

JOURNAL of the OIL AND COLOUR CHEMISTS' ASSOCIATION



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The laboratory evaluation of the behaviour of coloured pigments on ball-milling^{*}

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Summary

A method is described for the rapid determination of the optimum conditions for the dispersion of a pigment by ball-milling, using the *Red Devil Paint Conditioner*. The basic experiment was so designed that information on tendency to give "shock-seeding" and on ease of discharge from the mill was also obtained. The behaviour of different types of pigments under varying conditions of ball-milling is discussed.

For most of the work efficiency of dispersion was measured by a fineness of grind gauge, but a modified technique to investigate rate of strength development has been developed.

The overriding conclusion reached was that the need for correct formulation of ball-mill bases is even more important than has been suggested in the past.

Evaluation en laboratoire des reactions des pigments colorants lors du broyage par boulets

Résumé

Une méthode est décrite pour la détermination rapide des conditions optima pour la dispersion d'un pigment au moyen du broyage par boulets, utilisant le *Red Devil Paint Conditioner*. L'expérience fondamentale a été conçue de façon à obtenir également des renseignements sur la tendance à la granulation ("shock-seeding") et sur la facilité d'écoulement hors du broyeur. Cet exposé commente les réactions de différents types de pigments dans des conditions diverses de broyage par boulets.

Pour la plupart des travaux effectués, l'efficience de la dispersion fut évaluée à l'aide d'un appareil à mesurer la finesse du broyage, mais une technique modifiée a été mise au point pour étudier l'évolution du taux d'intensité.

La conclusion la plus importante tirée de ces travaux fut la suivante : la nécessité d'une formulation correcte des bases pour broyeurs à boulets est encore plus importante que ce qui avait été supposé par le passé.

Die Laboratoriumsauswertung des Verhaltens von Farbpigmenten beim vermahlen in der Kugelmühle

Zusammenfassung

Eine Methode für die schnelle Bestimmung der optimalen Bedingungen bei der Feinverteilung eines Pigmentes in der Kugelmühle wird beschrieben. Dabei wird der *Red Devil* Farbzusatz benutzt. Das Hauptexperiment war so geplant, dass ausserdem noch Angaben über eine Tendenz zur "Schockverkörnung" und über die günstigen Entnahmebedingungen aus der

* Read before the London Section 6 November 1963 and the Manchester Section 13 December 1963.

Mühle ermittelt wurden. Das Verhalten von verschiedenen Pigmenttypen und der wechselnden Bedingungen in der Kugelmühle wird erörtert.

Für den grössten Teil der Arbeit wurde die Dispersionsleistung nach Feinheit des Mahlgrades gemessen, aber darüber hinaus wurde ein verbessertes Verfahren zur Untersuchung des Stärkengrades entwickelt.

Die bestimmende Schlussfolgerung war, dass die Notwendigkeit zur angemessenen Zusammenstellung der Kugelmühlengrundstoffe noch wichtiger ist, als in der Vergangenheit behauptet wurde.

Introduction

It is well known that to attempt to disperse a mill base containing pigment, resin and solvent in the proportions to be used in the final paint is a very inefficient practice. The formulation of bases for single- or three-roll milling rarely presents problems. On such mills it is normal to use a concentrated resin solution and the proportion of pigment is adjusted so that the viscosity of the pre-mix will give the correct flow through the mill. This is relatively easily determined by trial and error since the process is under observation the whole time, is of short duration, and the consistency of the mill base can be varied by adding more pigment or more vehicle as appropriate.

In the case of ball-milling, however, the situation is quite different. The process is a lengthy one and under ideal conditions is carried out overnight without supervision. It is common knowledge that milling at high pigment concentrations and low resin solids produces the most efficient results, but apart from titanium dioxide there is little information on what "high" and "low" mean with different pigments.

An approximate idea of a ball-mill base can be obtained from a knowledge of the oil absorption of the pigment concerned although the information obtained from this figure is very limited. The technique most commonly used to determine mill base formulations is that due to Daniel¹, which involves titrating a certain weight of pigment with resin solution until the resultant paste will just flow. This is repeated using resin solutions of varying concentrations and the highest pigment/resin solution ratio which gives flow is chosen as the mill base. This technique has been used considerably over the past years and on paper seems relatively simple, but it does have a number of disadvantages, particularly :

(a) Although reasonable end points are obtained with most inorganic pigments, organic pigments do not behave as well and in some cases it is virtually impossible to determine the end point.

(b) Each titration involves considerable physical effort.

(c) Only the mill base formulation is obtained; no information is obtained on speed of milling and other features, which will be discussed later.

(d) The results obtained always have to be checked by small-scale ballmillings. It seems to be usual to add an arbitrary percentage of the order of 25 per cent to the proportion of resin solution found necessary in the titration. This is to attempt to overcome the thickening of mill bases which frequently occurs as milling proceeds.

It has also been suggested that pigment surface area can be used as a means of determining mill base formulation and it seems likely that this will give more reliable results than the Daniel technique because more account is taken of the increasing area to be wetted as dispersion proceeds. Nevertheless this technique still only gives the mill base formulation and again has to be checked by ball-milling trials.

It was felt that a systematic investigation, based on actual ball-milling tests, was indicated. The use of the *Red Devil Paint Conditioner*² for ball-milling trials has been described by Orwig³. This machine, shown in Fig. 1, provides



Fig. 1. Red Devil Paint Conditioner (with modified holder)

extremely severe agitation. The agitation is such that very rapid dispersion is obtained which gives the advantage that results are available more quickly. In fact, Orwig reported that one minute's dispersion on this machine was equivalent to approximately one hour's dispersion on a large-scale ball mill, although obviously there are many variables which could alter this factor. However, much more important than the speed of dispersion given by this machine is the fact that it can cope with mill bases of the viscosity of those used on the large scale. In this respect it differs from normal laboratory ballmilling equipment which can only handle mill bases of relatively low viscosity.

It was considered desirable to obtain information on the following features of the ball-milling of pigments : (i) the optimum pigment/resin/solvent ratio, (ii) the speed of dispersion of this combination, (iii) the tendency of the dispersed mill base to aggregate ("shock-seed") on addition of concentrated resin solution, and (iv) the ease with which the let down mill base could be discharged from the mill.

The Daniel flow-point technique and other possible methods for the determination of pigment/resin/solvent ratios do not give any information on the last three points mentioned above.

Design of experiment

In designing the basic experiment it always had to be borne in mind that an extremely large number of products had to be examined and it was necessary that this work should be carried out with the minimum of supervision. Several limitations were, therefore, imposed to reduce the volume of the work, although some of these were covered later with selected pigments by more extensive testing.

First, since no attempt was being made to evaluate the use of this machine as a ball-milling tool, but rather to compare the behaviour of pigments tested under similar conditions, no attempt was made to vary the size of vessel, the ball loading or the charge/voids ratio.

Secondly, it was decided that initially only one resin would be investigated and a long-oil linseed oil modified alkyd resin containing pentaerythritol (*Paralac 10*, from Imperial Chemical Industries Limited) was chosen.

Thirdly, the initial investigation was confined to one concentration of resin as grinding medium so that only the pigment/resin ratio had to be determined, and a 20 per cent solution of Paralac 10 in white spirit was used throughout. This restriction drastically reduced the amount of work involved and some explanation for this choice should be given. As the resin solids of the dispersing medium is reduced, milling efficiency first increases quite rapidly, then stays fairly constant until finally a point is reached where further reduction in resin concentration causes a decrease in efficiency. With most inorganic pigments, increases in efficiency are obtained as the resin solids of the dispersing medium is reduced below 20 per cent, although the improvement is not large. However, with some organic pigments, notably the arylamide reds and phthalocyanine blues, it is inadvisable to use much less than a 20 per cent resin concentration because efficiency of milling drops off considerably and the tendency to shockseed increases. Thus a 20 per cent resin solution gives efficient milling and at the same time is relatively "safe." This feature is discussed in more detail later.

Many ways of following rate of dispersion exist ; for example, changes in particle size, tinctorial value or gloss may be observed. For the basic series of tests, "seed level" readings on a fineness of grind gauge were used to determine degree of dispersion. Whilst there are objections to this method, it is probably the one most frequently used in practice and it has the merit of being simple and quick to operate. As with the choice of resin and choice of resin solids, other factors, particularly rate of strength development, could be investigated with pigments where these other features were of particular interest.

Thus the first stage of the experiment was to follow, by means of grinding gauge readings, the dispersion of a range of pigment/resin concentrations for each product, using a 20 per cent solution of *Paralac 10* in white spirit as dispersing medium. The second stage of the experiment was to test any mill base which had dispersed satisfactorily for tendency to shock-seed on addition of concentrated resin solution, and the third stage was to measure the flow of those mill bases which had dispersed satisfactorily and not shock-seeded on addition of concentrated resin.

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Experimental details

As manufactured, the *Red Devil Paint Conditioner* is only suitable for holding one paint can and a special holder was designed to carry six 4 oz bottles. This was constructed of rigid foam with an aluminium plate as base and, as shown in Fig. 2, the bottles fit into holes drilled in the foam, but care must be taken to ensure that a reasonable proportion of each bottle is exposed to the air to avoid excessive development of heat. The top of the holder consists of an aluminium plate covered with a layer of flexible foam and on the bottom plate are four locating lugs so that the holder can only be placed in the one position, in which the six bottles are arranged symmetrically about the plane of vibration.



Fig. 2. Modified holder for Red Devil Paint Conditioner

Four oz bottles were chosen as the most convenient size for this particular experiment, but holders can easily be made for larger bottles if more paint is required, or for smaller bottles if it is desired to carry out more experiments at the same time or to use smaller amounts of pigment.

The grinding charge consisted of 3 mm glass balls. Experience had shown that these gave an adequate speed of dispersion, did not affect the colour of the finish and were relatively cheap. Slightly more rapid dispersion was obtained if steel balls were used, but there was much greater danger of breakage and the colour of the finish could be affected. The standard charge of balls was 50 per cent of the volume of the bottle and the composition of each mill base was adjusted so that its volume equalled the voids' volume (i.e. approximately 20 per cent of the volume of the bottle).

Thus six different millings could be carried out at the same time, and it was found that if the ranges of concentration shown in Table 1 were used, only on rare occasions was it necessary to carry out a second set of millings

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on any one pigment. Weight concentrations have been used throughout for convenience but, from a theoretical point of view, volume concentrations are probably more significant. The ranges given in Table 1 for organic and inorganic pigments seem to be very different, but on a volume basis they are quite similar to each other.

Range of mill-base formulations				
Organic pigments	Inorganic pigments			
Wt. of pigment : Wt. of 20% alkyd resin (Paralac 10)	Wt. of pigment : Wt. of 20% alkyd resin (Paralac 10)			
20 : 80	55 : 45			
25 : 75	60:40			
30 : 70	65 : 35			
35 : 65	70 : 30			
40 : 60	75 : 25			
45 : 55	80 : 20			

Because the total *volume* of pigment plus resin solution was kept constant for each mill base, a different range of weights was needed for each specific gravity. The variation in specific gravity over the range of pigments to be examined was sufficient to make significant differences in volume if the same total weights were used throughout and it had been shown that variations in total volume of mill charge does have an effect on efficiency of dispersion as might have been expected.

For each pigment, a range of pigment weights and corresponding weights of 20 per cent resin solution was calculated and the six bottles charged with the appropriate quantities of glass balls, resin solution and dry pigment, in that order.

An oversize piece of polythene film was placed over the top of the bottle before screwing on the cap. In this way not only was the cardboard insert protected from destruction by solvent and glass balls but also a good seal was obtained. Agitation of the set of six mill bases was commenced and seedlevel readings were taken at five-minute intervals. Dispersion was stopped after 30 minutes.

To investigate tendency to give shock-seeding, concentrated resin solution (*Paralac 10W*, a 75 per cent solution of *Paralac 10* in white spirit) was added and the seed level taken again. The weight added was that required to raise the resin solids of the medium from 20 to 40-50 per cent. When this level of resin solids has been reached, further concentrated resin solution may be added without fear of shock-seeding. No excessive precautions were taken during this stage, other than making the initial additions slowly, and stirring the

mass with a glass rod. At the pigment concentrations and ball loading used, stirring was difficult and the conditions for this part of the experiment were undoubtedly severe. No attempt was made to equalise the temperatures of the mill base and resin to be added. This would have reduced the danger of shock-seeding, but is hardly a practicable procedure on the large scale.

All the let-down mill bases that had not shock-seeded were allowed to stand overnight and then tested for flow. This was done simply by removing the bottle top and replacing it with a gauze, clamping the bottle at 45° , open end downwards, and weighing the amount of pigment/resin slurry discharged in 15 minutes. It is very difficult to relate the ease of discharge from a 4 oz bottle containing 3 mm glass balls with the ease of discharge from a large ball-mill, but within each series these figures have some comparative significance. The possibility of measuring the viscosity of the let-down mill base was considered, but was ruled out partly because of the difficulty of deciding the rate of shear at which this should be done and partly because of the number of tests involved.

Assessment of results

A series of six graphs was drawn for each pigment, seed level being plotted against time of milling, and a note being made of any mixtures that shock-seeded on addition of concentrated resin. A series of curves typical of that obtained with a lead chrome pigment (CI Pigment Yellow No. 34) is shown in Fig. 3.

For the sake of convenience, a reading recorded as "less than 5 microns" has been plotted as if it were 3 microns, and although this represents the lower limit attainable using the Hegmann gauge, this does not, of course, mean that dispersion ceases at this point. This is one of the main drawbacks of the use of this type of gauge and is discussed in more detail later.

From this set of curves the most favourable combination could easily be selected. The 80/20 pigment/resin solution mixture was rejected because it gave shock-seeding on addition of concentrated resin solution; the 75/25 mixture showed slight aggregation on addition of concentrated resin and dispersed more slowly than the 70/30 mixture. The flow of the let-down 70/30 mill base was quite satisfactory and this combination was chosen because it gave better throughput and faster dispersion than mill bases of lower pigment concentration.

All the lead chrome pigments investigated gave similar results to this, that is, none of the bases that dispersed at the greatest rate gave shock-seeding or flow problems. However, these results were not typical of those obtained for an organic pigment, which are better represented by the series of curves shown in Fig. 4. The choice of optimum conditions was not as easy in this case.

The 45/55 pigment/resin solution mixture did not mill very satisfactorily; the 40/60 mixture was noticeably better than this, but in terms of speed of milling there was still further improvement as the pigment concentration was lowered. As the pigment concentration was reduced in stages from 35 to 20 per cent the speed of dispersion increased slightly, but the differences were small and at the same time the efficiency of dispersion as judged by throughput was reduced significantly. Using only these arguments, therefore, one would have been tempted to select the 35/65 mixture as representing the optimum conditions. When flow characteristics were taken into account, however, it



Fig. 3. (left) Typical dispersion curves for inorganic pigments in a 20 per cent solution in white spirit of an alkyd resin (Paralac 10)

Fig. 4. (right) Typical dispersion curves for organic pigments in a 20 per cent solution in white spirit of an alkyd resin (Paralac 10)

was found that in 15 minutes, 10g could be discharged from the let-down 35/65 mill base, whereas almost 30g could be discharged in the same period from the let-down 30/70 mill base. Under these conditions, therefore, we would choose 30 per cent pigment and 70 per cent of 20 per cent *Paralac 10* solution as the preferred mill base.

Most of the organic pigments gave results similar in general type to this, that is, the higher concentrations of pigment milled very slowly and in many cases showed shock-seeding on further addition of resin. As the pigment concentration was reduced, milling became faster, but adjacent curves became closer and closer together. The "inversion" shown in the Fig. 3 as being typical of the lead chrome pigments did not occur with most of the organic pigments until impracticably low pigment concentrations were reached. Under these circumstances, therefore, it was necessary to choose the mill base giving the best combination of results rather than the one which gave the fastest dispersion, because, from the point of view of the paint manufacturer, it is throughput of dispersed pigment which is important.

The explanation for these two types of results can almost certainly be found by considering the different behaviour of the two groups of pigments in a Daniel flow test. The inorganic lead chrome pigments give a very sharp end

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point, so that if the pigment concentration is a little higher than a certain level the mill base is too thick to disperse satisfactorily, but if the concentration is a little lower than this level the mill base is too thin to disperse satisfactorily. Most organic pigments give poor end points so that when a pigment/resin slurry just showing flow is obtained, a significant further quantity of resin solution may be added before the mixture becomes too thin to disperse satisfactorily.

The optimum pigment concentration (as opposed to the concentration which dispersed most rapidly) was considered to be that at which the mill base (a) did not give shock-seeding when concentrated resin was added after dispersion, (b) dispersed to give a good grinding gauge reading in a reasonable time, and (c) gave reasonable flow after let down.

The word "reasonable" has been used deliberately. It is not possible to quote acceptable figures for time of dispersion or for flow. What is considered to be a reasonable time of dispersion for a complex polycyclic pigment would not be considered a reasonable time for an arylamide yellow which is characteristically easy to disperse. Similarly, what is considered a reasonable flow for a lead chrome pigment would be unattainable in a benzidine yellow or phthalocyanine blue pigment.

In addition to recording optimum pigment concentration for dispersion in 20 per cent *Paralac 10* solution, the pigments were also classified according to their ease of dispersion. In this case only the ease of dispersion of the preferred mill base was considered and the time in minutes taken for this mill base to reach a seed level of 7.5 microns was noted. This level was chosen as a compromise for both brushing and spraying finishes, the criterion for the former normally being considered to be approximately 10 microns and for the latter approximately 5 microns. To give the time of dispersion in minutes might have been confusing when relating to normal ball-milling practice and, therefore, the pigments were classified on a 1-5 scale according to the time taken. The breakdown of these times is given in table 2, and if one minute's

Numerical assessment
5
4
3
2
1

Table 2	
Dispersibility	scale

dispersion on the *Red Devil Paint Conditioner* is approximately equal to one hour in a large-scale ball-mill, 15 minutes' milling on the sma¹l scale will be similar to an overnight milling on the large scale.

Selected results

Consideration of all the data obtained when this type of test had been carried out with the whole range of pigments showed how extremely important correct composition of the mill base is in ball-milling. Relatively small differences in pigment concentration, with certain products, had a very marked effect on speed of dispersion. The results also showed how difficult it was to predict a reasonable formulation on the basis of other pigment properties. The following two examples illustrate this.

The first is a comparison between a solvent stable, α form, copper phthalocyanine (*Monastral Fast Blue LBXS* from Imperial Chemical Industries Ltd., CI Pigment Blue No. 15) and a surface treated form of this product (*Monastral Fast Blue RFS*). Technologically, *Monastral Fast Blue RFS* differs from *Monastral Fast Blue LBXS* in that : (a) it has much greater resistance to flocculation, (b) it is weaker tinctorially (150 parts *RFS*=100 parts *LBXS*), and (c) it gives mill bases and inks of much better flow, even when account is taken of its lower strength.

It might have been anticipated that the mill base of *Monastral Fast Blue RFS* should have contained 50 per cent more pigment than that of *Monastral Fast Blue LBXS* in view of the tinctorial relationship between the two products, and it could have been argued that the difference should have been even greater than this because of the superior flow of the flocculation resistant brand. However, this was not the case.

The graphs of seed level against time for different mill bases of these two products are shown in Fig. 5. Twenty per cent has been chosen as the optimum pigment concentration for *Monastral Fast Blue LBXS*; there is no shock-seeding with this composition, dispersion takes place at a reasonable rate and efficiency drops at lower pigment concentrations. At 30 per cent *Monastral Fast Blue RFS* (that is the concentration equivalent tinctorially to 20 per cent *Monastral Fast Blue LBXS*) it was found that an "off-the-gauge" reading was only obtained after 30 minutes, and at the higher concentrations needed to give similar flow to 20 per cent *Monastral Fast Blue LBXS*, milling was even slower. It is clear that *Monastral Fast Blue LBXS* has much better flow under the high rate of shear in the ball-mill than its flow under low rates of shear suggests. For *Monastral Fast Blue RFS*, 20 per cent was chosen as the pigment concentration giving the best compromise between rate of dispersion and throughput, that is, the *same* concentration as that recommended for *Monastral Fast Blue LBXS*.

The second example of the difficulty of predicting mill base compositions is given by the comparison of two phthalocyanine greens, *Monastral Fast Green GNS* (CI Pigment Green No. 7) and *Monastral Fast Green 6YS* (CI Pigment Green No. 41), pigments of very similar technological properties, differing only in shade. The graphs for both of these products are shown in Fig. 6. *Monastral Fast Green GNS* gave what might be described as a typical "organic pigment" set of curves. As the pigment concentration was lowered



Fig. 5. (top) Dispersion of two phthalocyanine blues (CI Pigment Blue No. 15) in a 20 per cent solution in white spirit of an alkyd resin (Paralac 10); Monastral Fast Blue LBXS: solvent stable, alpha form. Monastral Fast Blue RFS : surface modified form of Monastral Fast Blue LBXS

Fig. 6. (bottom) Dispersion of two phthalocyanine greens in a 20 per cent solution in white spirit of an alkyd resin (Paralac 10); Monastral Fast Green GNS : CI Pigment Green No. 7. Monastral Fast Green 6YS : CI Pigment Green No. 41 from 40 to 20 per cent the curves become progressively closer to each other and speed of dispersion increases. The optimum pigment concentration was set at 20 per cent.

Monastral Fast Green 6 YS, on the other hand, gave what has been described as a typical "inorganic pigment" set of curves. As the pigment concentration was reduced from 45 to 30 per cent there was a significant increase in speed of dispersion, but further reduction in pigment concentration resulted in a drop in the speed of milling. There was little difficulty in choosing 30 per cent as the optimum pigment concentration for *Monastral Fast Green 6 YS*, at which concentration it was dispersed more rapidly than 20 per cent *Monastral Fast Green GNS*. If both products were compared at 20 per cent concentration, the *Monastral Fast Green GNS* dispersed more rapidly.

It must be emphasised that these results refer only to ball-milling and do not give any guide as to relative ease of dispersion by other techniques. For example, on a three-roll mill, *Monastral Fast Green GNS* is dispersed more easily than *Monastral Fast Green 6YS*.

In table 3 are given the optimum mill base concentrations of a selection of products together with times taken to disperse these mill bases to a seed

Product name	Generic name and CI ref	Ball-mill charge			Time to	Flow of
Froduct name	Generic name and Criter.	Pigment	Solid resin	Solvent	7.5 microns (min.)	base (g/15 min.)
Monolite Fast Yellow GNS	Arylamide Yellow G, CI Pigment Yellow No. 1	25	15	60	9	11
Monolite Yellow GTS	Benzidine Yellow, CI Pigment Yellow No. 12	20	16	64	9	4
Monolite Scarlet RBS	Toluidine Red, similar to CI Pigment Red No. 3	30	14	56	11	28
Monolite Red 4RHS	Arylamide Red, CI Pigment Red No. 7	20	16	64	8	9
Rubine Toner 2BS	Calcium 2B Toner, CI Pigment Red No. 48	20	16	64	7	4
Monolite Fast Blue 3RS	Indanthrone, CI Vat Blue No. 4	20	16	64	9	39
Monastral Fast Blue LBXS	Phthalocyanine Blue, CI Pigment Blue No. 15	20	16	64	9	3
Monastral Fast Blue RFS	Phthalocyanine Blue (flocculation resistant), modified CI Pigment Blue No. 15	20	16	64	12	31
Monastral Fast Green GNS	Phthalocyanine Green, CI Pigment Green No. 7	20	16	64	10	32
Monastral Fast Green 6YS	Phthalocyanine Green, CI Pigment Green No. 41	30	14	56	7	30
Pure Middle Chrome LGS	Middle Chrome, CI Pigment Yellow No. 34	70	6	24	Less than 5	67
Pure Scarlet Chrome LYS	Molybdate Orange, CI Pigment Red No. 104	70	6	24	8	64

 Table 3

 Formulation and dispersibility of ball-mill bases and flow after let-down

level of 7.5 microns and the amounts of let-down mill bases discharged in 15 minutes. This gives some indication of the way these factors can vary from one product to another. The large differences in flow between the let-down mill bases of different chemical types of pigment are of particular interest.

All the work described in this paper has been concerned with the dispersion of single pigments, but, so far, it has been found that satisfactory conditions for ball-milling mixtures of pigments can be determined arithmetically knowing the optimum conditions for dispersion of the separate components.

