



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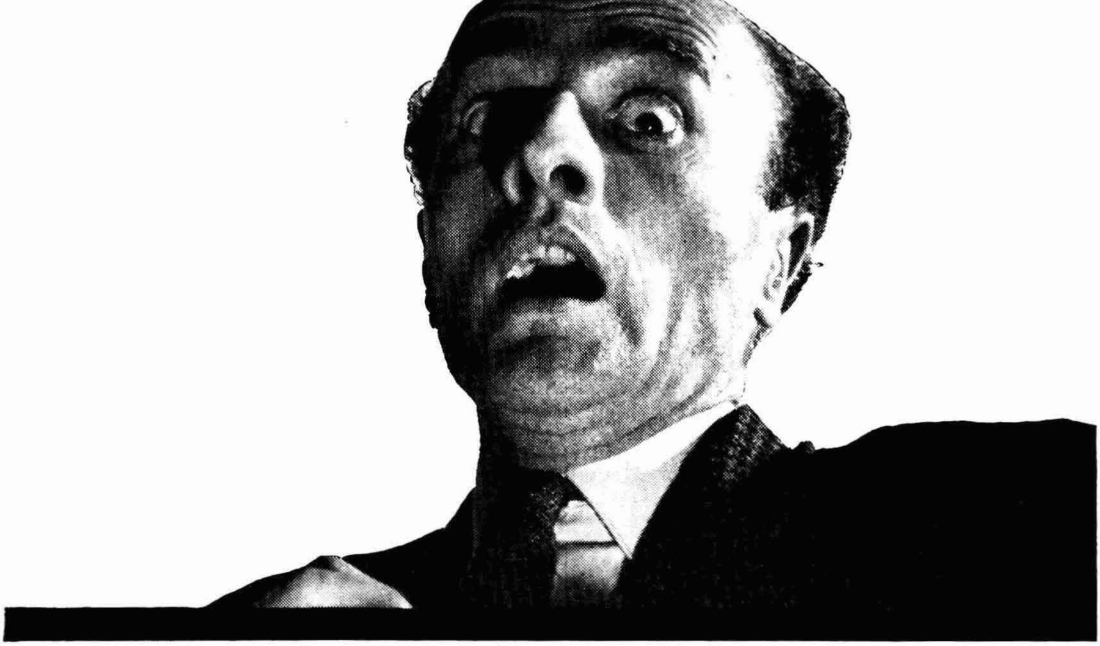

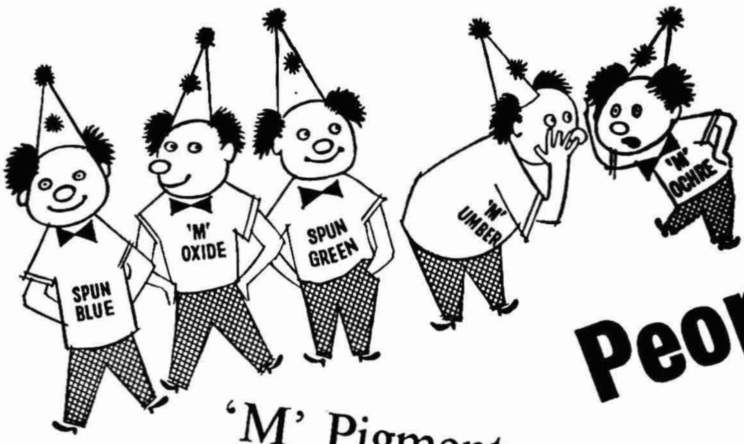

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