Effect of variation of resin solids

As stated earlier, the complete range of pigments being investigated was tested for dispersion in a 20 per cent solution in white spirit of *Paralac 10* because it was not practicable to investigate variations in both pigment/resin ratio and resin/solvent ratio. Detailed tests were carried out with a few pigments to determine the effect on dispersion of using media of varying solids content and these confirm that 20 per cent resin solids represents a suitable level.

As shown in Fig. 7, for example, a mill base containing 40 per cent of an Arylamide Yellow 10G, *Monolite Fast Yellow 10GS* (CI Pigment Yellow No. 3), was dispersed to an "off-the-gauge" reading in the same period of time using as medium 15, 20 or 25 per cent solutions of *Paralac 10* in white spirit. Above 25 per cent resin solids the speed of dispersion was slightly less, but at 10 per cent resin solids an "off-the-gauge" reading was not obtained in 30 minutes unless the pigmentation was reduced from 40 to 25 per cent.

Similar results were obtained with a phthalocyanine blue, *Monastral Fast Blue LBXS* (CI Pigment Blue No. 15), except that 20 per cent resin solids gave results a little better than 15 and 25 per cent.

A Toluidine Red, *Monolite Fast Scarlet RNS* (CI Pigment Red No. 3), behaved rather differently. At the preferred pigmentation (35 per cent), speed of dispersion increased steadily as the resin concentration was lowered from 25 to 2.5 per cent, the lowest tested. In white spirit alone, however, it was impossible to disperse the pigment. Obviously the resin requirement of this product is very low, although still positive. It is not known how many other pigments fall into this category.

In drawing these graphs, the time taken to give an "off-the-gauge" reading was deliberately chosen as the measure of efficiency of dispersion, because it emphasised the effect of changing resin solids. Very often the seed level curves flattened out just above 5 microns and this was not considered to be efficient dispersion, even though the pigment may have been sufficiently finely divided so as not to give "bits" in the finished paint.

It is not possible to give all the results obtained, but it is interesting to look at the "bunching" of the curves at different resin concentrations. Around the 20 per cent resin solids mark, the curves for different pigment concentrations are generally quite close together, but the further above or below this, the more the curves separate. Thus, as shown in Fig. 8, the nearer the optimum resin solids the less critical the pigment concentration.



Fig. 7. Effect of variation of concentration of alkyd resin solution (Paralac 10 in white spirit) on speed of dispersion; Monolite Fast Yellow 10GS: CI Pigment Yellow No. 3. Monastral Fast Blue LBXS : CI Pigment Blue No. 15. Monolite Fast Scarlet RNS : CI Pigment Red No. 3



Fig. 8. Dispersion of Arylamide Yellow 10G (Monolite Fast Yellow 10GS, CI Pigment Yellow No. 3) in alkyd resin solutions (Paralac 10 in white spirit) of different concentrations



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It is normal to refer to pigment/binder ratios when discussing the concentration of pigment in a finished paint or in a paint film and this ratio is often quoted when the formulation of ball-mill bases is being described. It is suggested, however, that if the optimum mill base for a certain product contains

			Parts
Pigment		 	 20
Solid resin	• •	 	 16
Solvent		 	 64

it is better to say that "this pigment is dispersed at 20 per cent pigment concentration in a 20 per cent solution of resin," rather than "this pigment is dispersed at a 1.25 : 1 pigment/binder ratio in a 20 per cent solution of resin."

The advantages of using "pigment concentrations" rather than "pigment/ binder ratios" are very apparent when variations in resin solids are being investigated. First, as the resin solids of the grinding medium is changed, it is surprising how slowly the relationship between the behaviour of various "pigment concentrations" changes, whereas in terms of "pigment/binder ratios" the changes are very great. For instance, in table 4, it is shown that

Table 4
Description of mill-base compositions
Monastral Fast Blue LBXS (C1 Pigment Blue No. 15)

Resin solids (%)	Optimum mill-base composition		
	Expre (a) % Pigment concentration	ssed as (b) Pigment : Binder ratio	
30	20	0.8:1	
25	20	1.0 : 1	
20	20	1.2 : 1	
15	20	1.7:1	
10	20	2.5 : 1	

as the resin solids of the grinding medium is changed from 10 to 30 per cent, the optimum pigment concentration for dispersing *Monastral Fast Blue LBXS* is unchanged at 20 per cent. However, the pigment/binder ratios of these same mill bases vary from 2.5:1 down to 0.8:1. Secondly, "pigment concentrations" give the manufacturer a more immediate indication of the throughput of a mill than "pigment/binder ratios."

Rate of strength development

In all the work so far described the efficiency of milling has been judged solely by seed level measurement, a simple test quickly carried out with a very small quantity of mill base. However, from the technological point of view the only direct significance of this test is that if the reading is not more than 10 microns in a brushing finish or not more than 5 microns in a spraying finish, the dried film will not show "bits." From this test it is not possible to judge the tinctorial properties, the flow properties or the gloss properties of a finish. In strong shades the last two features can be considerably affected by the coloured pigment used and its state of dispersion, but in shades containing a high proportion of white the paint manufacturer is usually concerned (after the prime need to produce a "bit-free" finish) with the strength given by the coloured pigment.

The basic experiment already described was modified so that rate of strength development could be investigated, again with the minimum amount of supervision.

First, the optimum mill base formulation was determined in the usual way. Then six 4 oz bottles each charged with this formulation were put on the *Red Devil Paint Conditioner* and dispersion commenced. The bottles were successively removed from the mill after 2, 5, 10, 20, 40 and 80 minutes and each was let-down with concentrated resin solution until the bottle was approximately 80 per cent filled. All six bottles were then thoroughly stirred with a glass rod.

Then, according to the pigment concentration of the stainer and the depth of shade of interest, a certain weight from each bottle was mixed with a fixed weight of standard white paint (the quantities involved were usually of the order of 2g of stainer and 10g of white paint). Although this operation seemed to be simple, it was not, and after attempting many different techniques the use of mechanical agitation was reluctantly abandoned. It was found that the most reproducible method was to add the stainer to the white paint in a shallow round-bottom glass dish and to stir by hand with a glass rod, continuing until the mixture was visually homogeneous. A pattern of each reduction was then made by draw-down technique, using a 0.003 in applicator bar, on card coated with clear nitrocellulose lacquer.

To obtain the fullest information, the six paints obtained should have been made up with varying known ratios of colour/white until patterns of equal visual strength were obtained. The relative strengths would then have been known quantitatively. This, however, would have been very time consuming and in most cases would not have been justified. The rate of strength development of any pigment is very high at the beginning of milling and then falls off, and the two important points to be ascertained are :

(a) The period of milling after which the rate of strength development becomes so low that it is uneconomical to continue, and

(b) The strength obtained at this point.

This information is given by simple visual examination of the patterns produced as described above. A graphical representation of strength development can be obtained by plotting the inverse of Luminance (1/Y), measured on a *Colormaster*, against time of milling. Care must be taken not to compare the 1/Y values of one set of patterns with those of a set prepared from another pigment, but for each product they can be used as a measure of visual strength, provided that there is no change of shade on milling.

Fig. 9 shows such curves for two phthalocyanine greens and a red vat pigment. It will be noted that the seed level for each point is given in parentheses. The graphs are similar in general shape and what might be called the reasonable "maximum working strength" is achieved at a seed level of just above 5 microns with *Monastral Fast Green GNS* (CI Pigment Green No. 7) and at the first "off-the-gauge" reading with *Monastral Fast Green 6YS* (CI Pigment Green No. 41) and *Monolite Fast Red YS* (CI Vat Orange No. 3). The pigments were milled at the appropriate optimum concentrations, determined by the test described in the early part of the paper.



Fig. 9. Rate of strength development—1 (seed levels in parentheses). Monastral Fast Green GNS : CI Pigment Green No. 7. Monastral Fast Green 6YS : CI Pigment Green No. 41. Monolite Fast Red YS : CI Vat Orange No. 3

Fig. 10 shows the strength development curves for a blue vat pigment (CI Vat Blue No. 4) and a Toluidine Red, *Monolite Fast Scarlet RNS* (CI Pigment Red No. 3), and it can be seen that both products continue to develop strength after the seed level readings have fallen below 5 microns. In these cases the grinding gauge readings can only be used as a measure of tinctorial strength above 5 microns. Toluidine Reds are normally used in self shades and development of gloss is therefore more important than rate of strength development. Further experiments showed that maximum gloss was achieved much more rapidly than maximum strength.

Although it is not known how many pigments continue to develop strength after "off-the-gauge" seed level readings are reached, it is clear that in most, if not all, cases quite significant strength increases are to be expected after readings of 10-15 microns have been attained. In many factories it is common

January



Fig. 10. Rate of strength development—2 (seed levels in parentheses). Monolite Fast Blue 3RS : CI Vat Blue No. 4. Monolite Fast Scarlet RNS : CI Pigment Red No. 3

practice to continue dispersion until the seed level falls below a certain figure, usually about 10 microns for decorative finishes, rather than continue until a certain strength is achieved. Although cost of further dispersion has to be considered in relation to the extra strength to be obtained and it is not necessarily advantageous that maximum strength should be developed, nevertheless some preliminary investigation could save manufacturers a significant proportion of their pigmentation costs.

Just as the earlier investigations showed the difficulty of predicting mill base formulations without actual tests, so no reason could be found to explain why some pigments continued to develop strength after reaching 5 microns while others did not. In Fig. 11 are shown the strength development curves of two phthalocyanine blues. The unmodified solvent stable α form developed maximum strength at about 5 microns seed level, but in the particular medium used (*Paralac 10* in white spirit) the flocculation resistant brand continued to develop strength steadily after the 5 microns level had been reached. It was known that this difficulty was not encountered in stoving media and so the effect of modifying the solvent was studied. As the dotted line graph shows, the use of a 3 : 1 white spirit/xylene mixture instead of white spirit alone made a significant difference, maximum strength being developed much more quickly, in fact in about one-fifth of the time taken when only white spirit was used.



Fig. 11. Rate of strength development—3 (seed levels in parentheses). Twenty parts phthalocyanine blue : 80 parts 20 per cent alkyd resin (Paralac 10). Monastral Fast Blue LBXS : CI Pigment Blue No. 15. Monastral Fast Blue RFS : Modified CI Pigment Blue No. 15

Variation in type of resin

The last experiment shows that considerable changes in dispersion characteristics may be expected if only the solvent is changed. If the resin is also varied, the overall picture becomes very complex. To date only a limited investigation has been made of this part of the field, and the work has been confined to phthalocyanine blues and greens.

When the resin was changed from *Paralac 10* to a medium oil length linseed oil alkyd resin (*Paralac 11*), again using white spirit as solvent, there was little change in behaviour.

However, when a short-medium oil length drying castor oil alkyd resin (*Paralac 64*) was used, with xylene as solvent, phthalocyanine blue has to be milled at a lower concentration than usual. In concentrated solution, *Paralac 64* is much more viscous than *Paralac 10*, and although dilute freely flowing resin solutions were used, the viscosity of these dilute solutions of *Paralac 64* rapidly built up as pigment was added.

Phthalocyanine green dispersed rapidly in a xylene solution of *Paralac 64*, but was then very sensitive to shock-seeding. This could be overcome if part of the xylene was replaced by butanol, although the speed of dispersion was somewhat reduced.

A similar situation arose with phthalocyanine blue in a solution in xylene/ butanol of a urea-formaldehyde resin (*Paralac 6001*). When xylene was used for further dilution of the resin, dispersion was very rapid, "off-the-gauge" readings being obtained in five minutes, but it was difficult to prevent shock seeding, and again the best performance was obtained with a xylene/butanol combination as solvent. Phthalocyanine green also dispersed more rapidly in *Paralac 6001* if xylene/butanol mixtures, rather than butanol alone, were used for dilution of the resin.

Conclusions

Future investigation in this field can hardly be carried out on a broad front because possible variations in resin and solvent combinations are infinite. However, in any one particular system, the resins, solvents and pigments are known and the investigational work involved in determining optimum ball-milling conditions is not excessive using the general technique described and the information now available. Most of the further work is likely to be concerned with individual problems and it is hoped that this paper has given some indication of how they may be tackled. It must be emphasised that most of these will be, very literally, *individual* problems, and one's experience can usually only be applied to reduce the amount of preliminary experimental work necessary rather than to eliminate it completely.

It has often been assumed that ball-milling at higher pigmentation and lower resin solids levels than those of the finished paint was advisable in order to disperse more pigment in each operation of the mill. This is less than half the story; not only is more pigment put in the mill but the larger load is dispersed much more rapidly than the smaller one. Indeed in many cases the smaller load might never be completely dispersed. So often at other than optimum pigmentations, graphs that flattened out at about 10-15 microns were obtained, and even if milling time was increased many-fold the seed level hardly moved from that figure. Any manufacturer operating under these conditions is losing pigment strength, is wasting ball-mill capacity and probably producing " bitty" paint. The aim of this paper has been to show that the preliminary experimental work necessary to prevent this situation is not prohibitively great either in terms of time or effort and will rapidly show dividends.

Acknowledgment

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Discussion at London Section

MR. M. R. MILLS asked if any work had been done on the effect of surface active agents on the optimum pigment concentration and flow rate.

MR. H. G. COOK replied that in the experiments described there had been no additions of wetting agents or dispersing aids. He added that if he was asked about

the value of such agents he always suggested that there should be certainty that tests without these agents had been carried out under optimum conditions. Change of pigment concentration could sometimes increase speed of milling tremendously and this feature only had been investigated.

MR. J. D. COHEN said that the improvement of the dispersion of phthalocyanine blue by replacement of white spirit by xylene, resin and pigment conditions being equal, was very interesting, and asked Mr. Cook to comment further on the effect of the solvent on dispersion.

MR. COOK said that work was limited on this subject, but xylene seemed to speed dispersion fantastically. The results shown were the effect of using a mixture of three parts white spirit to one of xylene. It was almost impossible to change to 100 per cent xylene and prevent shock seeding and this was presumably due to the increase in the speed of wetting of the pigment. No detailed theoretical explanation could at present be offered for this.

MR. J. A. L. HAWKEY suggested that since comparatively weak solutions of resin were used and heterogeneous material was involved at some stage of the dispersion, some part of the alkyd on which solution depended might be removed by the pigment surface and the resin might seed out. He thought that this might be an explanation of the improved dispersion with aromatic solvent since the dilution ratio would be higher.

MR. COOK agreed that this was possible. No difficulty had been experienced with the major investigation which was carried out with alkyd resin at a concentration of 20 per cent. At very low concentrations care had been taken to use fresh resin solutions.

MR. W. O. NUTT asked whether any attempt had been made to compare the rheology of the various pigment/resin "preferred concentration" mixtures at the rate of shear induced by the experimental ball-mill apparatus. In this event would it not be found that the viscosity/thixotropy characteristics would be similar for each different pigment/resin "preferred concentration" and would it not be the case therefore that the "preferred concentration" was a characteristic dependent on the pigment particle surface/resin interface properties?

MR. COOK replied that this would certainly be true if the exact conditions of shear in the ball-mill were known. He was sure that viscosity at that rate of shear would give more or less the same answer as had been obtained. The possibility of doing this had been considered, but had to be rejected in the face of practical difficulties and lack of knowledge of the rate of shear applicable. That the flocculation resistant blue gave better flow than the "normal" brand was obvious to anyone who had used both pigments. However, this improved flow at relatively low rates of shear was apparently of no significance as far as behaviour in the ball-mill was concerned. This showed that the main fault with the normal phthalocyanine blue was not poor flow but thixotropy. It had also been hoped to use viscosity measurement to determine ease of discharge from the mill rather than the empirical measurement of weight poured out of a bottle in 15 minutes, but again it was not known at what rate of shear the measurements should be made.

MR. K. POND asked whether Mr. Cook thought that thixotropic behaviour could confuse the determination of seed levels.

MR. COOK thought that this might be so, but that it was right to take the high figure as this would be the significant one. He added that the assistant who carried out the work had commented that he could tell from the type of seed level reading (i.e. whether it was sharp or diffuse) whether the material would mill satisfactorily. No attempt was made to correlate this with flocculation.

MR. J. W. BLAKEWAY asked what conclusion could be drawn from similar work on parallel roll mills.

MR. COOK said that naturally viscosity affected dispersion on a three-roll mill, but said that the results described did not apply to the conditions of shear or resin concentrations used on a three-roll mill. He suggested that the same general method of attack on the problem (i.e. the investigation of a range of concentrations for each pigment) would be valuable.

DR. S. H. BELL commented favourably on the preference shown by Mr. Cook for the term "seed level" to "fineness of grind" for gauge readings. In the case of pigments which showed further development of tint on grinding beyond the gauge readings which were taken as an end point, he pointed out that the tests had been done at the particular concentration selected from others in the preliminary work by using the gauge criterion. He asked whether the same concentration would have been obtained as being the most favourable one if the original criterion had not been based on the gauge reading but on tint strength development.

MR. COOK replied that with a number of pigments, decrease in gauge reading and increase in tinting strength did correlate, i.e. both sets of curves flattened out together. A direct check was made on only one pigment which did continue to develop strength after 5 microns, i.e. the "gauge" and "strength" curves separated. A number of concentrations was tested for strength development and seed elimination (dispersion was stopped at 80 minutes, but the strength was levelling out before that time). With this pigment the optimum concentrations chosen by "seed elimination" and by "strength development" were the same.

DR. L. VALENTINE asked how many inorganic pigments showed the "inorganic" pattern of behaviour in establishing the optimum milling conditions and noted that there seemed to be a few organic pigments that showed "inorganic behaviour." This seemed to tie up with Daniels' flow-point behaviour.

MR. COOK agreed that the results tied up with flow. With many inorganic pigments, the rate of dispersion was slow at both high and low concentrations, increasing rapidly to a maximum at the optimum concentration. This "inorganic" behaviour was obtained with chrome pigments, prussian blue and titanium dioxide, but not from yellow iron oxide, which behaved more like an organic pigment.

MR. COHEN commented that in the printing ink industry it was more customary to refer to "rocks" rather than "seeds."

MR. COOK responded that the same type of procedure could be used for gravure ink preparation, but the type of solvents used made for difficulties.

MR. R. F. KEY asked for more details of the Red Devil machine.

MR. COOK said that it was a paint conditioner on which fairly severe agitation was obtained so that water could be boiled on it in a few minutes without much difficulty if the vessel were insulated. It would recondition paint stored for about three years in about 5-10 minutes.

DR. BELL said that the lecturer had mentioned some work on grinding by his method of mixed pigments and had said that the best conditions were much as would be expected by an arithmetic calculation from the grinding of the two pigment components separately. He asked whether a similar result would have been obtained in the case of a titanium dioxide/phthalocyanine blue mixture, for example, if the criterion had been based on flotation effects in the finished materials rather than on gauge readings.

MR. COOK agreed that different states of flotation might be obtained under different dispersion conditions. This factor had not been investigated and it would have been difficult to do so.

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MR. NUTT asked if the "preferred concentration" mixture derived from the described range of examinations did not relate specifically to the rate of shear induced by the particular equipment used. He suggested that a totally different "preferred concentration" mixture would apply to equipment which induced a different shear rate. Finally he asked how valuable the information derived was in terms of the wide variation in performance of commercial ball-mills.

MR. COOK agreed that, on theoretical grounds, Mr. Nutt was probably right, but what evidence there was showed correlation between the *Red Devil* and large-scale ball-mills. As stated in the paper, Orwig had reported that one minute on the *Red Devil* was equivalent to one hour in a large ball-mill. In a small number of experiments Mr. Cook found that $1\frac{1}{2}$ minutes on the *Red Devil* was equivalent to one hour in a one-gallon mill, that is, not the same as, but similar to Orwig's results, suggesting that from a practical point of view there may not be much difference between different sized mills.

MR. A. T. S. RUDRAM asked whether the temperature developed in this type of milling would be expected to affect the shade and strength of the more soluble types of organic pigments such as arylamides due to particle growth.

MR. COOK replied that this had not been found in the work described in the paper. He thought that this feature was likely to be specific to the particular conditions of use and would affect shade and strength rather than seed elimination.

MR. D. F. BOWERMAN inquired if Mr. Cook had any comments on the development of gloss, particularly for pigments normally used near full colour.

MR. COOK replied that an experiment was carried out with toluidine red. This developed strength slowly in comparison with rate of seed elimination. However, rate of gloss development was faster than rate of seed elimination and maximum gloss was reached before an "off the gauge " reading.

MR. HAWKEY would have liked to hear some explanation of the difference in behaviour between what had been described as "inorganic" and "organic" behaviour. If some of the resin was immobilised in the pigment surface by adsorption during dispersion, he asked whether it would be possible that the ratio of pigment diameter to pigment diameter plus adsorbed layer would vary much from pigment to pigment and if the effective pigment volume would be changing during dispersion.

MR. MILLS asked whether the concentration of pigment used in the experiments had been expressed in terms of volume rather than weight and noted that the pigments used varied considerably in specific gravity.

MR. COOK said that it seemed that he was going to be forced to admit that differentiation of pigment behaviour into "inorganic" and "organic" was not really justified. He was unable to comment on the point concerning "effective pigment volume," but suspected that switching of resins and solvents would also affect this behaviour. The concentrations of pigment had been expressed by *weight* but, throughout, *volume* of resin+*volume* of pigment was kept constant.

Mr. Cook stated in a further communication that some further comment was called for concerning "organic" and "inorganic" behaviour on dispersion. It had to be made clear that basically there was no difference between these groups. For *every* pigment a point was reached where a further drop in concentration resulted in a decrease in milling time. With the inorganic pigments listed in the answer to Dr. Valentine's question, this "inversion" took place sharply over a narrow range of pigment concentrations. Some organic pigments behaved like this, but for most of them the "inversion" took place slowly over a wide range of pigment concentrations, and the concentration at which most rapid dispersion occurred was normally too low to be recommended for large-scale use. MR. KEY asked whether the Guggenheim equation was of use in determination of formulation.

MR. COOK replied that it had not been used.

MR. T. P. MAJOR commented that a certain phthalocyanine green had a tendency to exhibit development of strength on dispersion due to increase in temperature. He commented further on the statement that solvent additions had more effect than resin additions and asked if that would be true when using an isophthalic alkyd.

MR. COOK stated that he was not aware of such behaviour in a phthalocyanine green and had not examined isophthalic alkyds. However, it should be possible to explain the solvent *versus* resin effects on theoretical grounds. He thought that in many properties resins were more similar to one another than solvents were. If temperature was increased the rate of milling would also be increased, hence the rate of strength development.

MR. C. A. CAREY noted that it had been said that the flocculation resistant blue was a surface coated pigment and suggested that this surface coating probably gave surface active properties to the pigment, and therefore in considering the two phthalocyanine blues the conditions were different from those with other pigments.

MR. COOK replied that this was so, but that although the flocculation resistant blue was surface coated, the coating conferred surface inactivity as far as dispersion was concerned in that it milled more slowly than the normal blue. It was obvious that modifications to the surface of a pigment could have considerable effect (either way) and differences should be anticipated. The resin used in the experiments described had extremely good wetting powers and no dispersing aids were necessary therefore. With other resins, still greater differences might be obtained.

MR. I. C. R. BEWS, in proposing a vote of thanks, congratulated the lecturer on presenting the work in a truly logical and reasonable way just as the problem was tackled.

Discussion at Manchester Section

MR. J. SMETHURST, from the chair, said he would like to ask a question with regard to the mill base and the relative question of flow. Mr. Cook had said that another pigment which was chemically identical to one described would be different in its flow properties or could be. He asked whether the flow variations from batch to batch of one particular pigment were large.

MR. COOK replied that this was not so with the pigments examined.

MR. J. MITCHELL commented that the addition of xylol had been shown to improve the ease of dispersion of some of the treated phthalocyanine blue pigments. Following on this, he asked whether there was any evidence to suggest that the use of an aromatic white spirit would lead to similar improvements in the manufacture of decorative enamel paints.

MR. COOK said that, so far as the effect of solvent was concerned, the investigation had been limited to phthalocyanine blues and greens. The effect did depend on the concentration of xylene. The white spirit that was used was not "aromatic-free" and, in fact, contained 15-20 per cent aromatic solvents. To obtain faster milling it was still necessary to add xylene. He felt sure that a similar situation would exist in other groups of pigments.

MR. J. MACKINLAY asked if Mr. Cook could comment on the effect of subsequent *Red Devil* shaking on a dispersion which had reached its optimum performance by ball-milling.

MR. COOK said that if a dispersion was prepared in a laboratory ball-mill and then put on the *Red Devil* it was usually possible to get a strength increase. This was because of the difficulty of obtaining maximum strength in a laboratory ball-mill. Even with large-scale ball-milling the same thing could happen, particularly when the mill base had not been properly formulated. If one minute's milling on the *Red Devil* was as suggested equal to one hour in a ball-mill, it was easy to see how the long milling times occasionally reported in practice arose.

MR. M. BELL inquired whether flooding and flotation had been taken into account when measuring colour developments.

MR. COOK replied that although these effects had not been taken into account the conditions used throughout were strictly comparative and were such as to reduce flooding, floating and flocculation. The finishes produced were of relatively high viscosity and films were prepared by a drawdown technique, not by brushing.

MR. A. R. H. TAWN commented that it was interesting to note the efforts on the part of the lecturer to avoid any comparison of shear rate for the blue and the white. The method described used very little shear to produce reproducible results. He asked whether it was a laboratory method using mechanical stirrers or vigorous hand stirring in a beaker. He wondered whether the reason for this was a question of flooding with phthalocyanine blue at fairly high concentration and then adding a white suspension. He asked whether Mr. Cook was not frightened of reducing the particle size of the blue pigment or of increasing the tinctorial strength by stirring in beaker or brushing.

MR. COOK replied that actually the reverse of what the questioner had said was true. It had been seen how efficient the *Red Devil* was for agitation and yet when 2 grams of stainer and 10 grams of white paint were mixed on the *Red Devil* with glass balls for five minutes, this did not give satisfactory mixing. It was still possible to see streaks in the mix.

Various mechanical methods of stirring had been tried in the laboratory, both low and high speed, but the mixing action was still not satisfactory. It was surprising how much mixing took place in a dish with a glass rod, mainly because it was possible to see where the stainer had not mixed in properly and to concentrate on these regions. It should be remembered that *let-down* mill base and *finished* white paint were being mixed and under these conditions, even with high shear action, little or no extra dispersion was obtained.

MR. S. DUCKWORTH inquired whether the strength development was identical at any seed level even with different pigment concentrations and different vehicle solids concentrations.

MR. COOK said that with a toluidine red some investigation had been made to see if the first "off-the-gauge" readings corresponded to similar strengths when millings were carried out at different pigment concentrations. It had been found that, in fact, they did. No further work had been done on this aspect. In reply to a supplementary question, Mr. Cook said that further milling beyond the "off-thegauge" time always gave an increase in strength, but this was similar for different pigment concentrations. Parallel experiments with varying resin solids or with varying solvents had not yet been carried out.

MR. J. MITCHELL said the lecturer had indicated that the optimum conditions for ball-milling a Hansa Yellow 10G had been worked out and asked whether he had anything to add to indicate the strength development of the Hansa Yellow in these various ball-mill pastes. He wondered if Mr. Cook could comment further on the conditions which would lead to optimum strength development in ball-mill pastes of this type.

MR. COOK answered that it had been found that to measure the strength development of the Arylamide Yellow 10G was extremely difficult. Strength development was very rapid but, probably because the pigment was so sensitive to contamination and also because the Y values varied so little with strength increase, it had not been possible to obtain such smooth curves as with other pigments.

MR. J. J. KAVANAGH inquired whether the efficiency of dispersion fell off if a lower viscosity alkyd of the same type as *Paralac 11* was used which required a higher resin content in the mill.

MR. COOK said he felt that if the two resins were used at the same concentration this would be true, although different resins showing very different viscosities at higher concentrations had more similar viscosities at lower levels. If the resin solids were adjusted to give equal viscosities, however, he said he would hesitate to predict the result.

MR. H. F. CLAY asked if the *Red Devil* apparatus could be used with sand for evaluating pigments for use with the Sand Grinder.

MR. COOK answered that it was possible to grind with any type of grinding media. Glass balls had been chosen because they gave particularly good speed of dispersion, were a lot easier to clean than sand, and did not affect colour as steel balls would have done. He said that to determine a suitable formulation for a sand-mill he would certainly recommend a trial of this nature on the *Red Devil*, using 30-40 per cent resin, which would very quickly provide a range of concentrations for investigation on the sand-mill. It was advisable to use the *Red Devil* for such preliminary work and then to use the sand-mill for final formulation of mill base. The *Red Devil* could save a lot of time and trouble used in this way.

MR. H. R. TOUCHIN asked if Mr. Cook had compared the results obtained on the *Red Devil* with the Daniel flow-point method and whether the two methods gave the same optimum mill base composition.

MR. COOK replied that they did. One or two coloured pigments and titanium dioxide had been compared at the beginning. The results agreed quite well, but there were limitations to the Daniel flow-point method as had been stated earlier. Two laboratory assistants using the *Red Devil* had been able to do the work necessary to investigate fully about 16 different pigments per week. It would have taken a strong man to do that number of complete Daniel tests.
Metallic lead pigment for anti-corrosive protection

By G. L. E. Wild

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Summary

In a discussion of metallic lead used as an anti-corrosive pigment in paints the chemical properties of the metal are reviewed briefly on the basis of the Periodic table, a Pourbaix pH-Potential diagram and solubility data. Experimental evidence is produced to suggest that metallic lead is a scavenger of atmospheric oxygen and in consequence exercises anticorrosive properties over and above those shown in red lead. The weathering of metal-pigmented paints is discussed briefly with emphasis on the importance of a pigment's own corrosion products and their solubility in water.

Pigment au plomb metallique pour protection anti-corrosive

Résumé

Au cours d'un exposé traitant du plomb métallique utilisé en tant que pigment anti-corrosif pour peintures, les propriétés chimiques du métal sont brièvement décrites sur la base de la Table Périodique, d'un diagramme de "potentiel Hydrogène" Pourbaix et de caractéristiques de solubilité. Des preuves expérimentales sont produites pour suggérer que le plomb métallique refoule l'oxygène atmosphérique et, par conséquent, développe des propriétés anti-corrosives bien supérieures à celles caractérisant le minium. L'altération des peintures pigmentées au métal est commentée brièvement en insistant sur l'importance des propres substances corrosives d'un pigment et leur solubilité dans l'eau.

Die Verwendung von Pigmenten aus metallischem Blei als Korrosionsschutz

Zusammenfassung

Es werden Einzelheiten über die Verwendung von metallischem Blei als Korrosionsschutzmittel in Pigmentfarben angegeben; die chemischen Eigenschaften des Metalls werden im Zusammenhang mit dem Periodischen System, einem Pourbaix-pH-Potential-Diagramm und Angaben über die Löslichkeit erläutert. Aus den angegebenen Untersuchungen kann man schliessen, dass metallisches Blei Luftsauerstoff bindet und deshalb bessere Korrosionsschutzeigenschaften hat als Mennige. Das Verhalten von Pigmentfarben auf Metallgrundlage an der Luft wird kurz erläutert ; dabei werden die pigmenteigenen Korrosionsprodukte und deren Wasserlöslichkeit beschrieben.

Introduction

The use of lead pigments to control the corrosion of iron and steel has been known from ancient times and as a result of much investigation, their method of action is becoming understood more and more. On current views, red lead pigment, for example, is believed to act as an inhibitor towards the anodic reaction in a corrosion cell. Thus red lead pigment has been thought to promote, by its oxidising action, the precipitation of insoluble rust in intimate contact with the steel surface to stifle the anode reaction (rather than in the body of the liquid away from the anode site, as shown in Fig. 1), thus preventing further corrosion. More recently it has been shown by Mayne and van Rooyen¹ that red lead, by reason of its basic nature, can react with acidic scission products from the oxidation of drying oil in priming paints, giving rise to lead soaps of formic, propionic and higher acids. Some of the latter such as azelates, suberates and those of the pelargonate (nonanoate) type are corrosion-inhibiting towards steel, and are thought to act by becoming absorbed on the steel surface.



Fig. 1. Corrosion Cell on unpainted steel

The purpose of this short review of metallic lead used as an anti-corrosive pigment is to show some ways in which it differs from other established lead pigments. Also, after taking a glance at some of the basic physical chemistry concerned, to discuss lines for developing the subject further.

Properties of metallic lead

Lead compounds

The background for metallic lead is conveniently expressed by the Periodic table. Fig. 2 shows the main classes of compounds formed by Group IV elements. The characteristic sp^3 quadrivalency of the Group, giving rise to covalent lead compounds of the Pb (CH₃)₄ type, is not concerned in aqueous corrosion reactions.

The principal aqueous chemistry arises in the II oxidation state with a welldefined series of plumbous salts—indicated in Fig. 2 by $Pb(NO_3)_2$ —into which category fall the lead formate, pelargonate, etc. referred to above. In the IV oxidation state lead in aqueous solution forms complexes such as the indicated $Pb(OH)_6^{2-}$, in which the metal appears in the anion as plumbate.

Water soluble complex anions are also formed in the II state, namely the plumbites, indicated by $(HPbO_2)^-$. There appears to be variability in the

configuration of plumbite ions ; they are recorded in the first column in Fig. 2 for convenience.

P	-					
OXIDATION STATE	п	ъ	IX	ц	IX	IV
VALENCY	2 P _X P _Y plumbite	4 Sp	4 Sp ²	4 5p ³	6 Sp ³ d ²	8 Sp ³ d ⁴
CARBON		СН≡СН	CH2=CH2	C _n H _{2n+2}		
SILICON	(5iCl ₂)			Si ₆ H ₁₄	SiF62-	
GERMANIUM	GeF2 (SOLID)			Ge ₃ H ₈	GeC16 ²⁻	
TIN	SnCl2_ (HSnO2)			Sn (CH3)4	. SnC16	
LEAD	Рь (NO ₃) ₂ (НРЬО ₂)			РЬ(СН ₃) ₄	РЬ(ОН) <mark>6</mark>	TETRA-ACETATE

GROUP IN ELEMENTS

Fig. 2. Periodic Table Group IV. Main classes of compounds

Thermodynamic considerations

Which compound is likely to arise under any given set of experimental conditions can be surmised from thermodynamics.

Pourbaix diagram : Fig. 3 shows the Pourbaix pH-potential diagram² for the closed system metallic lead/water at 25°C. Such diagrams furnish a great deal of information in a small compass. The x-axis shows pH from -1 (10N acid), through the neutral point at 7, to 15(10N alkali). On the y-axis the potential of the system E is expressed in volts according to the IUPAC convention by which the strongly electron-attracting species such as oxygen, are towards the top of the scale with a positive sign and species which easily lose their electrons, such as sodium, are towards the bottom of the scale with a negative sign. Both chemical and electrochemical potentials are reduced to volt units so that they may be shown on the same scale, which is referred to the standard hydrogen electrode as zero. The parallel lines a and b on the diagram separate the region of thermodynamic stability of water. Above line b water tends to become oxidised to oxygen and below line a to become reduced to hydrogen. The other lines mark out the regions of thermodynamic stability of the main compounds which arise in the lead/water system. Pb(OH)2 is not shown but is said² to differ only slightly from PbO. The fine structure of lines on the acid side of the diagram shows the effect of varying concentrations of lead ions, the activity range from normal concentrations (10⁰) to the borderline of insolubility (10⁻⁶) being covered. The line for Pb++ at normal concentration corresponds to the standard



Fig. 3. Pourbaix pH-Potential diagram for the closed system lead/water at 25°C

electrode potential of massive lead namely -0.126 volts; finely divided metallic lead pigment tends to give more negative values.

At any point above this line lead tends to lose two electrons and become oxidised—to plumbous ions in the acid region, to litharge at the neutral point and to biplumbite ions on the alkaline side. At a still higher level of oxidation it loses two more electrons to become respectively plumbic ions, lead dioxide or plumbate ion. Although drawn on the basis of ideal rather than practical systems such diagrams can give some indication as to the favoured reaction under a given set of conditions. Whether or not the reaction proceeds depends on the values of overvoltages and energies of activation concerned.

Conditions under a paint film

Under a paint film in service it is generally considered that pH can vary from approximately 2 on the acid side (from acidic scission products of drying oils)

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to approximately 12 on the alkaline side (from alkali formed by electroendosmosis in sea-water). The general level of atmospheric oxidation is of the order of ± 0.8 volts (e.g. ferrous salts are readily oxidised to ferric salts, $E_0 = \pm 0.771$ volts). In a metallic lead paint in service on steel, therefore, it is seen that a large central area in Fig. 3 may be relevant, and that a variety of lead compounds are likely to become involved. Fig. 3 also shows with clarity that red lead, Pb_3O_4 , tends to be thermodynamically stable at an atmospheric level of oxidation, whereas metallic lead is thermodynamically unstable under such conditions and will tend to become oxidised.

Corrosion inhibition by lead pigments

Results have been published by Lewis and Evans³ and by Mayne⁴ which show that pieces of bright steel immersed in water in the presence of lead pigments—especially litharge, red lead and metallic lead—received protection against corrosion.

The tests were carried out in a number of different ways, in both closed and open containers, including static immersion and agitation. Some of this work has been repeated in the present writer's laboratory and results were in agreement with the authors' findings. The protection afforded by the metallic lead pigment however was outstandingly good and consideration of the conditions suggested that it might be acting, at least in part, by absorbing oxygen from the environment. (If oxygen is eliminated the cathodic reaction shown in Fig. 1 can no longer take place and the anodic corrosion action becomes stifled through lack of electron flow.)

Oxidation of metallic lead pigment

To study this idea further a similar experiment was carried out by agitating pigment and distilled water in a flask with a free air space and connected to a manometer tube to show changes in pressure in the flask. No steel was present in these experiments. Results are given in table 1 below :—

Time in ho	urs	0	6	24	48	72	96
Metallic lead		73.7	63.3	60.9	59.8	59.0	58.6
Litharge		73.7	72.8	72.8	72.8	72.8	72.8
Red lead		73.7	73.3	73.3	73.3	73.3	73.3

 Table 1

 Pressure inside the reaction flask in cms. of mercury

The results are shown graphically in Fig. 4 from which it is clearly see that, except for the small amount of air soluble in the distilled water, there has been no significant change in pressure in the flask when litharge and red lead were used, whereas with metallic lead the pressure has dropped progressively towards the theoretical limit corresponding to complete absorbtion of the oxygen originally present in the flask.



Fig. 4. Oxygen absorption

Oxvgen scavenging

These results suggest the metallic lead/oxygen reaction has a low energy of activation, proceeding easily at room temperature, and will serve to scavenge atmospheric oxygen. This idea, though not new, has received but little attention.

As regards soap formation metallic lead or its oxidation product would react readily with acidic scission products and would be expected to give rise. as in the case of red lead, to the anti-corrosive effect associated with formation of substances such as lead azelate and lead pelargonate in the paint film. Its evident oxygen-scavenging action could however confer corrosion-inhibition properties also under neutral or alkaline conditions, in regions (see Fig. 3) where lead soaps would not be formed.

Metallic lead as an active barrier

To coat a piece of steel with a paint containing metallic lead pigment appears tantamount to protecting the steel with a reactive barrier which absorbs oxygen. This barrier will no doubt function whether an oxidising oil medium or some other type of medium is used in the paint. Combinations such as metallic lead and chlorinated rubber can therefore be expected to give effective corrosion inhibition, despite the absence of soaps, and no doubt the longer the scavenging action persists, the better the paint.

Although such a paint would in any case continue to provide a physical barrier, its reactive oxygen-absorbing life ceases when the metallic lead ceases to be accessible. At this stage in practice active protection would be renewed by application of another coat of metallic lead paint. It is interesting to discuss briefly, however, some of the factors which may be expected to play a part in determining the active life of such a paint.

Solubility of corrosion products

When dealing with paints containing major proportions of metallic pigment it is necessary to consider the resistance of the pigment itself towards corrosion.

Corrosion and passivation

Referring back to Fig. 3 it can be seen that lead ions in aqueous solution can exist at activities up to 10^{-6} (the limit of solubility) over the whole of the central part of the diagram—either as plumbous or plumbite ion or as sparingly soluble plumbous hydroxide (region similar to PbO—see before). It is under the conditions expressed by this part of the diagram that lead will corrode. In the lower part of the diagram metallic lead holds its electrons and is stable, being here in a state corresponding to cathodic protection. In the upper central part of the diagram lead becomes passivated and protected from further corrosion by the deposit of an insoluble coating of PbO₂.

Carbonate and sulphate

Under actual weathering conditions carbon dioxide and sulphur dioxide are present in addition, The Pourbaix diagram for the system lead-water-carbon dioxide² (not depicted here) while essentially similar to Fig. 3 in general outline, shows an extensive region of passivation round the neutral point, where all but slight initial solution of the lead is prevented by a deposit of carbonate (solubility of the order of 10^{-3} gms. per litre). The effectiveness of such passivation is underlined by the case of some water pipes in Bath, installed by the Romans nearly 2,000 years ago, which are said to be still in usable condition.

Lead sulphate is only slightly less insoluble than the carbonate. Such coatings of carbonate and sulphate will therefore tend to remain on the paint film and will not be leached out by ordinary rain-water, as may occur with pigments giving rise to soluble sulphates.

The extent to which such a coating will in time prevent the lead oxidation reaction depends upon, among other factors, the permeability of the coating on the pigment particles towards oxygen. The impression gained in this laboratory from weathered metallic lead paint films is that atmospheric oxidation penetrates deeply, but more information on this point is desirable.

Oxidising paint media

In oxidising paint media which develop appreciable quantities of formic, propionic and higher acids on weathering, such acids will compete for the lead pigment and the picture will be more complex. In Fig. 5 a representation is given of the relative solubilities in water of the main compounds likely to arise in metallic pigmented paints weathering on steel, compiled from recent authorities^{5, 6}. (Quotable values for propionates were not to be found, therefore values for acetates are given, which may be expected to be similar.)



Fig. 5. Solubility data in gms. per litre. For convenience, values are depicted only up to 100 gms. per litre, though in several cases much greater. Broken lines show conjectural values

While most of the compounds quoted are far too soluble to give rise to passivation effects, undoubtedly such data are of great importance in determining the behaviour of metal pigmented paints, and are worthy of much study. In this short review it is only possible to comment on the beneficial tendency for lead corrosion products to be insoluble.

Thus the great success of lead anti-corrosive pigments in industrial atmospheres undoubtedly owes much to the low solubility of lead sulphate.

The low solubility of lead formate may also turn out to be of technological value, in cases when a drying oil medium is used.

Uses of metallic lead paints

Metallic lead paints have enjoyed wide popularity for many years for preventing the corrosion of steel.

Owing to their relative inertness to atmospheric attack, metallic lead paints by no means always receive topcoats : for example, some dockside cranes at St. Peters Port, Guernsey, have been repainted solely with metallic lead paint for 25 years.

The value of metallic lead primers was officially recognised many years ago by the issue of Ministry of Supply Specification TS226, which covered a minimum lead content and a test schedule for such materials. This has now been renumbered as Specification CS 2861A.

1965 METALLIC LEAD PIGMENT FOR ANTI-CORROSIVE PROTECTION

It may well be that metallic lead pigment shows for paint formulators a specially favourable balance between moderate reactivity on the one hand and susceptibility to passivation on the other. Certainly it remains a topic of much interest.

Acknowledgments

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The interior gloss retention of white enamels

An examination of some variables affecting the interior gloss retention of white enamels pigmented with mixtures of zinc oxide and rutile titania

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Summary

The gloss changes arising from changes in the pigment and binder phases of white enamels containing zinc oxide are described. The particle size of the oxide, its origin, the level at which it is present in the pigment phase and the total pigment concentration are all factors which may affect gloss, although the magnitude and direction of the changes vary according to the constitution of the medium.

An attempt to correlate gloss changes with the chemical and physical properties of the medium indicates that the degree of unsaturation (in simple drying oil media) is one major source of variation. In alkyd resins, such properties as Hydroxyl Value, viscosity and esterfying agent can be significant, but their effect is strongly affected by the modifying drying oil.

La retention de l'éclat interieur des emails blancs

Résumé

Cet exposé décrit les variations de l'éclat produites par les variations intervenant aux stades de pigment et de liant des émails blancs contenant de l'oxyde de zinc. La grosseur des particules de l'oxyde, son origine, le degré auquel il est présent au stade de pigment et la concentration totale du pigment sont tous facteurs susceptibles d'avoir une influence sur l'éclat, bien que l'importance et le caractère des variations diffèrent selon la constitution du vecteur.

En tentant de mettre en corrélation les variations de l'éclat et les propriétés chimiques et physiques du vecteur, le degré de non-saturation (dans les vecteurs à base d'huile siccative) apparaît comme étant l'une des principales sources de variation. Pour les résines alkydes, des propriétés telles que l'indice hydroxyle, la viscosité et l'agent estérifiant peuvent avoir de l'importance, mais l'huile siccative modifiante a une grande incidence sur leur effet.

Die Erhaltung des Innenglanzes weisser Emaillelacke

Zusammenfassung

Es werden die Glanzveränderungen untersucht, die sich in der Pigment- und Bindungsphase weisser Emaillelacke ergeben, die Zinkoxyd enthalten. Partikelgrösse des Oxyds, seine Herkunft und sein Anteil während der Pigmentphase, sowie die Gesamtkonzentration des Pigments können diesen Glanz beeinflussen, wobei Umfang und Richtung dieser Veränderungen von den jeweiligen Umständen abhängen.

Bei dem Versuch, die Glanzveränderungen mit den chemischen und physikalischen Eigenschaften des Bindemittels in Beziehung zu bringen, stellt sich heraus, dass der Sättigungsgrad (bei einfachen Bindemitteln auf Ölbasis) die Glanzveränderungen in erster Linie beeinflusst. Bei Alkydharzen können Hydroxylwert, Viskosität und Veresterungsfaktor von Bedeutung sein ; deren Wirkung wird jedoch von der modifizierenden Ölbasis stark beeinflu=t.

1965 THE INTERIOR GLOSS RETENTION OF WHITE ENAMELS

Introduction

The interior gloss retention of enamels has been discussed in numerous papers published over the past 25 years. The loss of gloss which sometimes occurs has been ascribed to defects in every portion of the formulation from solvents to driers. In the earlier papers¹⁻⁶ moisture in the ingredients, incorrect choice or quantity of driers, impure solvents and incorrect varnish making were all mentioned. When alkyd resins were introduced, the presence of crystalline bloom was noted and has been adequately explained.

In a number of papers,⁷⁻¹¹ mention is made of the change of gloss caused by the use of zinc oxide as a constituent of the pigment phase, but no evidence has been put forward to indicate if this effect is qualitative or quantitative with respect to the oxide content, or whether it is affected by the type of oxide, or the presence of other pigments. The effect of changes in varnish composition has not, so far as the author is aware, been investigated. One puzzling piece of evidence is that the loss of gloss of paints containing a proportion of zinc oxide is not consistent ; batches of the same formulation have been known to behave quite differently, some losing no gloss, whilst others lose appreciable gloss on interior exposure.

Variability in performance of a paint must, of necessity, be related to some change either in the formulation or in the environment. The mean particle size of pigments is controlled within fairly close limits by air permeability or nitrogen absorption methods but, nevertheless, particle size distribution can vary within certain limits without affecting the mean size. Solvents and driers are manufactured to close specifications and should not therefore have a marked effect on variability of performance of paints containing them. The medium, by virtue of its method of manufacture and the type of raw materials used, can vary in its properties; some are controllable but others, by virtue of those which are controlled, uncontrollable. The values of these variables can sometimes be determined by conventional chemical analytical techniques. Generally speaking, control of alkyd resin manufacture depends on the determination of acid value and viscosity. However, the hydroxyl value can only be controlled within fairly wide limits and does vary far more widely than the other properties of the resin. Other possible sources of variability are the viscosity of the original oil, its degree of unsaturation and, quite probably in the case of alkyds, the molecular weight distribution of the resin. Formulation variables such as pigment volume concentration may also be expected to contribute to gloss retention as well as the degree of dispersion of the pigment phase.

This paper attempts to examine the quantitative effect of a number of variables on gloss retention and is divided into two parts, the first dealing with simple oil media and the second with oleoresinous and alkyd media. In the latter section, the effect of additives has been somewhat elementarily examined.

Preparation and examination of paints

All paints were made by premixing the pigment and vehicle to give a heavy paste. The pastes were milled twice using a single roll mill and matured for 24 hours. Any necessary adjustment of medium and thinner content was made and the paints stored for 14 days. Paints were applied to burnished, degreased aluminium sheet and the painted panels were then stored for 24 hours and their gloss determined using an *Eel* gloss head and galvanometer; ten readings were taken on every panel. Further readings were taken at intervals for a period of 100 days.

In the preliminary work, some experiments were performed to determine the effect of environment and method of manufacture of the paint in order to determine the effects of these two variables. The major portion of the work relates to painted panels stored in a room in which temperature and humidity were closely controlled.

Analysis of results

Two statistical techniques are available for the determination of the significance of variables on any property, the analysis of variance and multiple correlation. The former method is the simplest to use and enables the person performing the experiment not only to determine the significance of the main effects but also that of all the interactions except the highest one. Multiple correlation techniques involve considerably more calculation but enable equations to be obtained which include all significant main effects. This, of course, is of value, since using optimisation techniques the highest value of the given measurement can be derived. All the data for multiple regression used in this paper was processed on the *IBM 7090* computer. All regression constants include are those significant at p=0.05.

Preliminary work

A paint known to have poor gloss retention was prepared by three different methods :---

(i) The pigment and 5 per cent excess medium over that required to satisfy the oil absorption of the pigment was milled as a very stiff paste stored for seven days and then diluted with medium and solvent to the finished formulation (P.3124).

(*ii*) The individual pigments were milled at pigment concentrations equal to those at which they were present in the finished product. These dispersions were stored for seven days and then mixed together using a high speed stirrer (P.3128).

(iii) The complete formulation was made up as a finished paint and stored for seven days (P.3125).

FOOTNOTE : In the analyses of variance a null hypothesis is invariably used. That is the investigator argues that no variable property (X) being examined has any effect on the property being examined (Y). The technique used is to compare the variance due to a particular change in a given property with the variance due to unknown properties and experimental error (known as the residual variance). The standard degrees of probability that the observed variation could have arisen by chance are expressed as p=0.20, p=0.05, p=0.01 and p=0.001. These indicate that there are probabilities of $(1-p) \times 100\%$, i.e. 80, 95, 99 and 99.9 per cent respectively, that the changes in property (Y) are really due to variation in (X). Generally p=0.05 is taken as the limit of disproof of the null hypothesis, or the limit corresponding to a real effect, but it is often argued that if p falls between 0.05 and 0.20 there is a strong probability that further more careful or more extensive tests will confirm the truth of the effect.

These paints were applied in triplicate to aluminium sheet and were exposed in the following three environments :---

- (a) Constant temperature and humidity (70°F, 22-25 per cent RH).
- (b) Laboratory conditions (60-70°F, 70-80 per cent RH).
- (c) Very poor intermittent illumination in an optical laboratory (65-70°F, 70-80 per cent RH).

After 100 days the following results were obtained :---

	Environment					
	a	b	c			
P.3124	67	45	63			
P.3125	53	43	63			
P.3128	48	38	54			

An analysis of these results indicates that the site is a significant factor affecting gloss retention but that the method of preparation (within these three methods) is not.

F(Site) =15.3 $\overline{2/4}\sqrt{p} > 0.05$

 $F(Preparation) = 5.8 \ \overline{2/4} \sqrt{p} > 0.20 < 0.05$

It was therefore decided to carry out all further tests in a constant environment (a).

Main programme of work

A number of simple oil media were examined using a normal grade of *White* Seal zinc oxide and rutile titania as the components of the pigment phase. (Appendix 1, Tables 1, 2.)

(i) Low	acid valu	e linseed	stand	oil	30	poise	(I.V.	100)
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(ii) Acid refined linseed oil	(I.V. 174)
(iii) Styrenated linseed oil	(I.V. 83)
(iv) Segregated fish oil I	(I.V. 209)

- (v) Segregated fish oil II (I.V. 225)
- (vi) Reaction products of linseed stand oil and zinc oxide containing : (a) 0.93 per cent ZnO, (b) 2.1 per cent ZnO, (c) 3.7 per cent ZnO.

Seven levels of zinc oxide with rutile titania were examined at three pigment volume concentrations in the first five media.

Ratio ZnO/TiO₂: 100/0, 80/20, 60/40, 40/60, 20/80, 10/90, 0/100

Pigment Volume Concentrations : 15 per cent, 20 per cent, 25 per cent.

In the short series of treated stand oils, five levels of zinc oxide were examined at three pigment volume concentrations :

Ratio ZnO/TiO₂: 100/0, 50/50, 20/80, 5/95, 0/100

Pigment Volume Concentrations : 15 per cent, 20 per cent, 25 per cent.

Regression equations for the first five media for paints containing zinc oxide were :

(1) Low acid value stand oil (G-53.6)=0.43 (L-51.7)+2.3 (P-20)

(2) Refined linseed oil (G-83.3)=0.10 (L-51.7)-0.28 (P-20)

(3) Styrenated linseed oil (G-72.2)=0.39(L-51.7)+0.45(P-20)

(4) Segregated fish oil (I) (G-25.9) = -0.01 (L-51.7) - 0.93 (P-20)

(5) Segregated fish oil (II) (G-26) =0.10 (L-51.7)+0.43 (P-20)

Including the iodine value as an independent variable, the following equation was derived :

(G-52.2)=0.3 (P-20)+0.16 (L-51.7)-0.26 (IV-159.2).

Since (P) is not significant, the regression equation for (L) and (I.V) becomes : (G-52.2)=0.16 (L-51.7)-0.26 (I.V-159.2).

Where G=final gloss, P=PVC, L=Level of ZnO in pigment phase percentage and I.V=iodine value.

It is of interest to note that when the paints containing titania as the sole pigment are analysed the regression equation

(G-69) = -0.410 (P-20) - 0.24 (IV-159.2)

is obtained.

The set of paints made up from oils 6a-c yielded the following equations : (i) Paints containing zinc oxide

- (G-46)=0.30 (L-43.75)-5.66 (Z-2.24)+0.19 (P-20),where Z=level of zinc oxide reacted with stand oil.
- (*ii*) Paints containing titania only (G-44.3)=-3.30 (P-20)-7.73 (Z-2.24).

The effect of particle size and composition of zinc oxide on gloss retention

Similar methods to those previously described were used to examine a range of oxides of varying mean particle size, purity, shape and manufacture. (Appendix 1, Table 3.)

Indirect process zinc oxide

Reference	Particle size ⁴ μ	Description					
Z1	0.09	Small irregular crystallites					
Z2	0.26	Small irregular crystallites					
Z3	0.41	Irregular crystallites					
Z4	0.42	Irregular, rod and plate particles					
Z5	0.57	Highly acicular					

Regression equation for paints containing zinc oxide :

(G-48.7)=1.46 (P-20)+0.18 (L-51.7) 109.22 (P.S-0.38)

Direct process zinc oxide (Appendix 1, Table 4)

Reference	Particle size ⁴ μ	Percentage lead as PbO	Description
Z6	0.23	3.0	Irregular
Z7	0.28	5.0	Slight acicularity
Z 8	0.33	5.0	Irregular

Regression equation for paints containing zinc oxide : (G-50.7)=1.74 (PVC-20)+0.45 (L-51.7)-83.93 (PS-0.28)

Oleoresinous media (Appendix 3)

Only one varnish of this type was examined, a 4:1 oil length congo/linseed oil/wood oil product. Two oxides, Z_1 and Z_2 , were examined in this medium.

Regression equation for paints containing zinc oxide : (G-67.7)=0.29 (L-51.7)-0.78 (PVC-20)-54.25 (PS-0.17)

Since PVC is shown to have no significance, the equation becomes : (G-67.0)=0.3 (L-51.7)-54.5 (PS-0.17)

Alkyd resins (Appendix 4, Tables 1, 2)

The number of possible identifiable variables in alkyd resins is extremely large and their analysis, if undertaken by a resin manufacturer, would involve considerable computational work. The variables which have been taken note of in this work are those for which data are normally available to the paint manufacturer, although some experimental resins have been included for which much more data were made available by the manufacturers. The work has been divided into two portions, the first part dealing with commercially available resin for which acid and hydroxyl values have been determined and the series of experimental resins. Details of these will be found in the Appendix 4.

Early work was confined to one resin, A.24, a long oil linseed/DCO glycerol alkyd. The following regression equations were obtained :

Indirect oxides Z1-Z5

(G-71.1) = -0.03 (L-51.7) - 1.50 (PVC-20) - 66.28 (PS-0.34)

Since (L) is not significant, the equation for particle size and PVC recalculates (G-71.1) = -1.5 (PVC-20) - 66.27 (PS-0.34)

Direct oxides

(G-71.0)=0.19 (L-51.7)-1.18 (PVC-20)-256.66 (PS-0.27)

Effect of change in alkyd resin on gloss retention

These experiments were designed to examine the differences in behaviour between alkyd resins of similar published specification, as well as between alkyds of differing types. The broad classes examined were as follows; the numbers in brackets indicating the number of resins examined :

Oil modification

<i>(a)</i>	Linseed oil				(12)
(b)	Linseed oil/wood oil				(5)
(c)	Dehydrated castor oil		 	•	(4)
(d)	Linseed oil/dehydrated castor oil				(2)
(e)	Soya bean oil				(4)
(f)	Miscellaneous (tobacco seed and	tall oil)			(2)

Variations in oil length and esterifying agent.

Variation in pigment volume concentration (15, 20 and 25 per cent) and in level of oxide in the pigment phase (0, 10, 25 and 40 per cent of ZnO). One oxide, a standard white seal grade, was employed (Z_2) .

Acid values and hydroxyl values were determined according to standard methods.

The total number of resins examined was 29 (Appendix 4).

The results obtained from these experiments can best be examined by variance analysis, each class of resin being individually examined.

	Medium (V)	PVC (P)	Level of Oxide (L)	V×P	V×L	L×P
All alkyds	>0.001	>0.001	>0.001	>0.05	>0.01	<0.20
Linseed oil alkyds D.C.O. alkyds	>0.001 <0.20	>0.001 <0.20	>0.001 <0.20	>0.001 >0.001 <0.20	<0.001 >0.001 <0.20	<0.20 <0.20
Linseed/wood oil alkyd	>0.001	>0.05 <0.01	>0.01 <0.001	>0.20 <0.05	<0.20	>0.05 <0.01
Linseed/D.C.O. alkyds	>0.20	>0.20	>0.20	<0.20	<0.20	<0.20
Soya alkyds	>0.05 <0.01	<0.20	>0.001	>0.20 <0.05	>0.05 <0.01	>0.20 <0.05

Significance of Effects and Interactions

These results indicate, as might be expected, that variations in the significance of the effects, due to change in formulation, highly affect the results. Analysis of the sub-groups within the linseed alkyds gave the following (Appendix 5, Tables 1-5):

	Medium (V)	PVC (P)	Level of oxide (L)	V×P	V×L	L×P
Linseed penta alkyds	>0.001	>0.001	>0.001	>0.05 <0.01	>0.05 <0.01	<0.20
Linseed glycerol alkyds	>0.001	<0.20	>0.001	>0.05 <0.01	>0.05 <0.01	<0.20
Penta alkyds 68% Oil	>0.001	>0.20 <0.05	>0.01 <0.001	>0.05 <0.01	>0.05 <0.01	<0.20
Penta alkyds 65% Oil	>0.05 <0.01	>0.05 <0.01	>0.05 <0.01	<0.20	<0.20	<0.20
Glycerol alkyds 70% Oil	>0.05 <0.01	<0.20	>0.01 <0.001	>0.05 <0.01	>0.20 <0.05	<0.20
65 % Oil	>0.05 <0.01	>0.05 <0.01	>0.05 <0.01	<0.20	>0.20 <0.05	>0.20 <0.05

Examination of the final gloss readings of the penta linseed alkyds indicates that differences in behaviour between alkyds is due to the pattern of gloss change with changing zinc oxide content. Some resins show a steady fall in gloss with increasing oxide content, whereas others show a minimum gloss rating at the 10 per cent level of oxide after which at the 25 per cent level the gloss increases. The resins in the former class were analysed separately (A2, 4, 5, 6 and 8).

V	Р	L	V×P	V×L	$L \times P$
>0.001	>0.001	>0.001	>0.20<0.05	>0.20<0.05	< 0.20

The regression equation for these resins taking oil length, PVC, L, acid value and hydroxyl value with consideration gave :

(G-38.4) = -0.98 (*PVC*-17.5) -0.38 (*L*-9.9) +0.18 (*OL*-67.1) +0.56 (*OH*-43) +0.25 (*AV*-9.1)

Since it was indicated that neither oil length nor acid value were significant, the recomputed equation reads :---

(G-38.4) - 0.98 (PVC-17.5) - 0.38 (L-9.9) + 0.59 (OH-43)

Experimental alkyds

Four sets of alkyd resins were made available by two resin manufacturers. Each manufacturer supplied one linseed/pentaerythritol alkyd series, the other two series were based on tobacco seed and a linoleic rich oil.

Set E.1 (Appendix 6, Table 1)

Long oil linseed alkyds 1

	Resin Reference		Po Ex	lyol cess	A V	cid alue	Vi (s	iscosity stokes)	· Hy V	droxyl 'alue	-
	53 54 57 58 A			0 25 25 0 17	1	4.7 7.8 0.4 9.0 9.8		35 27 35 31 41		12.1 45.3 46.2 22.0 35.0	_
				Va	irianc	e analy	sis		×		
			V	P		L		VP		VL	LP
All alkyds	ia	>0 <0	.05 .01	>0.0	001	>0.0	01 001	<0.20	>	0.20	>0.05 <0.01
penta cont Excess penta	tent	<0 <0	.20 .20	>0.0 >0.0 <0.0	001 05 01	>0.0 <0.0 >0.0 <0.0	001 05 01	<0.20 <0.20	<	0.20 0.20	>0.05 <0.01 <0.20

Regression equation for paints containing zinc oxide

(G-39.2) = -1.21 (*PVC*-35)-0.10 (*L*-49.5)+0.17 (Penta XS-13.4)+0.18 (*AV*-10.3)+0.17 (*OH*-32.1)+0.10 (ζ -33.7)

Recalculation excluding the non significant factors (Penta Excess, Acid Value, Hydroxyl Value and oil Viscosity) gives the equation :— (G-39.2) = -1.2 (PVC-35) = -0.09 (L-49.5)

Set E.2 (Appendix 6, Table 2)

Long	oil	linseed	alkyds 2
	7		ï

Resin Reference	Acid Value	Hydroxyl Value
89	7.4	10.6
91/1	27.4	16.4
91/2	15.2	16.4

January

V	Р	L	VP	VL	LP
<0.20	>0.01<0.001	>0.01<0.001	>0.20<0.05	>0.05<0.01	<0.20

Regression analysis for paints containing zinc oxide. (G-39.5) = -1.44 (PVC-40) -0.11 (L-54.4) -1.81 (OH-14)+0.42 (AV-16.7)

Only P is significant so the final regression equation becomes :— (G-39.5)=1.41 (PVC-40)

Set E.3 (Appendix 6, Table 3)

Resin reference	Acid value	Hydroxyl value
74	5.3	32.7
78	6.5	21.5
81	7.5	10.5

Lineoleic rich alkvds

Variance analysis

V	Р	L	VP	VL	LP
< 0.20	< 0.20	>0.20<0.05	>0.20<0.05	>0.20<0.05	< 0.20

Regression analysis for paints containing zinc oxide.

(G-35.6)=2.82 (OH-21.6)+26.52 (AV-6.4) -0.79 (L-51.7) -0.28 (PVC-40) No variation is significant.

Set E.4 (Appendix 6, Table 4)

Tobacco seed alkyds

Resin ref :	Oil viscosity c/s (η₀)	Percentage polyol excess	Acid value solid resin	Viscosity as supplied (stokes)	Solids content (ηa)	Hydroxyl value base resin
86	50	0	13	20	79.3	30.5
87	50	0	15	15	80.0	25.5
88	50	30	8	18	79.7	65.0
89	50	30	5	22	80.0	57.5
95	105	0	14	70	73.4	25.0
96	105	0	16	60	77.3	33.7
97	105	30	5	71	76.3	20.0
98	105	30	9	204	78.0	22.4

			the second s	Contraction of the local division of the loc	States of the second	
Alkyd classification	V	P	L	VP	VL	LP
All alkyds	>0.05 <0.01	>0.001	>0.001	>0.05 <0.01	>0.20 <0.05	>0.01 <0.001
Low viscosity oil	>0.001	>0.20 <0.05	>0.001	>0.20 <0.05	>0.20 <0.05	>0.20 <0.05
High viscosity oil	>0.001	>0.001	>0.001	>0.001	>0.001	>0.20 <0.05
Stoichiometric polyol content	>0.001	>0.05 <0.01	>0.001	>0.001	>0.20 <0.05	<0.20
Equal excess polyol	<0.20	>0.20 <0.05	>0.001	>0.20 <0.05	>0.20 <0.05	>0.05 <0.01
Low viscosity oil and stoichiometric polyol	>0.001	>0.05 <0.01	>0.001	>0.05 <0.01	>0.05 <0.01	>0.20 <0.05
Low viscosity oil and excess polyol	<0.20	<0.20	>0.01 <0.001	>0.20 <0.05	<0.20	<0.20
High viscosity oil, stoichiometric polyol	>0.001	>0.001	>0.001	>0.05 <0.01	>0.05 <0.01	>0.05 <0.01
High viscosity oil, excess polyol	>0.20 <0.05	>0.05 <0.01	>0.001	>0.05 <0.01	>0.05 <0.01	>0.05 <0.01

Variance analysis

Regression equation for paints containing zinc oxide

 $(G-33.5)=0.05 (PVC-20) -0.24 (L-18.7) -1.24 (OH-34.9)+0.63 (\eta oil-77.5) -10.90 (AV-10.6)+1.99 (\eta alkyd-60)+0.90 (Polvol XS-15)$

None of the variations are significant.

The effect of additives

It is well known that certain metallic naphthenates will act as dispersing agents for pigments and will therefore improve the initial gloss of paints containing them. Calcium, zinc and copper naphthenates were therefore milled into a paint known to lose gloss severely on interior exposure. A series of concentrations of the naphthenates were employed and the following results obtained :—

Nonkthereste				%	on Pign	nent			
Naphthenate	0	0.1	0.2	0.3	0.4	0.5	1.0	2.0	3.0
Calcium Zinc Copper	29 29 26	34 26 32	$\left \begin{array}{c} - \\ - \\ 41 \end{array} \right $	29 24 43	$\left \begin{array}{c}\\\\ -48 \end{array} \right $	24 24 49	23 22 49	34 26 51	26 18 53

Gloss after 100 days (black glass=100)

No correlation exists between the gloss and the calcium or zinc naphthenate content of the paints. In the case of copper naphthenate, however, the relationship is significant and positive.

r = 0.679 (p > 0.05 < 0.01)

Accelerated testing for gloss retention

Several suggestions have been made for determining the long-term gloss retention of enamels, such as stoving for certain times and temperatures but the results of tests of this type may well be affected by the heat stability of the medium. A simpler method might be to keep the paint film from drying for a

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period, thus allowing any changes in the physical state of the pigment system to occur with the minimum amount of hindrance and then permitting drying to take place normally. Changes which might occur during this inhibited drying period would be due to flocculation and/or reaction between the medium and any potentially active pigments.

A number of tests were carried out on paint films by storing the freshly applied film in an atmosphere saturated with solvents. Using white spirit alone, at laboratory temperatures it was found that the paints dried overnight, without any loss of gloss when compared with the similar paint dried in the open air. Eventually a mixture of white spirit and xylene was found to keep the film wet for a period of 48 hours.

An alternative method which it was thought might yield information was to examine very thin films microscopically by transmitted light before and after they had been subjected to shear. This was done by placing a small drop of paint on a microscope slide and gently dropping a cover slip on to the film. The cover slip was then carefully pressed down on to the paint and moved with an oscillatory motion until a very thin layer of paint remained. This was immediately examined at a magnification of $\times 1000$ and an assessment made of the degree of dispersion. The sample was then stored for 24 hours and reexamined without disturbance and again after the sample had been sheared between cover glass and slide. In this way a comparison between the freshly sheared film and the "rested" film was possible. The surfaces of some paint films have been examined by the shadowing techniques and an attempt has been made to relate surface properties with gloss.

	Paint															
	. A	B	C	D	E	F	G	Н	J	K	L	Μ	N	P	Q	R
Inhibited Drying	69	72	64	69	75	75	70	77	72	74	74	71	76	19	73	49
Air Dry+100 Days' Exp.	69 M	66 M	62 M	66 M	71 S	72 S	69 S	72 S	70 M	71 M	70 S	68 M	56 S	30 VH	64 S	34 M

The results obtained from these tests are given below :

V.H.-very high. M.-medium. S.-slight

A significant correlation exists between the two series of results r=0.888 $(p \ 0.001)$ (ID=0.99 AD+4.49, residual variance at regression line=+6.13). It will be noticed that although the subjective assessment of the degree of flocculation has some bearing on the gloss retention, the variations of gloss within any group of paints described as having similar degrees of flocculation is quite wide.

Optical examination of films

In the case of a mixed pigment system, such as that under discussion, interest is obviously focused on the distribution of the two pigment phases, consequently the films were dyed prior to examination with a solution of Alizarin S which is specifically adsorbed on to zinc oxide and which also slightly stains the whole of the surface, thus reducing back reflection from the film. All photomicrographs were taken on a Vickers projection microscope at $\times 250$ using a 16 mm. NAO.3 objective and a yellow filter.

It will be noted that as the recorded gloss decreases, so the degree of flocculation (as shown by the dark spots) increases (Figs. 1-3).





Fig. 1 (top left). Unhazed paint, gloss 95 per cent, r-titania 40 per cent, zinc oxide (Z1) 60 per cent, 25 per cent PVC in low acid value stand oil

Fig. 2 (top right). Hazed paint, gloss 44 per cent, r-titania 95 per cent, zinc oxide (Z2) 5 per cent, 20 per cent PVC in low acid stand oil

Fig. 3 (left). Hazed paint, gloss 24 per cent, r-titania 70 per cent, zinc oxide (Z2) 30 per cent, 15 per cent PVC in segregated fish oil Electron microscopy of films of low gloss also demonstrates the same relationship (Figs. 4-7).



Fig. 4 (top left). Freshly applied film, gloss 28 per cent, r-titania 40 per cent, zinc oxide (Z5) 60 per cent, 25 per cent PVC in low acid value stand oil

Fig. 5 (top right). Aged film, gloss 6 per cent, r-titania 40 per cent, zinc oxide (Z5) 60 per cent, 25 per cent PVC in low acid value stand oil

Fig. 6 (bottom right). Freshly applied film, gloss 34 per cent, zinc oxide (Z5) 100 per cent, 25 per cent PVC in low acid value stand oil

Fig. 7 (bottom left). Aged film, gloss 14 per cent, zinc oxide (Z5) 100 per cent, 25 per cent PVC in low acid value stand oil

Correlation between hardness, water absorption and interior gloss retention

Film hardness and water absorption might well have some effect on gloss retention. A short statistically planned experiment was therefore performed :---

Medium :

Penta alkyd blended with 20 poise stand oil at five levels

Pigment :

Zinc oxide blended with four levels of rutile titania

Pigment Volume Concentration : 15 per cent, 20 per cent, 25 per cent

Preparation and testing of the panels for gloss retention tests was carried out as previously described. Samples for water immersion tests were applied by doctor blade to previously weighed glass slides and allowed to dry for 24 hours in the laboratory and then placed in a desiccator for 24 hours. They were then reweighed and immersed in tap water. After immersion, panels were dried quickly between filter papers and reweighed. At the end of the tests the panels were again dried in a desiccator and immediately reweighed.

The panels for hardness tests were also applied to glass slides and allowed to dry for 24 hours. Pendulum hardness tests (Persoz) were determined at regular intervals for a period of one month.

Property	V	Р	L	VP	VL	LP
Gloss retention	>0.001	>0.001	>0.001 >0.05	>0.01 <0.001	>0.001 >0.01	<0.20 <0.20
Hardness Water absorption	>0.001 >0.001	<0.20 >0.05 <0.01	<0.01 >0.20	<0.20 <0.20	<0.001 <0.20	<0.20 <0.20

The following results were obtained :--

Variance analysis

Regression equations (film property against formulation variables)

Gloss retention :	G=0.262 (percentage alkyd in binder)+0.136 (level of oxide in pigment)+0.547 (PVC) -9.35 (p >0.001)
Hardness :	H=36.70 -0.32 (level of oxide in pigment)+0.05 (PVC)+0.25 (percentage alkyd in binder) -5.82 (film thickness) (p >0.001)
Water absorption :	W=0.0004 (level of oxide in pigment) -0.0032 (percentage alkyd in binder) -0.0296 (PVC) ($p<0.20$)

Correlation between gloss, hardness and water absorption (G-18.43)=0.095 (H-39.03)-23.8 (W-0.409)

Hardness is not a significant variable : therefore this equation simplifies to :— G=22.17W+9.36 (p>0.001)

Discussion

Even a cursory examination of the results of the statistical analysis of the final gloss of the enamels examined indicates the wide differences in behaviour which can occur when changes are made in a formulation.

In order to simplify the results, it is probably most profitable to examine the effects due to change in pigmentation and to see how these are affected by change in medium.

Logically it would be expected that increasing the total pigment volume concentration would reduce gloss retention. However, zinc oxide behaves anomalously in that the normal grades confer enhanced gloss retention with increasing PVC. In a paint containing both zinc oxide and an inert pigment, therefore, there may well be two opposing influences at work, the order of these influences depending on the amount of each pigment present and the total PVC. These two parameters, total pigment volume concentration and the percentage of zinc oxide in the pigment phase may well be related in their effect on gloss retention.

Of the simple oil media, two, the low acid value stand oil and the styrenated linseed oil show the anomalous behaviour due to the presence of zinc oxide, with gloss retention increasing with increasing PVC and level of oxide in the pigment phase. In refined linseed oil increasing the PVC results in reduced gloss retention whilst increasing the perentage of zinc oxide in the pigment mixture results in increased gloss retention. Pigmentation effects in the two fish oils are not significant at the level of p=0.05 and can therefore be omitted from the discussion.

One of the major differences between these five drying oils is in their degree of unsaturation as measured by their iodine value. It is therefore possible to combine this series of results and analyse them including iodine value as a variable.

This equation indicates that as a group, the total PVC has no effect, but increasing the percentage zinc oxide in the pigment results in increased gloss retention. More important ; increasing the degree of unsaturation of the drying oil reduces gloss retention.

This indicates the possibility of a reaction between the products of oxidation and degradation formed during ageing and the zinc oxide, due to its basic character, these reaction products having a deleterious effect on gloss retention. This negative effect of increasing iodine value however is also seen in enamels pigmented solely with rutile titania, wherein the increased pigmentation reduces the gloss retention.

The effect of zinc soaps in a simple pigmented oil medium are also shown to be deleterious when a stand oil/zinc oxide reaction product containing various quantities of reacted zinc oxide is examined. Gloss is reduced as the zinc content of the binder increases, although, as with stand oil alone increasing PVC and percentage zinc oxide in the pigment benefit the gloss. The reaction product pigmented solely with titania exhibits the same behaviour.

The single oleoresinous varnish examined shows differences in behaviour to those of a simple oil medium in that within the range of PVC's examined this property has no effect on gloss, whilst increasing the percentage of zinc oxide in the pigment mixture results in increased gloss. It is known that certain resin acids have strong dispersive properties and it would appear that the presence of compounds of this type in the congo ester varnish have offset the effect seen with stand oil, of low PVC's yielding paints of poor gloss retention. It would be unwise to draw too many conclusions from this one set of tests, since such variables as oil length and type of oil have not been examined.

The alkyd series of enamels presents a very complex picture. The possible number of variations in alkyd resins is legion, not only in the ingredients, but also in the method of manufacture, a state made worse by the fact that many of these (probably significant) variations are unknown to the paint manufacturer.

Discussing the known variations in alkyd resin properties and the commercial product, it is seen that in the majority of resins, increased PVC and percentage zinc oxide result in enamels showing poorer gloss retention.

The results obtained from the commercial alkyds produce no pattern, so that the formulator is faced with the problem of examining all resins individually in order to obtain data on gloss retention. There does however appear to be one exception to this, involving a limited number of pentaerithrytol alkyds based on linseed oil, where the hydroxyl value has a statistical effect. Here increased hydroxyl value confers better gloss retention. The use of this information is limited, in that it is first necessary to know the gloss percentage oxide effect at each PVC, since from the table of results it is seen that these selected resins have a similar gloss pattern.

The experimental alkyds show similar variations in performance with respect to change in pigmentation and percentage zinc oxide as do the commercial ones. Comparing the effect of these changes of formulation in the two linseed oil penta alkyd series it is seen that gloss decreases with increasing PVC in both sets, but whereas gloss also decreases with increasing zinc oxide in Set E1, the effect in Set E2 is not significant. Hence in Set E2, any level of zinc oxide could be used in a mixture with titania at a low PVC without gloss retention suffering significantly. No other physical or chemical property of the alkyd resins used in these two series has any genuine effect on gloss retention.

In the semi-drying oil modified alkyds no variation in properties had any significant effect on gloss retention, neither did any variation in pigment concentration or percentage zinc oxide.

The effect of change in the type of drying oil modification in alkyds can be assessed fairly easily. Linseed oil modification provides best gloss retention, followed by linseed oil/wood oil, dehydrated castor oil and the semi-drying oils. Glycerol esterification provides superior gloss retention to pentaerythritol under the stated test conditions. The oil length has a variable effect, but a figure of 65 per cent seems to provide the best performance.

It would seem that, examining alkyd resin variables which are not generally known to the user, wide variations in gloss can occur due to the direction in which a particular variation affects gloss retention. This may well be due to the type of product formed when zinc oxide reacts with constituents in the alkyd and also the effect of these reaction products on the physical state of the pigment system during the drying and ageing of the film. Dealing with the physical properties of the oxide itself and their effect on gloss retention, in all paints examined there is an inverse relationship between the particle size of the oxide and gloss, or the finer the oxide, the better the gloss retention. The only other variation in the zinc oxides examined, the method of manufacture of the oxides shows quite clearly that indirect process oxides possess superior gloss retention to direct process oxides.

The remainder of the experimental work reported, dealing with the effect of naphthenates on gloss retention and the effect of variation in hardness, water absorption and drying time in gloss indicate that copper naphthenate improves gloss retention, whilst the other naphthenates examined, calcium and zinc have no significant effect.

The percentage alkyd present in a medium composed of alkyd and stand oil is a significant property, and as might be expected, improved gloss retention results from increasing the alkyd content. In this series of paints it is noteworthy that the stand oil portion of the medium appears to have a greater effect on pigment/medium gloss relationships than does the alkyd portion. Increasing the PVC and the percentage zinc oxide in the pigment phase both benefit the gloss retention.

Although the percentage zinc oxide has an effect on the hardness of a film, as does the PVC, there is no correlation between hardness and gloss retention. There is however a very high correlation between the water absorption of a film and its gloss retention.

This raises the subject of the effect of change in the relative humidity of the environment during the period of test. Undue sensitivity of moisture will result in volume changes with change in humidity. Since water vapour is capable of behaving as a plasticiser, the film could well change in flexibility many times, this combined with the normal increase in hardness during the film's life could result in surface irregularities, as well as in changes in the physical state of the film.

The comparison between the results of the accelerated tests and the normal interior exposure shows that the method used has some merit. Some subjective relationship also exists between the apparent degree of flocculation of the pigment phase and the gloss of the film.

Conclusions

The use of multiple regression techniques has enabled the effect of a number of variations in paint formulation on interior gloss retention to be determined. The equations so obtained can be subjected to optimisation techniques in order to obtain the highest gloss retention in a particular system.

A number of possible causes of loss of gloss can be suggested, most of them arising from changes taking place in the film and affecting the physical state of the pigment. A major cause would seem to be flocculation due to the presence of zinc soaps of some particular types. This, however, cannot be the sole cause, since the particle size of the zinc oxide present has a significant effect and increasing surface area of the oxide results in improved gloss retention. The fact that a correlation between water absorption and gloss exists indicates that changes in the state of the film during ageing are of considerable significance.

The anomalous behaviour of zinc oxide indicates that the presence of zinc soaps themselves flocculate the remaining oxide, since at high pigmentations. where zinc oxide paints possess good gloss retention pigment mobility within the film will be at a minimum and the state of its dispersion cannot change greatly. This will apply equally to paints containing mixtures of pigments.

Chemical and physical properties of the medium itself have an important bearing on gloss retention. These effects, however, can vary both in their significance and in their direction, so that no firm general conclusions can be drawn from the results. This applies particularly to commercially produced alkyds of similar published specification, where wide differences in gloss retention occur.

In general linseed oil alkyds possess best gloss retention, followed by linseed/ wood oil, dehydrated castor oil and semi-drying oil alkyds. Alkyds esterified with glycerol appear to give slightly better gloss retention than those esterified with pentaerythritol. No large effect could be found in changing the oil length. but there would seem to be some slight advantage in those of 65 per cent oil length.

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Appendix 1

Table 1

Oil media, gloss readings (black glass=100)

% Zinc oxide in	Low acid value stand oil	Refined linseed oil	Styrenated linseed oil	Segregated fish oil (I)	Segregated fish oil (II)						
phase		% Pigment volume concentration									
	25 20 1	25 20 15	25 20 15	25 20 15	25 20 15						
100 80	90 56 72 82 61 60	78 84 83 70 78 76	86 84 83 88 93 89	16 43 32 28 25 27	46 32 21 32 16 25						
60 40	78 57 42 68 44 30	80 87 86 85 81 90	82 81 77 67 74 44	21 16 21 22 18 22	35 27 28 28 20 23						
20 10	55 33 2 34 40 3 85 87 0	85 85 83 91 89 88	68 65 63 49 49 57 73 81 86	23 24 25 15 35 54	22 15 25 13 32 28						
100 80 60 40 20 10 0	90 56 7. 82 61 60 78 57 42 68 44 30 55 33 22 34 40 33 85 87 90	78 84 83 70 78 76 80 87 86 85 81 90 85 85 83 91 89 88 63 60 66	86 84 83 88 93 89 82 81 77 67 74 44 68 65 63 49 49 57 73 81 86	16 43 32 28 25 27 21 16 21 22 18 22 23 24 25 15 35 54 33 34 34	46 32 32 10 35 22 28 20 22 12 13 32 82 8						

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% Zinc oxide in	Stand	l oil+0.9	93%	Stan z	d oil+2. inc oxide	1%	Stand oil + 3.7% zinc oxide			
plgment			% P	igment v	olume co	oncentra	tion			
	25	20	15	25	20	15	25	20	15	
100	61	58	55	57	74	70	45	43	43	
50	65	65	58	57	65	68	37	42	41	
20	53	43	39	55	57	55	32	28	28	
5	29	36	52	25	24	20	25	25	25	
0	28	48	68	43	56	71	24	25	36	

Table 2

Table 3											
Indirect	process	zinc	oxides	in	stand	oil					

% ZnO in pigment	ļ	25% PVC				Ì	20% PVC				15% PVC				
pigment	Z_1	Z_2	Z_3	Z^{i}	Z_5	Z_1	Z_2	Z_3	Z^{4}	Z_5	Z_1	Z_2	Z_3	Z4	Z_5
100	92	90	63	41	27	92	73	56	37	34	90	72	31	36	37
80	95	82	75	49	35	91	64	61	31	35	87	60	43	18	29
60	92	78	49	39	50	87	57	37	23	28	79	42	34	17	16
40	93	68	45	35	49	82	44	38	22	24	67	30	42	14	15
20	96	55	36	36	40	75	33	41	19	25	66	28	49	28	14
10	75	34	43	29	40	66	40	50	21	23	59	35	57	46	50
0	90	85	94	100	88	90	87	97	95	86	88	90	88	93	88

Table 4

Direct process zinc oxide in stand oil final gloss readings

% ZnO in pigment		25% PVC			20% PV0	c	15% PVC			
pigment	Z_6	Z_7	Z ₈	Z_6	Z_7	Z_8	Z_6	Z_7	Z_8	
100	80	72	80	71	69	78	45	67	70	
80	78	77	75	63	69	67	50	56	51	
60	80	68	68	54	62	52	45	47	38	
40	61	62	64	41	50	39	39	38	28	
20	56	46	31	39	33	20	32	34	16	
10	48	36	8	41	32	13	46	34	30	
0	90	92	88	89	90	87	88	86	87	

Appendix 2

Indirect process zinc oxide/r-titania in congo ester

% ZnO in	25%	PVC	20%	PVC	15% PVC		
pigment	Z_1	Z_2	Z_1	Z_2	Z_1	Z_2	
100	70	81	77	75	86	68	
80	65	85	74	65	82	70	
60	71	88	75	71	88	68	
40	66	70	81	56	87	77	
20	60	34	73	44	80	54	
10	57	28	47	42	56	53	
0	49	44	58	51	61	53	

Appendix 3

 Table 1

 Indirect process zinc oxide/r-titania in alkyd resin medium

% ZnO in	1	25% PVC					20% PVC					15% PVC			
phase	Ζ1	Z_2	Z_3	Z_4	Z_5	Z_1	Z_2	Z_3	Z_4	Z_5	$ Z_1 $	Z_2	Z_3	Z_4	Z_5
100	95	79	69	12	22	92	72	92	47	55	100	72	88	63	50
80	100	83	89	12	28	88	79	94	50	69	92	76	92	72	60
60	82	76	88	21	41	80	74	95	62	51	88	74	96	63	70
40	86	76	88	25	51	88	76	95	63	62	95	77	65	65	60
20	80	72	85	37	51	89	75	90	62	51	93	74	93	67	64
10	70	74	78	45	50	84	70	93	61	58	86	69	93	71	57
0	85	93	100	89	87	95	93	99	100	95	95	96	98	97	90

Table 2
Direct process zinc oxide/r-titania in alkyd resin medium

% ZnO in pigment		25% PVC			20% PVC	С	15% PVC			
phase	Z_6	Z_7	Z_8	Z_6	Z_7	Z_8	Z_6	Z_7	Z_8	
100	76	61	73	85	79	77	88	82	82	
80	83	73	55	89	78	74	83	75	73	
60	85	77	46	90	85	73	85	83	73	
40	86	76	24	83	78	64	83	76	62	
20	81	56	23	82	69	59	83	68	57	
10	75	55	27	82	61	50	85	59	49	
0	87	92	82	99	91	94	.99	94	90	

Appendix 4

Alkyd resins (commercial)

Ref.	% oil	Type of oil	Acid value	Hydroxyl value	Alcohol
	69	Lingerd/DHCO			
A 1	65	Linseed	80	18	Pentoarythrital
2	68	Linseed	5.6	38	Pentaerythritol
2	68	Linseed	12.2	57	Pentaerythrital
3	68	Linseed	7.2	20	Pentaeruthrital
+ -	70	Linseed	7.0	10	Pentaerythritol
6	70	Linseed	11.2	49	Pentaerythritol
7	65	Linseed	10.0	32	Pentaerythritol
Ŕ	65	Linseed	19.5	24	Glycerol
9	65	Linseed	15.1	22	Glycerol
10	70	Linseed	43	46	Glycerol
ĩ	70	Linseed	8.0	26	Glycerol
2	70	Linseed	16.2	35	Glycerol
3	62	D.H.C.O.	13.0	38	Glycerol
4	66	D.H.C.O.	6.5	47	Glycerol
5	69	D.H.C.O.	4.4	13	Glycerol
6	70	D.H.C.O.	5.1	15	Glycerol
7	50	Linseed/Wood oil	8.3	58	Glycerol
8	65	Linseed/Wood oil	13.0	27	Glycerol
9	65	Linseed/Wood oil	4.2	20	Glycerol
20	67	Linseed/Wood oil	19.2	11	Glycerol
1	72	Linseed/Wood oil	11.4	15	Glycerol
2	70	Linseed/D.C.O.	6.1	25	Glycerol
3	60	Linseed/D.C.O.	8.2	27	Glycerol
4	68	Linseed/D.C.O.			
5	65	Linseed/D.C.O.	5.0	24	Glycerol
6	57	Linseed/D.C.O.	12.5	83	Glycerol
7	85	Linseed/D.C.O.	4.8	10	Glycerol

whhe	1		1	í I		1	I	1											
	A 12		70			25	46.8 39.8 42.4												
	Ĺ					15	43.4 27.5 33.8												
	=					25	44.8 39.8 36.0												
	A1		7(lcoho		15	45.0 47.3 44.0												
	0			fying a		25	45.0 26.0 27.6												
	A1		70	l esteri		15	48.2 25.3 31.5												
				lycero		25	47.6 38.6 36.4												
	A9		65	0		15	49.6 41.5 41.9												
~	~			Ì		25	50.0 43.9 42.5												
i=50)	A8		65			15	49.3 49.0 47.2												
glass		ength			ation	25	41.9 27.1 31.8												
black	LA	d oil le	70		ncentr	15	43.0 32.0 31.3												
ble I dard (Linsee		Ì	ume co	25	44.8 39.6 33.9	e 2											
Tat	stand A6		70		ent voli	15	46.6 46.4 42.8	Tabi											
lkyds					Pigme	25	35.2 23.8 21.5												
cial a	A5		68	cohol		15	38.8 32.3 27.2												
nmer		A4							ing al		25	48.2 47.1 45.6							
Col	A4																		
				Penta		25	49.3 39.7 39.9												
	A 3		68		-	15	50.6 37.7 37.7												
						25	37.0 38.5 34.6												
	A2		65			15	50.2 42.2 40.2												
						25	35.4 28.4 25.5												
	A		65		-	15	42.3 31.3 31.6												
	1	% ZnO in	phase				0 10 25												

		1	1	I	1	ļ	1		
	A16			70		25	38.1	25.2	C VC
	4					15	30.8	29.1	1 12
1000000	15	r oil		6	ohol	25	28.9	44.6	ALL
and the second se	A	ated casto	ength	9	rifying alc	15	47.2	26.3	0 17
And a second sec	A14 Dehydrogena		Oil I	6	cerol ester	25	36.7	21.9	100
	A	De		9	Gly	15	26.1	24.9	V DC
	[3			5		25	34.8	25.6	1 00.
	IA.			6		15	22.5	21.3	B / C
		ui OuZ /0	pigment	pilase			0	10	

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January

1965 THE INTERIOR GLOSS RETENTION OF WHITE ENAMELS 73

Table 3	
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	A	17	Al	18	Al	19	A2	20	А	21		
		Linseed/wood oil										
% ZnO in pigment	t Oil length											
phase	6	5	65		67		72		50			
				Glye	cerol est	erifying	, alcoho	0]				
	15	25	15	25	15	25	15	25	. 15	25		
0 10	50.2 48.5	52.5 39.5	48.8 45.8	45.0 36.9	47.6 40.4	46.3 38.8	48.8 44.6	42.8 34.6	42.1 33.3	38.3 26.0		
25	46.5	40.0	43.2	40.2	39.7	35.4	40.6	32.9	31.3	26.6		

Tab	le 4
Commercial alkyds	(black glass=50)

% ZnO in pigment phase	A	22	A23						
	Linseed/D.C.O.								
	Oil length								
	7	0	60						
	Glycerol esterifying A1.								
	Pigme	nt volume	concentr	ation					
	15	25	15	25					
0	48.9	38.1	48.3	47.1					
10 25	34.0 32.1	25.3 29.6	42.9 37.6	38.9 26.2					

Table 5

	A: So	25 ya	A	26 	A27 Segregated soya						
% ZnO in pigment	Oil length										
phase	6	5	5	7	85						
	Glycerol esterifying A1.										
		Pign	nent volur	ne concen	tration						
	15	25	15	25	15	25					
0 10 25	36.3 11.8 12.5	30.1 13.2 13.3	30.9 19.9 22.7	25.7 20.7 21.8	35.1 5.5 4.5	33.3 11.5 19.0					

Appendix 6

% ZnO in pigment phase							Resi	n refe	erence	e					
	153		154		157		158				- (
					Р	igmei	nt vol	ume	conce	entrat	ion				
	45	35	25	45	35	25	45	35	25	45	35	25	45	35	25
100 50 5	3 14 30	36 42 47	46 49 51	23 40 42	26 43 41	55 53 50	25 36 45	49 42 42	51 52 53	2 12 41	33 39 44	40 48 48	5 33 47	46 46 44	49 50 53

 Table 1

 Gloss of long oil linseed alkyds (black glass=50)

Table 2Gloss of long oil linseed alkyd resins

	Resin reference										
% ZnO in pigment	8	9	91	/1	91/2						
phase	Pigment volume concentration										
	45	35	45	35	45	35					
100 · 50	40 39	50 47	17	34 54	18 37	34 59					
5	38	41	39	57	27	59					

Table 3Gloss of linoleic rich alkyds

% ZnO in pigment phase	Resin reference										
	7	4	7	8	81						
	Pigment volume concentration										
	45	35	45	35	45	35					
100	30	39	29	29	21	39					
50	34	28	38	35	30	36					
5	151	39	47	35	28	43					

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% ZnO in pigment phase

Gloss of tobacco seed alkyds

Table 4

Surface coatings in atomic energy installations^{*}

By H. Wells

A.E.R.E., Harwell

Summar y

The subject is discussed under three headings : (i) Surface coatings for contaminated areas, (ii) Surface coatings exposed to high level radiation, (iii) Surface coatings complying with "metal free" standards.

The paint is discussed as being used for a decontainable surface, and the mechanism for the diffusion of radioactivity through the film is discussed.

The uses of various types of paint are mentioned and the problems of natural radiation are touched upon. Finally, metal free paints and their use in high radiation areas are mentioned.

Revêtements dans des installations d'énergie atomique

Résumé

Le sujet est discuté sous trois en-têtes :

- (i) Les revêtements pour zones contaminées.
- (ii) Les revêtements exposés à un niveau élevé de radiation.
- (iii) Les revêtements conformants aux spécifications stipulantes le débarrassement des métaux.

On discute des peintures utilisables pour des surfaces décontaminables et le mécanisme de la diffusion de la radioactivité à travers la pellicule.

On constate l'emploi des types différents des peintures et on touche aux problèmes de radiation naturelle. Enfin on décrit des peintures débarrassées des métaux et leur application dans des régions de haute radiation.

Anstrichmittel für Installationen, die Atomischer Strahlungsenergie ausgesetzt sind

Zusammenfassung

Die Materie wird unter drei Überschriften besprochen :

- (i) Anstrichmittel für verseuchte Zonen.
- (ii) Anstrichmittel, die intensiver Bestrahlung ausgesetzt sind.

(iii) Anstrichmittel, die den Metallfreiheit fordernden Vorschriften genügen.

Bei den besprochenen Anstrichfarben wird angenommen, dass sie sich zur Entseuchung eignen, und ausserdem wird der Mechanismus der Diffusion von Radioaktivität durch Filme beleuchtet.

Die Anwendungsarten verschiedener Typen von Anstrichmitteln werden aufgeführt, und die Probleme natürlicher Strahlung berührt. Schliesslich werden noch metallfreie Anstrichmittel, sowie deren Verwendung in Zonen, die starker Strahlung unterliegen, erwähnt.

Introduction

Surface coatings are used in atomic energy installations in a variety of applications, some of which are orthodox, e.g. decorative paints and protective coatings for structural steel, some of which are very unorthodox. Paint manufacturers have been somewhat confused when, asked to specify surface coatings for the latter applications, the so-called critical areas in atomic energy establishments and more recently in nuclear power stations.

^{*}Read before the London Section, Thames Valley Branch, 27 February, 1964.
It is proposed, therefore, to consider initially these requirements, namely (a) surface coatings for contaminated areas, (b) surface coatings exposed to high level radiation, (c) surface coatings complying to a so-called metal-free and clean conditions standard.

The requirements will be dealt with separately, but it should be understood that in certain circumstances two or even three of the requirements may be necessary for one application.

Under (a) the reasons for applying decontaminable coatings will be stated, some information will be given on the types of contamination that can exist, and some postulations will be made on the mechanisms of contamination.

Certain types of surface coatings that have shown excellent resistance to contamination, both in the laboratory and in practical applications, will be mentioned.

The effects of high level radiation on surface coatings will be covered in very general terms. The effect of radiation on single polymers in itself is a very involved and difficult subject, and the effects of radiation on a complex system such as surface coatings which may contain polymer, pigment, plasticiser, stabiliser and residual solvent is obviously even more complex. It should be useful, however, to indicate the type of area where coatings will be subject to high radiation levels, and give some approximation of the levels at which the various types of coatings may be expected to degrade to any reasonable extent.

Probably the most mystifying requirement is for "metal free" paints, which are used in areas where "Magnox" clad fuel is handled. The list of precluded metals or their salts in such surface coatings has recently been extended in the light of recent research, and the full list of precluded metals is included in this paper.

Radiological contamination

For personnel working in an area contaminated with radioactive isotopes the danger to health is twofold. First, there is an external radiation hazard which can be of sufficient intensity to be potentially injurious to a man spending the whole of his working life in such an environment. This danger comes principally from isotopes emitting β particles and γ rays, since the range of α particles in air is very limited.

Secondly, there is an internal radiation and toxic hazard brought about by the ingestion, inhalation and absorption of radioactive materials into the body. All radioactive materials are potentially dangerous in this respect. This includes the α particle emitting isotopes because actual contact with tissue is involved.

At this point it is pertinent to indicate how minute is the mass of radioactive material with which we are concerned. Consider first the external hazard, any radiation excess to normal background is potentially dangerous to health. It has, however, been possible from clinical studies to fix levels of radiation below which no harmful effect would be expected on a normally healthy person. The unit of adsorbed energy, the *rad*, is 100 ergs per gram, and the maximum permissible level of mixed radiation over the whole body, where β ray exposure to the lens of the eye is negligible, is 1.5 rads per week, of which not more than 0.3 rads must be due to γ radiation.

To illustrate the imperceptibly small mass of material required to produce this level consider one instance in detail. Assume that 0.1 micrograms of P-32, which emits β radiation of maximum energy 1.7 MeV is located as a point source. The adsorbed dose for tissue three feet away from the point source during a 40-hour week would be 1.7 rads which is well in excess of the maximum permissible level. The result of this is that as small an amount as one microgram of P-32 distributed over a room 12 feet square would render the room unfit to work in until some form of decontamination had been carried out.

The requirements for safety in regard to the internal radiation hazard are even more stringent. The maximum permissible level for one of the more toxic α emitters is as little as 1×10^{-4} micrograms per cubic metre of air.

The level of contamination permitted on surfaces is to a large extent governed by the need to keep the air free from contaminated dusts. Ideally any contamination of a surface by radioactive materials should be completely removed but safe working levels have been calculated and amount to 1×10^{-4} microcuries (μ c) per sq. cm. for most radioactive isotopes and $1 \times 10^{-5} \mu$ c per sq. cm. for certain α particle emitting isotopes e.g. Pu, Po, Ra, Ac, Pa. In addition to the requirements of safety, radiological contamination can cause spurious results in counting equipment or on photographic plates.

Having thus dealt with the need for reducing contamination to acceptable levels consider in what forms contamination can be present and the properties required of a coating to render it decontaminable.

In a research establishment the radioactive contaminant can be almost any element in the periodic table. In actual fact the usual forms of contamination are used nuclear fuel and "fission products," the waste products separated from the fuel. The relative abundance of the various elements contained in fission products depends upon reactor conditions, time of irradiation and the decay period after the fuel has left the reactor. The constitution of fission products six months after removal from what may now be termed a conventional gas cooled reactor is very approximately :—

Group		Percentage
0	Inert gases	10
1	Alkali metals, e.g. caesium	8
2	Alkaline earths, strontium, barium	25
3	Rare earths	35
4	Zirconium	9
5	Niobium	9
Fransitional	Ruthenium	4

The physical form of the contaminating material ranges from dry metallic dusts and swarfs, often found in remote handling cells, to the strong nitric acid solutions used in separation plants where nuclear fuel is processed.

In order to choose a paint to perform as a decontaminable surface it is necessary to know the form of contamination that it is most likely to be subjected to. In many cases it is possible to predict this since the areas involved have been designed for a specific purpose, but in others e.g. decontamination centres, the type of contamination will vary from day to day. In these cases one must extemporise and put forward recommendations that cover all the condi-

January



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tions as fully as possible. The radioactive contamination may be held loosely on a paint surface as a dust, perhaps trapped in pores, cracks and other irregularities, or it may be associated with a film of oil, grease and other inactive dirt. Contamination of this type is not usually difficult to remove except where it is trapped in cracks. A first criteria for a decontaminable paint surface is that it should be smooth, glossy and free from checking, crazing and other surface defects.

There are many ways in which the radioactive contaminants can be held more firmly to the surface and may penetrate into the paint film. These include adsorption and ion exchange from solution ; chemical reaction between radioelements and constituents of the paint film, i.e. reactive pigments and extenders ; absorption into pores and by processes of activated diffusion ; and solvent extraction by plasticiser and retained solvent.

All organic surfaces, including paint, possess ionogenic groups and in contact with aqueous solutions these groups may ionise and the paint film behave as a low capacity ion exchanger. At pHs below 2 very little ionisation of the cation exchange groups, e.g. carboxylic acid group occurs and at a pH of 6 and over many isotopes form colloidal solutions. The uptake of cations under certain conditions, however, is marked but decontamination using what amounts to a regeneration of the ion exchange resin is not difficult. The low pH decontaminating solutions used in these processes usually incorporate chelating agents capable of forming anionic complexes with the contaminant. The anionic complex so formed is repelled from the similarly charged "co-ion" fixed to the exchanger.

In addition to the exchange capacity of the vehicle the extender too may have anion exchange capacity or may be capable of exchanging a stable isotope with a radioactive isotope of the same element.

Penetration of contamination into paint films is often noticed but two environments are more prone to this than others. One concerns situations where nitric acid solutions of fission products are encountered particularly in the presence of Butex*. The second condition is where the paint spends prolonged periods of immersion in contaminated water, e.g. cooling pond equipment and transport containers. In these cases the ease with which radioactive ions diffuse into the paint varies with the age of the paint film and the length of time the paint is submerged. A direct relationship between the water vapour transmission rate and the rate of diffusion of ions into a film has not been observed, but water uptake must play an important part in the process. Many papers have been published demonstrating the flow of ionic current through organic membranes, and it may be recalled that in a work by Cherry and Mayne¹, two methods whereby ionic currents may flow through an organic membrane were discussed. Where only a small amount of moisture had been adsorbed, conduction through the film was ascribed to a process of activated diffusion, i.e. fixed ions in the membrane acted as stepping stones by which oppositely charged ions could move through the membrane. Where a sufficient amount of water had permeated the coating the possibility then rose of large hydrated ions travelling through any pores that were formed. The formation of pores of this size must affect the ease with which radioactive ions penetrate into the coating.

*Dibutoxy dibutyl ether.

In a lecture designed to cover a broad field it is not possible to dwell too long on certain specialised topics as the mechanisms involved in contamination and decontamination especially since a lecture on this subject was given recently to the London Section by Mr. R. B. Wilson of the Admiralty².

The rest of the discussion on the decontamination aspects of surface coatings will be confined to a review of the types of paint commonly encountered in radioactive areas indicating some of the variations in formulation that affect their decontaminability.

Paints based upon chlorinated rubber have been used as decontaminable coatings for some years and for many environments have not been bettered. They are used extensively in areas where their chemical resistance, including resistance to decontaminating agents, and impermeability to water, has enabled the area to be maintained at very low levels of contamination. The formulation of chlorinated rubber based paints for application to small plaques under laboratory conditions is relatively easy and the fact that the use of such materials under practical conditions is altogether a different matter is recognised. This is one reason why there has been reluctance to attempt to propound specifications for such materials and their formulation has been left to experienced manufacturers.

The following points on formulation do appear to be important in manufacturing a decontaminable paint :---

- (i) The amount and type of plasticiser used. In this respect both chlorinated paraffin and chlorinated diphenyl have been used successfully (types containing $\simeq 50$ per cent Cl₂). In many cases the chlorinated diphenyl is preferred owing to its superior radiation resistance.
- (ii) The use of suitable pigments at the correct pigment volume concentration. Titanium dioxide is the most common pigment encountered in decontaminable paints. In practice, not a great deal of difference is encountered between good quality rutile and anatase pigments, but laboratory tests indicate a preference for the purer anatase materials in decontaminable paints. Zinc oxide and lithopone are unreliable and of the extenders barium sulphate is unacceptable. The amount of pigment used is important since the penetration of contamination into the paint film increases with increasing pigment concentration. Indeed it is not unusual to read of unpigmented or low pigment content finishes suggested as the final coat because of this occurrence. (Low pigment content being defined as less than 20 per cent pigment volume concentration.)
- (*iii*) The importance of solvent balance is widely recognised in the importance of paint production and an unfavourable balance leading to high solvent retention impairs the ease of decontamination. Where high boiling solvents are used to improve brushing qualities, it is important to avoid those that decrease the chemical resistance of the paint film since it has been shown that the decontaminability is also reduced.

Vinyl paints

Paints based upon vinylidene and vinyl polymer have been used more extensively in the United States than in this country. Here the vinyl formulations have been used more as strippable coatings than as fixed systems. Strippable coatings were

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originally used in the United States and were based on the philosophy that it would be impracticable to decontaminate the surface and that complete removal of the activity would necessitate removal of the paint film. Strippable paint coatings are still used to a limited extent in this country, in areas where contamination is gross or where a first line of protection is thought necessary. In the latter case they are used over a fixed system, generally an epoxide, that is unaffected by the solvents of the strippable paint. The problems of using strippable coatings are not great but one that frequently occurs is where the coating is not, in fact, stripped off for a long time. Often in these cases a combination of ageing and radiation embrittles the coating and makes removal difficult especially when the workers are wearing pressurised suits.

Epoxide resin paints

Cold cured epoxide resin paints are less readily decontaminated than the chlorinated rubber based materials. In addition to the variables quoted for chlorinated rubber formulations, the type and amounts of curing agent as well as the conditions under which they are used, affect the decontaminability of the paint. In general the amine adduct is preferred to the amine and polyamide curing agents. There is an improvement in the decontaminability of the amine and amine adduct cured epoxides if they are given a post-curing treatment. The decontaminability of the polyamide cured epoxides is improved slightly if the mixed paint is allowed to stand for a few hours before being applied. Solventless epoxide resin systems and stoved epoxide and phenolic coatings in general can have good decontamination properties.

The isocyanate cured epoxide resin paints are little different in decontamination than the amine cured materials, although much better decontamination results have been obtained from dialkanolamine modified epoxides cured with isocyanates.

Polyurethane paint

The catalyst cured polyurethane paint has not been as widely used on UKAEA establishments as the epoxide resin based materials. There are signs however that the use of polyurethanes is increasing. The decontaminability of the commercially available paints tested has varied, giving in a few cases results as good as the amine adduct cured epoxides, but usually being as poor as the polyamide cured epoxides.

All the paints so far mentioned have been applied in areas or on equipment where a specific decontamination problem exists, the epoxides being used in areas where oil, abrasion or high radiation make them more suitable than the chlorinated rubber. In many areas, however, the walls and equipment will only become contaminated if a major accident occurs. In these areas the paint is required for normal decorative and anti-corrosion purposes with the probability that they will never require decontaminating in their lifetime. In these areas an orthodox decorative gloss paint is preferred.

Before leaving the subject of contaminated paints comment on one aspect of paint in a specialised field is called for. There are occasions when most of the paints supplied by industry contain too much natural radioactivity for them to be used in the proximity of very sensitive counting equipment. The standard H. WELLS

of cleanliness required around these monitors is very high in order to reduce the background of ionising radiation. For instance, any steelwork that has stood outside before installation has to be cleaned of radioactive fall-out before it can be incorporated into the equipment. The paint for use on this steel and other surfaces must be low in potassium because of the interference from naturally occurring potassium-40. A study of the potassium content of some extenders emphasises the need for careful selection of materials.

		Percentage of potassium-40
China clay	(Aluminium silicate)	0.7-2.0
Bentonite	(Aluminium sodium potassium silicate)	1.02.0
Mica	(Aluminium potassium silicate)	8.0-10.0
Labradorite	(Aluminium sodium calcium silicate)	0.5 0.7

Many pigments, including certain grades of titanium dioxides, include radium and thorium daughters among their impurities at a level high enough to make them unacceptable for this purpose.

Radiation effect

The areas where surface coatings are required to withstand high level radiation are fortunately relatively small. There are, however, surfaces in close proximity to reactors, and cells, in which irradiated fuel is processed or examined, where organic coatings will be subjected to high level irradiation by either neutron, γ or β radiation. The surfaces of cooling ponds where irradiated fuel is stored under water prior to processing, are also liable to be degraded.

Descriptions of the different types of radiation can be obtained from a variety of textbooks and it is probably unnecessary to define the difference between a neutron and a β particle.

When atomic radiation passes through polymers energy is dissipated to a large extent in the excitation of electrons and through ionisation, to a much smaller extent by atomic displacement, and to even smaller extent by transmutation.

Ionisation and electronic excitation processes bring about the breakage and rearrangement of chemical bonds, and the formation of free radicals. The resultant chemical reactions may cause cross-linkage, double bond formation, gas liberation, hydrogenation, vulcanisation etc. In the presence of air, water or other media interaction may occur. It is obvious therefore that profound changes in the physical properties of polymeric materials, in this particular case surface coatings, may be expected when the magnitude of incident radiation is significantly high.

It is to be further expected that changes in plasticiser, stabiliser, and to a certain extent solvent, where this is retained, may equally affect the behaviour of the film under radiation. A considerable amount of work has been carried out on the effect of radiation on organic materials including polymers, and it is not difficult for anyone sufficiently interested in the topic to obtain some basic knowledge of these effects on certain basic polymers which may be used in paint formulation. It may be useful, or perhaps interesting, for those with but a passing interest in the subjects, to indicate broadly an order of radiation stability of certain polymers and some examples of the wide differences that can occur using different plasticisers or curing agents.

It should first be reiterated that radiation damage only occurs after a paint film has been subjected to a very high amount of radiation, above 1×10^6 rads.

In order of increasing stability :---

	Appro	oximate deterioration dose
(<i>i</i>)	Cellulose esters	1×10^{6} rads.
<i>(ii)</i>	Polymethyl methacrylate	5×10^6 rads.
(iii)	Neoprene	5×10^7 rads.
(iv)	Chlorinated polymers	5×10^7 rads.
(v)	Catalysed epoxide resins	$5.0 \times 10^{8} - 2 \times 10^{9}$ rads.
(vi)	Alkyd resins and polyesters	$5.0{\times}10^8{-}1.0{\times}10^9$ rads.
(vii)	The phenolics urea and melamine formaldehyde	es $1-5.0 \times 10^9$ rads.

Plasticisers may exert a protective influence in paint formulations, and we find that chlorinated diphenyl plasticisers used for example in chlorinated rubber can raise the level at which radiation effect occurs, to circa $1-2 \times 10^8$ rads.

In general aromatic curing agents confer greater radiation stability on epoxides than the aliphatic types. This can be utilised more in stoving epoxides than in the conventional air-drying types, but certain solventless epoxide coatings utilise aromatic amines as curing agents.

In areas where radiation is of a high order conventional cold catalysed epoxide resins are used mainly. These combine reasonable contamination and corrosion resistance with good radiation resistance. Radiation will tend to cure further the paint film by the formation of free radicals and the overall energy transmitted to the system.

For underwater applications the more reliable water resistance of chlorinated rubber paints utilising a formulation best suited to radiation conditions is to be preferred.

The effects of radiation on surface coatings is a very interesting subject, and one which has been only superficially covered, possibly because of its complexity. It is intended to carry out more work in this respect as soon as possible.

Metal free paints

Consider finally the concept of "metal free", "non-obnoxious" or clean conditions paints. One of the disadvantages of using magnesium alloys for cladding fuel elements is the compatibility problem. A study of the metallurgy of magnesium alloys in respect of the demands made of it revealed a list of elements that could be tolerated in very low concentrations if at all. It was presumed that there was a possibility during the handling of the fuel elements for a flake of paint to be deposited on to the can and thus carried into the pile. H. WELLS

Once inside the pile the organic matter would decompose and deposit the inorganic filler on to the magnesium alloy. Should the filler contain more than the allowed quantity of undesirable elements there is the possibility of a failure occurring in the cladding leading to a release of radioactive material.

A specification given below was drawn up precluding the use of certain elements above the desired limit, and advice was sought that the specification was within the capacity of the paint industry. The areas where this type of material would be used may also demand other attributes of the paint films such as decontaminability and/or radiation resistance. Above all the material must possess the attribute of a good paint film in respect of adhesion, durability and corrosion resistance.

In areas through which magnesium alloy canned fuel elements or their components pass, all paint must be specified as free from the elements listed below at least to the degree stated in table 1.

(a) Group 1 metals	Mercury
(b) Group 2 metals	Barium, bismuth, cadmium, gallium, indium, lead, lithium, thallium, tin, zinc.
(c) Group 3 metals	Aluminium, antimony, calcium, cerium, copper, nickel, praseodymium, silver, strontium.

Application	Aggregate of group 1 metals	Aggregate of group 2 metals (percentage)	Aggregate of group 3 metals (percentage)
Primer	Nil	1.0	3.0
Undercoat and finish	Nil	0.1	1.0

Table 1	
Specification for	metal-free paints

In addition boron should not exceed 50 parts per million. Silicon, whether free or combined, should not be more than 5,000 parts per million calculated as SiO₂ in the dry film.

A related material is the clean conditions primer ; this is intended to protect steelwork from corrosion for the period between manufacture and the reactor becoming critical. It is applied to surfaces inside the pressure shell of the reactor where intense radiation and high working temperatures are expected. No paint film will withstand these conditions and the primer is expected to dust off. The primer must not give off any resinous matter that will deposit inside the shell. The dust released from the primer will be deposited over the fuel elements and the graphite moderator. Therefore the composition of this material is subject to the same limitations as the metal free paint from the point of view of compatibility with the added concern due to the much larger amounts of material involved. Also there is the effect of this material on the graphite to consider. The specification for the graphite itself is very rigid and any material that impairs the efficiency of the graphite must be excluded from the primer. Certain materials that are acceptable in small amounts would be deleterious in the amounts accrued from the primer. In this respect iron which may be accepted in the metal free paint would not be acceptable in the primer since the amounts involved would lower the ignition temperature of the graphite and thus add to the fire risk should a power surge occur.

References

1. Cherry and Mayne, J.E.O., Off. Dig., 1961, 33, 469.

2. Wilson, R. B., JOCCA, 1964, 47, 308 (summary of paper).

Discussion at the Thames Valley Branch of the London Section

MR. W. J. ARNOT asked about the performance of black bitumens.

MR. WELLS said that the colour was against them; the colour should be light to show any contamination.

MR. DRISCOLL referred to the list of the order of increasing stability of polymers and pointed out that the number of rads was quoted, but there was no rate factor.

MR. WELLS said that the rate did not have an effect on paint films, although the effect of rate was more pronounced in bulk polymers. Oxygen played a very important part in breakdown, the breakdown pattern being different on the surface from that in the bulk of the polymer. One hundred and six rads in one hour rapidly used up the oxygen nearest the surface; if the same dosage were spread over 100 hours the oxygen could reach the polymer so that low radiation over a long period was therefore more destructive. Quaternary carbon atoms increased the vulnerability of a polymer.

MR. REID asked for details of the type of breakdown which occurred.

MR. WELLS said that radiation could cause chain cleavage or cross-linking, or both simultaneously. Methyl methacrylate gave blistering associated with chain cleavage. Cleavage gave rise to softening and cross-linking to brittleness. The more aromatic the molecule, the greater was its radiation stability.

DR. G. M. HENDERSON asked if leafing aluminium pigments would stop penetration.

MR. WELLS replied that he had done no work on this aspect as he was not concerned with research on paint formulation. A leafing pigment might be useful.

DR. M. ELLINGER said that it was unexpected that anatase titanium dioxide should be better than rutile, as rutile did not chalk.

MR. WELLS said that anatase would not be used externally. Ninety-nine per cent of the paint was used indoors and chalking did not occur. The difference between the two grades was not very great.

MR. L. L. PEARSON asked why paint was used at all; why were not ceramics or stainless steel chosen?

MR. WELLS replied that cost was the main objection. Paint was used for ceilings, walls and sills. A good vitreous tile was better for flooring than substitutes based on epoxides or polyesters. Stainless steel was very expensive and could be worse for contamination than paint.

MR. R. G. ALLEN asked what quantity of paint per year was used.

MR. WELLS said that not very much was used for contamination areas, particularly as research and nuclear power stations were being more selective in their use of specialised paints. Some of the work involved in developing these paints might find applications elsewhere, e.g. a non-contaminatable paint could be interesting to the food industry. MR. REID asked about the effect of the substrate, e.g. concrete compared to steel.

MR. WELLS replied that no marked effect on contamination had been noticed.

MR. P. WHITELEY asked what kinds of primer could be used in view of the last paragraph in Mr. Wells's paper.

MR. WELLS said the specification was compiled by the Engineering Group at Risley in collaboration with representatives of the paint industry. The specification precludes the use of most anti-corrosive pigments but pigments such as titanium dioxide are acceptable.

MR. P. WALKER said that as a specification has been established precluding the use of certain elements, were tests in fact carried out and, if so, by whom, and at what cost?

MR. WELLS replied that the responsibility for testing and inspection was that of the Standards and Inspection Group at Risley. There has been much collaboration between Risley and paint suppliers to reduce the possibility of "metal free" paints becoming contaminated at the supplier's premises but ultimately complying with the regulations was a matter of the honesty of the paint supplier.

MR. WHEELER asked how radiation quality was measured. Was the type and its energy important?

MR. WELLS said that the type was important; and the quality was measured by its energy distribution. The energy absorption pattern of polymers resembled in some ways that for UV radiation.

MR. PEACOCK proposed a vote of thanks for a very interesting paper, which should be helpful to chemists who were asked to supply radioactivity-resistant paint by return! Do you realise **GLOVERS** Technical Information Booklets may have the answer to your problem? Have you received copies of:—



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The Council has authorised the production of an exclusive Tie for Members of the Oil & Colour Chemists' Association and it is expected that the ties will be available early in November. The Association's insignia will be woven in red and gold silk on a dark blue terylene background.

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Review

INTERNATIONALE BIBLIOGRAPHIE DER FARBENLEHRE UND IHRER GRENZGEBIETE, VOL. 2, PERIOD 1950-54

By MANFRED RICHTER. Musterschmidt Verlag, Göttingen, Berlin-Frankfurt, 1963. Pp. xi+837. Price DM 57.

This thick volume brings together over 4,500 references to published work on colour and related subjects, collected not only from journals of Europe and the United States but also from those of the Soviet Union and Japan.

The range of subjects covered is extremely wide and includes the chemistry and physiology of colour vision and studies of the colour matching properties of the eye; instruments for colour measurement and their applications to industrial problems of colour control; the optical properties of colouring materials and calculation of the colour of mixtures; light-fastness tests; the effects of illuminant on colour; colour reproduction by printing and photography; the principles and methods of colour television, as well as such subjective matters as colour perception, studies of colour space, colour harmony, the psychology of the use of colour in factories, hospitals, schools, etc.

The references are numbered and listed in alphabetical order of authors, but each is also given one or more reference numbers relating the nature of the subject matter with the appropriate subdivision of the International Decimal Classification. In some cases as many as five or six such reference numbers are given, presumably recording different aspects of the subject or the content of matter subsidiary to the main subject.

Some of the references, particularly those taken from the colour reference service of the journal *Die Farbe*, also give short abstracts of the subject matter in German.

The final section of the book is a subject index in which the references in each narrow division of the subject are grouped together under the headings of their appropriate decimal classification numbers. To anyone familiar with this classification this could provide a useful means of selecting the papers relating to a given subject, but the very large numbers of references found under some headings suggests that the classification may not be sufficiently finely subdivided in some parts to deal with all the ramifications of these specialised subjects.

The period covered by this volume runs nominally from 1950 to 1954, but in fact a number of earlier references are also included, going back as far as the early 1940s, which had presumably been omitted in preparing the first volume of the series.

This is a well organised and comprehensive bibliography which should serve as a valuable source-book for workers in any of the numerous fields of colour science and technology, limited, of course, to the period which it covers.

The past few years have, however, shown a rapidly growing development of certain aspects of colour science, notably the application of instrumental methods to industrial colour matching and colour checking problems, and it REVIEW

appears to the reviewer that the book would have been even more valuable if the period covered could have been extended to a date closer to the date of publication. No doubt in future volumes it will be found possible to shorten this gap.

D. L. TILLEARD.

Information received

New leaflets

KRONOS TITANIUM PIGMENTS Limited announce that new information leaflets 2/E and 17/E entitled "Interactions between titanium dioxide pigments and other substances" and "A method of determining the dispersibility of titanium dioxide pigment on ball milling" are now available.

A better counter

A new, more sophisticated model, say COULTER ELECTRONICS LTD about the *Coulter Counter Model C*. Some of the advantages listed are, improved statistical sampling of fluids, direct percentage read-out of any channel and modular electronic construction for speedy replacement of faulty components.

CIBA's new subsidiary

WM. ASKE & CO. LTD have recently become a wholly owned subsidiary of CIBA (A.R.L.). The Managing Director of Askes states that he is confident that the CIBA (A.R.L.) research and production resources will enable his company to take a more leading part in the development of new products in the field of foundry techniques.

Organic reagents for metals

From HOPKINS & WILLIAMS LTD comes Volume 2 of their publication "Organic Reagents for Metals." Divided into two parts (a) Absorptiometric and Gravimeteric Reagents, (b) Complexometric Indicators, the book describes the background, preparation and method of use of many reagents. Fuller information can be gained from the references listed after each chapter. The price is 30s.

CORRECTIONS

"Modern Aqueous Organic Pigment Dispersions," by P. Birrell, November 1964.

It is regretted that the following references were omitted from the first lines of the four paragraphs appearing under Figs. 7 and 8 on page 882 :

1st Paragraph — Fig. 7 2nd Paragraph — Fig. 8 3rd Paragraph — Fig. 9 4th Paragraph — Fig. 10

Also Figs. 2 and 3 on page 880 and Figs. 7 and 8 on page 882 should read :



ARYLAMIDE YELLOW G



RED 4B TONER



VICTORIA BLUE LAKE



BRILLIANT ORANGE GR.

Correspondence

Dr. L. A. Jordan

SIR,— May I beg space in your *Journal* to say "Thank You" to the very many people who have paid tribute to Louis Jordan?

The letters which have reached me from all over the world are so numerous that it will be several weeks before I have answered each individually.

Do not let us grieve : he died as he would have wished—" going to work " and that is the epitaph he would have chosen.

Yours sincerely,

Oakhill Drive, Langley Road, Surbiton, Surrey. Marjorie R. Jordan. 12 December 1964.

Editorial

Putting a figure to it

Many years ago an eminent scientist made the following statement: "When you can measure what you are talking about and express it in numbers, you know something about it; but when you cannot measure it, when you cannot express it in numbers, your knowledge is of a meagre and unsatisfactory kind."

How stands the technology of surface coatings when faced with this statement? Do we, as practising scientists and technologists, really interest ourselves in the absolute physical properties of our final product, the dry paint film, or is our standard still that of 20 or 30 years ago? One may argue that such a physical property as tensile strength in a paint film is meaningless, since a film is always in contact with a substrate. However, is not cracking a manifestation of a state of imbalance between tensile strength and adhesion? When physical tests are performed, do we know what they measure? The scratch test for instance, does it measure hardness, or adhesion, or elasticity? Hardness testers of the indentation type are available, and have been for many years; does anyone in the paint industry use one? Instantaneous measurements compared with indentations for varying periods of time will give a measure of the deformation or cold flow properties, both useful but neglected attributes.

Some years ago the TNO published some thought-provoking results from tests on unsupported films; no one seems to have taken this up, so one must assume that the industry as a whole is not really interested in objective testing, but is prepared to continue as before.

Is it not significant that competing industries such as the plastics industry is able to produce tables of physical properties of their products without difficulty, whilst our industry is still quoting "passed $\frac{1}{8}$ " bend test?

Bristol

The printability of paper and board

On Friday 30 October 1964, at 7.15 p.m., 30 members and visitors attended the 136th ordinary meeting of the Bristol Section, held at the Royal Hotel, Bristol.

The second paper of the series "Painting, Printing and the Substrate," entitled "The Printability of Paper and Board," was presented by Miss E. J. Pritchard, of PATRA.

Printability tests were legion. Numbers of these tests could only be regarded as approximate as they were largely empirical and lacked a precise meaning. Rather than attempt to deal briefly with all these tests, three methods on which recent work had made notable advances were discussed in detail : picking on offset lithographic papers, oil absorbency and smoothness.

By the use of the methods described for picking and oil absorption it was possible to derive paper constants that were independent of the liquids used for testing.

With smoothness it had been shown certain sizes of pits were of more importance than others, and that frequency distribution curves were necessary before the effect of surface contours on printing results could be predicted.

During the discussion which followed the paper, Miss Pritchard answered lucidly the questions asked of her and many questions were discussed across the floor, members representing both the paint and printing ink industries taking part. It is hoped that the paper will be published.

Irish Branch

Mixing, dispersing and wetting equipment

The first meeting of the 1964-65 season was held in Jury's Hotel, Dublin, on Friday 25 September 1964, under the chairmanship of Mr. M. O'Callaghan. Twenty-one members and 17 visitors attended this meeting, when a paper was given by Mr. H. Wadham entitled "Mixing, Dispersing and Wetting Equipment."

This paper created great interest which roused many interesting questions. These were put by Mrs. Stokes, Mr. O'Callaghan, Mr. Lyons, Mr. Summerville, Mr. Cotter, Mr. Darwen, Mr. Quick, Mr. Murphy and Mr. McKirnan. A vote of thanks was proposed to Mr. Wadham by Mrs. H. F. Stokes, and this was ably seconded by Mr. R. Cotter. The meeting closed at 10.45 p.m.

F. D. H. S.

L. T.

London

Mould resistance of paints in the tropics

The third technical meeting of the session was held at Manson House on 12 November when, with Mr. M. R. Mills in the chair, Mr. P. Whiteley, of the Building Research Station, spoke on "Mould Resistance of Paints in the Tropics."

Mr. Whiteley said that two distinct types of growth were encountered, moulds which required organic material for growth and which occurred under conditions of high humidity, even temperature and sometimes absence of light, and algae, which were able to synthesise their food requirements and required copious liquid water, daylight and mineral nutrients. There had been increased complaints about mould growth since the last war and even hard glossy alkyd paints were susceptible to mould growth. Growth of algae was confined to exterior surfaces with good access to water. Porous surfaces, e.g. cement-based materials, were most susceptible, and avoidance of algal growth was largely a matter of design of buildings. Mould growth was a greater problem as moulds had very low nutrient requirements, e.g. growth could occur on dust on a glass surface.

There were three main effects of mould growth: (a) a simple surface deposit, (b) discoloration through stains from the mould, and (c) actual destruction of the film. The micro-climate was of importance in mould growth, e.g. high humidity caused by vegetation, cooling of surfaces due to air conditioning, etc., with consequent condensation.

Mr. Whiteley then described a number of series of test exposures at Accra (dry, high humidity) and Lagos (considerably more rain). Generally organic fungicides tended to lose their activity in 12-18 months on exterior exposure, and paints containing zinc oxide showed up well. American-style house paints also showed good results against mould growth, but lower durability than alkyds. The film behaviour of paints showed differences between various grades of zinc oxides. In a final series of tests, two mercury fungicides were examined at various concentrations, but their effect was lost after about 12 months, and again by comparison zinc oxide-containing paints showed up well. Mr. Whiteley said that there were limitations in the various laboratory tests which could be applied for screening.

In the lively discussion which followed the paper, questions were asked covering the mode of action of zinc oxide, and of the other fungicides, the effect of different substrates, and the effect of legislation on the use of poisonous materials. Those taking part in the discussion were Mr. H. A. Newnham, Mr. M. J. Meason, Dr. H. G. Rains, Mr. R. J. McCausland, Mr. J. K. Rankine, Dr. B. S. Gidvani, Mr. D. Cameron, Dr. L. A. O'Neill, Mr. L. Dickson and Mr. E. J. Howes. The vote of thanks to the lecturer was proposed by Mr. M. J. Meason.

V. T. C.

Southern Branch

Durability testing of titanium oxide pigments

The third technical meeting of the current session was held on 9 November at the Queen's Hotel, Southsea, when, with Mr. C. Carey in the chair, Mr. E. Oakley gave a paper entitled "Durability Testing of Titanium Oxide Pigments."

Mr. Oakley began by describing the methods used for assessing the durability characteristics of titanium pigments in terms of gloss retention and chalking tendencies. Both outdoor exposure sites and laboratory apparatus were used to produce the data required. Accelerated testing in the laboratory was preferable in terms of time taken to produce results, but emphasis was placed on the necessity to obtain correlation between the accelerated and natural methods. Difficulty had been experienced in this respect, making it necessary to use the exposure site as the ultimate standard. Data was given on the rate of breakdown of various paints at different exposure sites and in the "Weather-O-Meter," showing the amount of acceleration obtained at Florida being about $\times .2$ and the "Weather-O-Meter" about $\times .4$ compared with this country.

The speaker said that various trials of this nature covering a full range of titanium pigments in various media had produced similar results in the degree of acceleration obtained at different sites, but occasionally anomalies occurred in the "Weather-O-Meter." This aspect of the work was still being pursued.

The paper concluded with a silent film in colour, to which Mr. Oakley added a commentary. The film showed the amenities available at the Carlton weathering station

for testing paints. The film showed that a full range of substrates and techniques of exposure were fully exploited to gain the maximum information. Of particular interest was the range of meteorological instruments used to record weather conditions, including a solarimeter and "wet-time" recorder.

Following the paper, a very interesting discussion took place, during which Mr. Oakley dealt with points raised by his talk.

The evening concluded with a vote of thanks being proposed by Mr. J. Smith.

A. F. R.

Thames Valley Branch

Antimony oxide flame retardant paints

The third technical meeting of the 1964-5 session was held on 17 November at the Royal White Hart Hotel, Beaconsfield, when Mr. A. G. Walker, a member of the Branch, spoke on "Antimony Oxide Flame Retardant Paints."

Mr. Walker traced the history of the use of antimony oxide as a flame retardant in plastics, textiles and other inflammable materials. He pointed out that in order to obtain the maximum effect it was necessary to form antimony trichloride *in situ* and this could be accomplished by the use of antimony oxide in combination with a chlorinated compound such as chlorinated paraffin, chlorinated rubber or pvc.

The technical requirements of an antimony oxide-containing flame retardant paint were discussed, with special reference to the spread of flame test described in BS 476. The results obtained by distilling mixtures of antimony oxide and chlorinated paraffin were given, from which the optimum ratios were predicted. These indicated that when a liquid chlorinated paraffin was used, the best results should be obtained when the ratio corresponding to SbOCl was present, whereas when a solid chlorinated paraffin was used, a ratio corresponding to SbCl₃ was necessary.

Detailed formulations of emulsion, gloss and lustre flame-retardant paints were given, when it was pointed out that the ratios of chlorine to antimony were as predicted by the distillation experiments. Results given indicated that the application of these paints did not reduce the good flame-spread characteristics of non-combustible boards and when used on combustible substrates improved the classification. Such materials, unlike many other flame-retardant paints, were fully decorative.

The talk was followed by a discussion in which Mr. Thursby, Mr. Simpson, Mr. Lewis, Mr. Rankin, Mr. Fletcher, Mr. Arnot and Mr. Brighton took part. A vote of thanks for a paper on a very important subject was proposed by Mr. Tatton.

W. S.

Manchester

Dispersion stabilisation mechanisms in paint systems

The second technical meeting of the session took place on 20 November in Manchester, when Mr. W. Black presented a paper entitled "Dispersion Stabilisation Mechanisms in Paint Systems."

Mr. Black began his lecture by mentioning that considerable progress had been made in the understanding of colloid stabilisation in recent years. He reminded the audience that in paint manufacture the first problem was that of wetting the pigment with a liquid. Wetting behaviour was usually described with reference to Young's equation and surface-active agents were often used to improve the wetting behaviour of pigments. The phenomenon of adsorption at interfaces was fundamental in the application of wetting agents and it was important to observe that dynamic rather than equilibrium values were usually applicable in practical systems. The lecturer commented that although thorough wetting of the pigment was required for good dispersion, it was not, by itself, a sufficient condition.

The lecturer then went on to discuss the nature of the forces between particles dispersed in liquid media. The attractive forces, such as Van der Waals' or dipoledipole forces, were responsible for flocculation and some repulsive force or energy barrier was required to inhibit flocculation. In the case of classical colloidal sols in aqueous media the interaction of the electrical double layers formed round the particles in solution was sufficient to stabilise very fine dilute suspensions. The importance of adsorption behaviour in the formulation of the double layer was emphasised. The quantitative development of this charge stabilisation theory by Verwey and Overbeek, and Derjaguin and Landau, showed the dependence on electrolyte content, size of particles, composition of the solid, dielectric constant and other physical quantities and, while the theory was satisfactory for inorganic electrolytes, it did not explain the enhanced stabilisation of sols stabilised by non-jonic surface-active agents or by polymeric materials. More recently it had been suggested that the existence of steric barriers could explain this enhanced stability, and entropic repulsion, caused by restriction of the freedom of interpenetrating polymeric chains, had also been suggested.

In non-aqueous systems of low dielectric constant, Mr. Black said it appeared likely that, in even moderately concentrated suspensions, overlapping of the electrical double-layers around the particles occurred and this, coupled with the reduced surface potential, meant that charge stabilisation was unlikely to be a major factor in non-aqueous systems. Other complications which occurred in non-aqueous systems were the flocculating effects of water and other non-miscible solvents. It was also possible that an aqueous film around the particles could lead to better adsorption by some ionic process occurring inside the aqueous envelope.

The flocculation behaviour of phthalocyanine blue in stoving media was illustrated and its virtual elimination by surface coating with a barium lake of a sulphonated copper phthalocyanine and rosin was, according to the lecturer, probably due to a steric barrier. The different degrees of flocculation of a phthalocyanine blue in different paint media were shown and sedimentation volume tests correlated well with the visible differences between spray and dip application. In a thixotropic alkyd paint the reduction in thixotropy caused by Arylamide Red 4RH used in a full shade was shown to be due to preferential adsorption of the thixotropic constituent in the paint. Pre-coating by recovering the pigment from such a paint and redispersing in fresh alkyd was shown to overcome this defect.

The lecturer concluded by illustrating the large differences in surface behaviour of many pigments and pointed out that such differences combined with the use of a wide variety of solvents meant that it was extremely unlikely that any single agent could be found which would give stable pigment dispersions in a variety of nonaqueous media. Adsorption was a fundamental process in dispersion stabilisation and the best methods of pigment dispersion are often those which increased the adsorption and binding of agents to the surface.

The discussion was introduced by the Chairman, Mr. H. F. Clay, and questions covered both theoretical and practical aspects. Amongst others, Dr. D. A. Plant asked why the attractive forces between the adsorbed layers on the particle surfaces were not sufficient to cause flocculation, and Mr. Black explained that the attraction of part of the molecules of the adsorbed species for the medium was an important factor in steric and/or entropic repulsion. Mr. T. E. Johnson commented that technology still seemed to be ahead of colloid science and wondered how soon it would be possible to predict, for practical systems, potential energy diagrams of the type shown by the lecturer. Mr. Black thought that this was very unlikely in the near future. The difficulties in calculating attractive forces, in measuring size

distribution, in adequately defining physico-chemical properties of the resinous medium and in measuring adsorption of complex substances were immense. He did not, however, feel that attempts to understand these processes were merely academic, since by application of the general principles it was always possible to indicate areas where empirical investigations could lead to improved dispersion stability.

The lecturer agreed with Dr. W. Carr that charge stabilisation could occur for larger particles in non-aqueous media and not for smaller particles, and it was quite conceivable that more than one stabilisation mechanism could be present in a single system. In reply to another questioner, the lecturer said that to reduce flocculation differences between two pigments in a mixed pigment system he would suggest attempting to coat one pigment surface to make it more like the other so that adsorption of resin binder and other constituents would be similar.

In proposing a vote of thanks, Mr. J. J. Kavanagh thanked the lecturer for his most interesting and stimulating lecture. Mr. Kavanagh commented on his continued interest in the subject due to the early application of theories at the bench and considered that this was a most interesting subject in which further clarification of the mechanisms involved would be of considerable value. The audience of approximately 75 members and guests acknowledged their support in the usual manner.

W. F. MCD.

Midlands

What is pigment dispersion?

At a meeting held in Birmingham Chamber of Commerce on 20 November, with Mr. R. D. Calvert as Chairman, Dr. F. M. Smith gave a paper entitled "What is Pigment Dispersion?"

Dr. Smith began by saying that his talk was mainly concerned with organic pigments in printing inks. Dispersion was a relationship between pigment and vehicle, and there was very little evidence to suggest that this relationship involved monolayers. There was no agreement between measurements of surface area in nitrogen and in liquids, and experiments with adsorption of oleic acid on titanium dioxide pointed to active sites rather than to monolayers.

Electron micrographs showed that pigment particles had a variety of shapes and dimensions, and were packed together in various ways. Opacity was related to refractive index and to particle size, and opacity was greatest when the particle size was about half the wavelength of the incident light. Tinctorial strength and opacity thus gave some indication of average particle size, but attempts to correlate refractive index with specific surface area were invalidated by birefringence. The actual process of printing could affect colour and give bronzing. The *Coulter Counter*, which recorded change in conductivity as particles passed through a 30 micron orifice, could be used only for extremely dilute suspensions.

Heats of wetting were usually exothermic ; thus the wetted-out state was thermodynamically favoured. Materials could be adsorbed by a dipole mechanism without ionic charges or there could be double layers arising from ionic charges. This adsorbed materials helped repel one particle from another. No valid conclusions about dispersion could be drawn from sedimentation volumes and attempts to describe dispersion in terms of rheology were misleading as the time factor was usually ignored. By assuming spherical particles, uniform distribution and simple packing, it was possible to calculate the distances between particles for different particle diameters at various pigment volume concentrations. Dr. Smith concluded by defining perfect dispersion as a state in which aggregates were reduced to primary particles in a medium which wrapped, coated and wetted the particle so as to preserve its unit character.

After a short discussion period, Mr. G. Whitfield expressed the appreciation of the Section to the speaker for a well-prepared and most interesting talk on a fundamental subject.

L. R. S.

Newcastle

Industrial finishes

Dr. H. J. Sharp addressed the Section on this subject at its meeting on 12 November 1964. The Chairman, Mr. J. G. N. Smith, presided.

Dr. Sharp dealt first with the alternatives to organic finishes—vitreous enamel, bonded plastic films, electroplate, diffusion coatings and conversion coatings. All had their special properties and uses, but organic coatings (at $4d.-9\frac{1}{2}d$. per sq ft, applied) were the cheapest; vitreous enamel could vary between 6d. and 1s., nylon powder-coating 1s. 3d., and plastic pre-coated steel sheet 1s. 9d. Resistance to heat, deformation and moisture was discussed and, in connection with moisture, Dr. Sharp said that articles intended for "dry" use could suffer up to eight weeks' humid conditions during transit to the United States.

Dealing next with specifications, he said that only long-term service tests were truly reliable; consequently changes could not be made lightly. It was necessary to have short-term tests for day-to-day quality control. The deficiencies of these tests were seen, however, when an attempt was made to devise a whole series of tests for resistance to alkali, detergents, etc.; analysis of the results showed that they all measured only the moisture resistance of the films. Nevertheless, specific breakdown by these various agents did occur during intermittent exposure to them in the field, but it could not be reproduced in the laboratory.

In production quality control the paints were tested in the laboratory, and if satisfactory in production runs. Great emphasis was placed on the regular checking and, if necessary, prompt correction of stoving oven conditions.

Finally, Dr. Sharp outlined the five questions composing a "value analysis": (*i*) What is it? (*ii*) What will it do? (*iii*) What does it cost? (*iv*) What else will do the job? (*v*) What will that cost? These were simple but, if asked regularly, valuable. Consideration of all the elements of cost was most important.

Question time was lively and brought out a number of interesting new points. The lecturer's company had investigated water-based finishes and electrodeposition, but due to deficiencies of gloss and colour had not yet adopted them. New materials were always given fair consideration provided the claims were interesting and backed by adequate test results and the price was reasonable. When asked to enlarge on the point about laboratory tests with aqueous solutions measuring only the water resistance, Dr. Sharp said that some places were notoriously bad for washing machines, e.g. Malta, where the water may contain 0.1 per cent sodium chloride : this had a bad effect in practical, intermittent, long-term exposure, yet in the laboratory one might equally well use distilled water.

Dr. Sharp went on to say that in 10-15 years there would be no organic finishes used in the domestic appliance industry. The future lay with plastics, and at present the two most interesting were injection-moulded propylene co-polymers and compression-moulded polyester/fibreglass. The key to their use lay in designs compatible with their visco-elastic properties, and he was now studying this problem himself.

If paints were to be used he would like to see more latitude under varying conditions of stoving. A good approach to this might be a "triggered" system, which was stable under a certain threshold temperature, above which it cured very rapidly. He asked the opinion of the meeting about how British compared with United States' paints, saying that some years ago the latter were generally the better; the Chairman replied that this was not in his opinion generally so now; it was in some fields, in others we had the lead.

In giving the vote of thanks, Mr. N. Ellis agreed with this assessment; and complimented the lecturer on the clarity of his address.

D. M. J.

New South Wales

Pigments for printing ink

At the meeting of the Section held on 15 October, Mr. A. R. Barlee presented a paper entitled "Pigments for Printing Ink."

First, mention was made of the way in which printing inks were divided into four subdivisions, with reference being made to the distinguishing features of each group as they related to ink formulation, printing technique and the type of printed article. This then led to a consideration of the type of the various properties that were required of a printing ink and of the extent to which these could depend on the pigment; at this stage some of the methods that could be used to assess these properties were described.

After considering the way in which the Australian market for coloured pigments was split between the principal colour-using industries, an attempt was made to analyse the printing ink share in terms of the main pigment types which this industry consumed. A brief discussion about the distinguishing differences in performance between pigments of similar colour but different chemical type was followed by mentioning the way in which some of the required properties can be controlled during manufacture of the pigment.

L. J.

Scottish

The development of water soluble resins

The November meeting of the Section was held in More's Hotel, Glasgow, on Thursday 12 November at 7.30 p.m., when Mr. A. G. North delivered a lecture on "The Development of Water Soluble Resins."

Generally speaking the water soluble resins which were used for stoving purposes were not basically different from the solvent soluble type, most of them being alkyd/ phenolic, alkyd/melamine or maleinised oil systems. In order to achieve water solubility the amino resin must be etherified with a water soluble alcohol, for example when methanol replaces the more usual butanol. Since the acid value of the alkyds used was higher than normal, this resulted in a final system which possessed lower alkali resistance than was obtainable with solvent soluble systems. When pigments were incorporated these must not be sensitive to bases and they must contain no water soluble material. The resins possessed good corrosion resistance in their own right and, if corrosion inhibitive pigments had to be used, zinc chromate or lead chromate were to be preferred. Pigmentation should be at a lower level than for solvent based systems, a desirable PVC being 35 per cent, although a figure of 30 per cent was to be preferred, provided it is permissible.

One or more of a variety of additives might be necessary in water soluble systems. A small quantity of an alcohol was desirable to improve dispersion; lower alcohols increased the evaporation rate, while higher alcohols, such as butyl dioxitol, helped to keep the surface open. The use of such alcohols, in the small quantities which were customary, did not introduce any real hazard with regard to flammability or toxicity. The addition of an anti-foaming agent might be necessary, particularly in flow and dip coating techniques. Xylene and white spirit functioned quite well in this respect and the problem which was most likely to be encountered was loss by evaporation from the dip tank. If cissing or cratering were experienced due, perhaps, to a greasy metal surface or an excess of anti-foam, control by means of additives might be necessary. High molecular weight acrylic material was useful for this purpose while certain water soluble silicone analogues were available.

So far as usage of water soluble finishes was concerned, the conventional methods of application might be employed. Spraying could be done with normal equipment, a significant feature being that this could be achieved at appreciably higher viscosities than applied for solvent soluble systems. For dip application, water dispersible bentonites, used at a level of about 0.5 per cent, give superior pigment suspension and improve flow. Both conventional and *Rotodip* methods could be used. Water thinned products found many outlets as general industrial finishes, any lack of use tending to be due to conservatism and similar factors. One of the significant advantages of these materials was the possibility of improving the overall finishing lay-out, in that articles might move directly from the water rinse to the dip coat without any intermediate drying. Other advantages lay in freedom from solvent wash and the feasibility of electrodeposition.

In the electrodeposition process the object to be coated was made the anode in a low-solids paint bath (*circa* 10 per cent solids) and either a constant or variable voltage applied. The main advantages of the system were threefold : (*i*) If the system was correctly designed, very uniform coatings could be obtained; (*ii*) the deposited paint was of high viscosity and did not run; (*iii*) the deposited paint was insoluble in water and did not wash off and was not subject to solvent wash.

So far as the mechanism of the electrodeposition process was concerned, under the applied current the resin ion moved towards the anode and ammonia moved towards the cathode. As it approached the anode the resin became more acidic and eventually precipitated, the precipitated particles moving towards and depositing on the anode. The process involved is electrophoresis. Electro-endosmosis resulted in the exudation of water from the deposited resin. With emulsions and pigmented systems the basic mechanism was the same. Several factors related to the operation of the tank. Using constant current, there was a direct relationship between time and weight deposited, but, in practice, this was difficult to arrange. Using constant voltage, the normal method adopted, rate of deposition decreased with time. Faster rates of deposition resulted in coarser particle size, while low rates of deposition gave a finer, smoother surface with better gloss. Lower pH values in the bath gave heavier depositions, while higher values produced better paint stability. In practice, a compromise had to be struck in the region of pH 8. High pH could also result in some re-solution and this could be a problem in large dip tanks in which articles such as car bodies were being handled. The composition of the anode had a direct bearing on the results obtained in that electrolysis resulted in solution of the metal which tended to discolour the deposited paint; this was one of the basic disadvantages of the system at the moment and tended to limit its use to darker colours. The difficulty could be overcome, to some extent, by first depositing a barrier coat of conducting primer.

During discussion, Mr. Hutchison inquired about the present situation relative to water soluble decorative finishes. Mr. North replied that the main problem was getting rid of water solubility, while there was also the additional difficulty of discoloration arising from sensitivity to ammonia. There was no doubt, however, that such finishes would eventually be produced although, just at present, no one appeared to be quite sure how good they had to be before they would be accepted. Mr. Williamson asked what effects would be expected from hardness in the water used, and Mr. North replied that it was necessary to use as pure a system as possible. No adverse effects were experienced up to a certain level and, in general, there was a tolerance for each ion which varied with the ion and the resin. Mr. Pisacani asked what standards of surface preparation were required for electrodeposition. In reply, Mr. North said that, originally, it was thought that extremely clean surfaces were necessary. Now, however, due to modifications in resins and the use of additives, it was possible to deposit on fairly dirty surfaces.

A vote of thanks to Mr. North was proposed by Mr. W. Maxwell Young.

A. F. MCL.

Eastern Branch

Work study

A lecture on "Work Study," by Mr. J. Gloag (Heriot-Watt College, Edinburgh), was given at the first meeting of the new session of the Eastern Branch on 14 October 1964, held at the North British Hotel, Edinburgh.

"A fair day's pay for a fair day's work" was the early principle laid down by one of the originators of organised work study, F. W. Taylor, in the United States. The more detailed working of his plans in those days (1897) included the following :

- (i) Job selection/man selection.
- (ii) Encouragement for each man to perform up to the best performance in his class.
- (iii) Payment of bonus for such above-average work.
- (iv) Work measurement.
- (v) Functional foremanship.

Taylor was later named "the father of scientific management."

Mr. Gloag defined the "science" of management as more of an attitude of mind which directs attention away from the division of profit between management and men, towards the increase of profit to the two groups as a unit.

Finally, Mr. Gloag tabulated the logical and stepwise solution to management problems that come up in the day to day working of a factory and he stressed the need to eliminate working on "hunches."

The second half of the meeting was devoted to questions and discussions around the subject of Mr. Gloag's lecture.

Mr. Phillips mentioned his experience with the Bedaux system in 1930. Mr. Hutchinson also asked about this system and its use for piece-work in the textile industry.

To Mr. Holmes' question on Henry Ford's work study methods, Mr. Gloag replied that Ford was probably an originator himself in this field.

Mr. Bullions discussed the subject of overtime working, and Mr. Willis stated the problem of a short working week giving rise to men with double jobs. Mr. Gloag stated that this was a problem in the United States and, in answer to Mr. Sim, he enlarged on views he had formed during his recent visit there.

Mr. Bullions proposed the vote of thanks to Mr. Gloag and also extended a welcome to the visitors present.

Student Group

Tutorials

The November meeting of the Student Group was held in More's Hotel, Glasgow, on Saturday 14 November at 10 a.m. This was the second of this session's tutorials, when Mr. P. Birrell dealt with "Pigments," Mr. J. Miller with "Two-Pot Systems" and Mr. J. H. C. Davies with "Plastics." The meeting was well attended and Mr. D. M. Stewart proposed a vote of thanks to the speakers at the conclusion.

A. F. MCL.

West Riding

The colour of pigment crystals

The second meeting of the session was held on 13 October at the Great Northern Hotel, Leeds, when Dr. D. Patterson read a paper on "The Colour of Pigment Crystals."

The speaker introduced his subject by discussing the importance of particle size in determining the colour of pigments. This was illustrated in the case of white pigments by showing the increased scattering which resulted when the particle size of glass fragments was reduced. Similar dilution of colour could be seen when massive crystals of coloured minerals were crushed. This led to the view that the important optical properties of pigments which govern their colouring performance were the refractive indices and the absorption coefficients. In principle, he said, it was possible to calculate the colour of a pigment dispersion from a knowledge of these constants, but in practice the detailed analysis was not yet possible. It was necessary to use approaches to the problem such as those of Kubelka and Munk and developed by Duncan and others, in which the absorption and scattering coefficients of pigments were related to the reflectivity of a film in which they were dispersed. The scattering coefficients in turn depended on the refractive index and particle size.

However, Dr. Patterson said he had made some measurements of both refractive index absorption coefficient of a number of azo pigments. Previously the only available values were those of Cooper, reported in the Journal in 1948. The work described was carried out on recrystallised pigments and it had been checked by X-ray diffraction photographs that the crystal structures of the pigments had not been altered by the recrystallisation. Absorption measurements on the crystals were made using polarised light and a specially built microspectrophotometer. In every case it was found that the crystals were dichroic, i.e. the absorption of plane polarised light depended on the direction in which the light was travelling relative to the crystallographic arrangement of the molecules in the crystal. The crystals were also found to be birefringent, that is, had at least two different refractive indices, again depending on the direction in which polarised light travelled in them. Absorption curves for the crystals were shown for light travelling in the fast and slow directions through the crystal. In all cases the absorption in the slow direction was greater in extent, and the maximum absorption was at a longer wavelength, than in the fast direction. The reason for these differences, he said, could be understood when the mechanism of absorption of light by a single molecule is considered, as it will be greatest when the plane of polarisation of the light coincides with those of the π orbitals. However, unless the crystal structure of the pigment was known, it was not possible to relate the absorption properties of the crystal directly with those of a single molecule. There was, he confessed, unfortunately a dearth of knowledge of the crystallography of organic colouring matters.

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In conclusion, the speaker said that the relevance of these findings in the fields of pigment usage was that if by some shearing process, such as extrusion or brushing, pigment particles became preferentially oriented in a medium, the colour would change with the direction of viewing. In the theoretical field it made the calculation of the colour of a dispersion from the basic optical properties of the constituents even more complicated than had been anticipated.

L. J. W.



LOUIS ARNOLD JORDAN, C.B.E., D.SC., A.R.C.S., F.R.I.C., M.I.CHEM.E.

LOUIS ARNOLD JORDAN

23 September 1892 to 1 December 1964

A Personal Appreciation

Dr. Louis Arnold Jordan, C.B.E., was the Director of the Research Association of British Paint, Colour and Varnish Manufacturers from its inception in 1926 until his retirement in 1959. He was 72.

After a distinguished academic career at the Royal College of Science he was concerned during the 1914-18 war with explosives and other chemical matters and was a member of the British Commission to Italy, receiving the honour of Chevalier of the Order of the Crown of Italy. Then followed post-war appointments in the chemical industry before a period, 1923-26, as scientific adviser to the State of Bhopal.

Always active in scientific associations and technical education, his offices over the years included those of Chairman of the Committee of Directors of Research Associations, Chairman of the Council of the Society of Chemical Industry, President of the Oil and Colour Chemists' Association, Chairman of the City and Guilds Advisory Committee on Paint Technology, and Chairman of Governors of Kingston College of Technology. He had been a member of the Senate of London University. From 1958 to 1962 he was Professor of Chemistry at the Royal Academy of Arts.

His pioneer work in co-operative scientific research for the British industries concerned with coating compositions, natural and synthetic resins, pigments and vegetable oils attracted attention overseas; in the United States, in Europe and in the East, particularly India, where his advice was sought by the Government of India on technical organisation and research in the lac industry.

In 1961 he made the last of a series of three recent visits to India. He also visited Thailand and East Pakistan on lac problems. He made official visits to Malaya and to East and West Pakistan to report on wider issues of scientific activity and research organisations in those countries. In 1948 he was created C.B.E.

(With acknowledgement to The Times Obituary, 4 December 1964.)

It seems but a short time since we recorded our tribute to Dr. Jordan at the time of his retirement. His activity did not then cease and to the record then given (*JOCCA*, October 1959) there are additions of official journeys on scientific missions, continuous contact with paint technical matters at home and abroad through the International Union of Pure and Applied Chemistry, continued technical education work and advice to industry. The record, summarised in *The Times* obituary, is clearly of achievement, eminence and a crowded life, but this appreciation is more concerned with the man. For many will remember him in the personal sense, and memories come crowding back; memories of a large, forceful personality of great ability; friendly, humorous, sometimes controversial; an individualist with a strong sense of occasion; a great character. One who was quietly proud of his staff's successes, giving them full support in any office which they held, had a deep love for his Research Station and performed many, frequently hidden, acts of kindness.

He had tremendous drive, strongly expressed ideas and, in pressing towards a mark, firmness of purpose and quality of exposition which enabled him to capture the imagination and loyalty of many, though occasionally treading on an errant toe. Not infrequently his ideas and schemes were ahead of the times ; some not then accepted have since been looked at again. He was a big man, big enough to accept strong disagreement by intimates, but any discordance was tempered, and made temporary, by that strong sense of humour and friendliness. If undue familiarity by a subordinate led to near impudence he called it " impishness," and laughed not at, but with, the would-be opposer.

His urgency of purpose sometimes led to bizarre situations. A wartime memory is of a needed report for a Ministry for which someone was carpeted for dilatoriness. The panels had not yet been examined because they needed five days' ageing. There's a war on, he said, stop up all night and age them. Such situations dissolved in merriment and were recognised as manifestations of his desire for action, and his own teeming activity and mental productivity. Indeed, if any proof of that were needed, the war provided it. He was here, there and everywhere ; flying to inspect camouflage schemes for which materials were developed at Teddington, lecturing to service personnel, controlling a wide range of investigations for Ministries and Services-and finding time for senior office in the fire service, with the staff as an operational unit.

Active in technical education, he was himself a natural teacher. A pioneer and leader himself, he admired leaders and pioneers in science, industry and the arts. The transitional regions between craft and science fascinated him, well beyond our own industries. He had a strong feeling for the past (it was an experience to hear him, perhaps over a late night drink at an OCCA conference, on the history of India, for example), but significantly his two favourite titles among his writings were *Paint, the Art* and the Science and Forward Thinking on industrial materials and processes.

For ourselves, the members of OCCA, the cold record says that he was our President in 1947-49 and became an Honorary Member in 1955; but the warm memories will be more personal and of the manner as well as the content of speeches at dinners, inaugurations of new Sections and other special occasions. He was an excellent impromptu speaker but, for the weightier philosophical and technical addresses, many small hours were spent in careful selection and arrangement of material and the writing and rewriting of the telling phrase. Then there was his pride in being the first wearer of the Presidential Insignia. He was much concerned with the design and with the motto, Et mente et manu, and its signification, much in his mind, as we know, when he prepared the first Carwood Memorial Lecture in 1963.

Nor must we forget the part played by Mrs. Jordan, his constant companion and support. She is much admired by the many who know her, and her joining with him over the years in the full spirit of OCCA and PRS functions, formal and social, is pleasantly memorable. She always enjoyed his enjoyment as well as his more serious successes. Many in OCCA will remember him on social occasions for more than his speechmaking. Many of his staff will recall him at parties dressed up as Father Christmas, or sitting in the front row for the sketches, waiting to be burlesqued by some member of the staff, laughing when he recognised the portrayal of some of his idiosyncrasies, but complaining afterwards if he failed to recognise himself in them. He much enjoyed a parody of the Admiral's song in *HMS Pinafore* which contained the words *The paint trade said* they must attain the making of their products on a higher plane, they got together and they founded the . . . and now I've got my name in the Director-ee.

And he sang at parties ; moreover, he rehearsed. There was a point halfway up the staircase where he would stand and sound off a broad, deep note which rang round the entrance hall with a mighty resonance. And on that same staircase, there on the bottom step in troublous times, he would sit, after the day's work, with his head in his hands ; then suddenly he would get up and say "Well, let's get on with it."

The development of the Research Association was the core of his life's work and, as we said in our 1959 appreciation, he established and fostered "an organism (not just an organisation) with a character all its own" and many past and present staff members are sensible of it. They got their training there, they did their work, they had their meeds of praise, and sometimes their admonitions, and they are proud of it. The sense of entity came, no doubt, from the development from small beginnings under strong, purposeful guidance and sense of mission, but also from personal acts of kindness by The Old Man. He was nearly always called that among his staff, even in his forties, occasionally "Dr. Jordan" and seldom "the Director "; a merely formal recognition of his position seemed inadequate.

Now *The Old Man* has gone, but it will be many, many years before he is forgotten. His passing has brought numerous private tributes from at home and abroad; tributes to his wide ranging intellect, activity and achievement—and expressions of gratitude.

Torquay Conference

Recent progress in coatings technology

The names and brief biographies of the lecturers for the Association's Conference at Torquay were announced in the November and December issues of the *Journal*.

The Honorary Research and Development Officer (Mr. A. T. S. Rudram) has now reported to Council on the order in which the lectures will be given and the Chairmen for the three sessions as follows :

Wednesday 26 May (9.30 a.m.-12.30 p.m.)

Chairman—Dr. F. W. Salt (Head of Long-Range Research, Swansea Laboratories, British Iron and Steel Research Association).

Alkali Silicates in Surface Coatings (with special reference to Zinc-Alkali Silicate Systems), by Mr. A. Pass and Mr. M. J. F. Meason. The first part of the paper will give a brief account of the history of soluble alkali silicates, their properties and manufacture on a commercial scale, including some reference to recent developments in the production of high ratio silicates and of lithium silicates. A general account will be given of the industrial uses of such materials, and particularly of the somewhat limited usage of the so-called "neutral" silicates in rather specialised coating materials up to the early 1940s, when zinc metal-alkali silicate coatings first appeared on a commercial scale.

A second part of the paper will discuss the development and commercial applications of zinc metal-alkali silicate coatings from the introduction of the earliest types requiring a heat treatment prior to exposure, to the more recent "self-curing" types which can be applied to large structures *in situ*. The properties



The Palace Hotel at which the technical sessions and social functions of the Conference will be held

and technical value of these coatings will be discussed, with special reference to specific application where they appear to offer some advantages over more conventional coating methods.

The final part of the paper will give an account of some experiments carried out on the setting mechanism of zinc metalalkali silicate films. The chemistry of the soluble silicates is highly complex, and various explanations have been put forward to account for the film-forming properties of the alkali silicate as such and the zinc metal-alkali silicate systems. It is hoped that the present experiments will help to clarify the position.

The Electrodeposition of Paint; Some Basic Studies, by Mr. A. R. H. Tawn and Mr. J. R. Berry (Cray Valley Products Ltd.).

The paper will treat the subject of electrodeposition of paint essentially from the standpoint of physical chemistry. It will be presented in two parts. In the first the nature of the phenomena involved in the transport and deposition of the paint solids will be discussed, with particular reference to the relationship between composition and behaviour. Laboratory studies of selected topics centring on the properties of some "model" polymers and mixtures thereof will be described.

The second part of the paper will cover a study of some basic factors of technological significance, *viz.* electrical efficiency, paint losses and their minimisation, "throwing power" and bath control.

The Influence of the Surface-Treatment of Titanium Dioxide Pigments on Electrocoating, by Dr. H. Rechmann.

Numerous titanium dioxide pigments, actually on the market, are post-treated with aluminium and silicon compounds. The present paper deals with the influence of different quantities and of quantity proportion of these two substances upon the behaviour of the pigment during electrocoating. Migration and tendency to flocculation, as well as the brightness and gloss of baked enamels, are tested in the presence of various vehicles whereby the pigmentation, as well as the duration of electrophoresis, has been varied.

Thursday 27 May (9.30 a.m.-12.30 p.m.)

Chairman—Dr. L. Valentine (Director of the Paint Research Station). Surface Coating of Metals in a Glow Discharge, by Dr. T. Williams.

The paper is concerned with the formation of thin (less than 5 microns thick) non-porous polymer films on metal surfaces in a glow discharge. A summary is given of the relevant work reported in the literature. Experimental investigations, carried out to determine the effect of various discharge parameters on the rate of polymerisation and the properties of the films produced, are described in detail. Possible polymerisation mechanisms are discussed and some of the phenomena associated with the process are theoretically considered.

The possibility of depositing thin films on moving strip is critically examined. Estimates are given of the capital and running costs of a possible commercial plant; these are compared with the capital and running costs of an equivalent roller coating unit.

The article is concluded with a list of potential uses for glow discharge coatings.

Characteristics of Electrostatic Powder Spraying, by Dr. K. M. Oesterle and Dr. I. Szasz.

Following the use of the falling beam technique to investigate the kinetics of lacquer spraying, it can be shown that an analogous method can be developed for electrostatic powder spraying and deposition.

The results obtained show that the following powder characteristics have a considerable influence, namely (i) dielectric constant, (ii) conductivity, (iii) self-electrification, (iv) particle size, shape and charge, (v) water absorption.

The properties of electrostatic spray apparatus which influence the result

include the electrical and geometric configuration of the electric field and atmospheric conditions.

A special procedure enables the suitability of the powder for electrostatic deposition to be established by a few measurements and provide for optimum spraying and deposition effects. Practical examples will be shown.

Powder Coatings—Adhesion and Permeability Studies, by Mr. L. A. Tysall and Dr. J. R. Weber.

The paper will deal with an examination of the adhesion and water permeability of films of several different types of epoxy powder coatings and a comparison of these with films of thermoplastic powder coatings, a solventless liquid epoxy coating, epoxy compositions similar to the powders but containing solvents, a conventional solvent-based epoxy coating and a high quality alkyd/MF coating.

The effect of varying the stoving schedule and the pigmentation for the epoxy powders will be reported. Adhesion to various metal substrates will also be reported. The correlation between these properties and such film properties as the resistance to boiling distilled water will be discussed.

Friday 28 May (9.30 a.m.-12.30 p.m.)

Chairman—Dr. W. F. Watson (Director of Research, Rubber and Plastics Research Association).

Allyl Ethers in Solventless and Water-Based Coatings, by Dr. L. A. O'Neill and Mr. R. A. Brett.

Of the various ways of curing films at normal temperatures, autoxidation is the most widely exploited. The property of drying oils of curing by autoxidation was utilised long before the structural features promoting autoxidation were known. Mere unsaturation in an organic compound does not necessarily lead to ready autoxidation and the essential structural requirements for an autoxidising system have been examined.

One of the simplest groupings with which autoxidising properties can be introduced is the allyl ether group, which has already been used for the development of some products, for example allyl starch and allyl ether modified unsaturated polyester resins. The mechanism of autoxidation of the allyl ether system has therefore been studied with the aim of indicating its most useful potential field.

The allyl ether compounds are particularly attractive with the present interest in solventless and water-based systems. For solventless coatings the simple allyl ethers which are low viscosity liquids can be blended with autoxidising resins such as alkyds and build themselves into the cured film. For waterbased systems the allyl ethers may, in the form of partially etherified polyhydric alcohols, be incorporated into watersoluble alkyds. Paint vehicles have been developed by both these routes and their properties examined.

The Chemistry of the Vinyl Cyclic Acetals and Their Air-Drying Reactions, by Dr. S. Hochberg.

The preparation of a synthetic liquid which will harden on exposure to air, like the common drying oils or like the oil modified alkyd resins, has long been an objective of research in the finishing industry. After a considerable amount of experimental and theoretical work, the vinyl cyclic acetals and some of their derivatives were selected as promising commercial materials.

The vinyl cyclic acetals are derived from acrolein and compounds containing two neighbouring hydroxyl groups. Such compounds absorb oxygen and are converted into acrylates. By the use of compounds containing additional functionality, molecules containing a plurality of vinyl cyclic acetal groups and other functioning unsaturation can be developed.

The synthesis of these air-drying compounds and their subsequent reactions which lead to the formation of films will be discussed. The relationship of the chemistry of these compounds to the types of materials obtained on drying will be taken up. Side reactions leading to undesirable effects will also be taken up. While the air-drying compounds have not become commercial film formers, their chemistry will be of interest to people interested in oxidation, polymerisation and formulation of finishes.

Polyurethane Systems for Solvent-Free Coatings, by Dr. H. Gruber.

Polyurethane lacquers are renowned for their high resistance to mechanical and chemical stresses. One of the characteristics of these coatings is their capacity for variation of formulation for a very wide range of applications. By varying the choice of isocyanate and/or the hydroxyl-containing component, elastic or enamel-like coatings may be obtained.

These same virtues are possessed by the solvent-free polyurethane systems and by varying the choice of the liquid base products the final compound can range from hard to soft. The raw materials for these coatings can be based on natural products such as castor oil.

A survey is given of the mechanical properties of coatings based on castor oil and a number of isocyanates.

For the production of pore-free compounds it is necessary to combine any moisture present in the raw materials. The method of combination is discussed.

Finally, a survey of the practical uses of solvent-free polyurethane systems is given. The very latest experience with these materials is made available.

Reprints of these synopses are available to members and non-members upon application to the General Secretary.

In addition to the foregoing papers, on the afternoon of 28 May, following the Association's Annual General Meeting, three workshop sessions will be held, admittance to which will be by ticket only. The titles of these sessions will be "The Influence of Moisture on Surface Coatings," "Critical Design of Experiment" and "International Harmonisation of Testing Methods."

Programmes and registration forms for the Conference will shortly be sent to all members of the Association in the United Kingdom and General Overseas Sections; copies will also be sent to the Honorary Secretaries of the Sections in South Africa, Australia and New Zealand in the event of any members from these Sections wishing to attend. The Council has fixed the registration fees at £6 for members, £4 for wives and £8 for nonmembers, with a reduced fee of £3 for junior members. Non-members will receive copies of the *Journal* containing the discussions.

Headquarters for the Conference will be the Palace Hotel, Torquay, and accommodation will also be arranged in the Babbacombe Cliff, Queens, Osborne and Victoria Hotels. A Civic Reception will take place on the evening of 26 May and the Association Dinner-Dance on the evening of 28 May. A full and varied social programme has also been arranged for the afternoons and details are given in the Conference programme.

All registrations must be received at the Association's offices not later than 1 April, since the allocation of hotel accommodation has to be made immediately after that date.

Non-members wishing to receive Conference programmes and registration forms should write immediately to the General Secretary, Oil & Colour Chemists' Association, Wax Chandlers Hall, Gresham Street, London, E.C.2.

17th Technical Exhibition

The Exhibition, which will take place for the first time in the Great Hall of Alexandra Palace, London, N.22, provides a focus for the technical display of advances made in the raw materials, plant and equipment for the paint, printing ink and allied industries. In order to assist those visiting the Exhibition, a map showing the various ways in which Alexandra Palace can be reached from Central London will be reproduced in each copy of the Official Guide, which will be sent, without charge, to all members of the Association in January. Copies will also be sent individually to chemists and technologists in Western Europe and Scandinavia, to technical colleges and, through the courtesy of the trade associations, to companies in the paint, printing ink and pigment industries in the United Kingdom.

The map, which appeared on page 843 of the October issue, is also being reproduced separately in a folder which gives directions in four languages (French, German, Italian and English) and copies of these will be sent with the Official Guides despatched to the Continent. Copies are also available to intending visitors and they will be despatched without charge upon application to the General Secretary of the Association. The four-language cards will also be sent to many paint and printing ink manufacturing companies on the Continent of Europe.

A free bus shuttle service will be operated by the Association from Wood Green Underground Station (Piccadilly Line) and there are ample free car parking facilities at Alexandra Palace.

The total number of stands allocated for the Exhibition will be 105 and, of the companies showing, 24 have never shown at previous OCCA Technical Exhibitions, while 16 others did not show at the 1964 Exhibition. Amongst the overseas countries from which exhibits will be shown are Belgium, Denmark, France, East and West Germany, Holland, India, Norway, Sweden, Switzerland and the United States of America.

The Committee is pleased to report that the Rt. Hon. Lord Hill of Luton, P.C., Chairman of the Independent Television Authority and a Director of Laporte Industries Ltd., has accepted its invitation to be the Guest of Honour at the Exhibition Luncheon, which will be held at the Savoy Hotel, W.C.2, on 22 March, and to reply to the Address
of Welcome by the President, Dr. J. E. Arnold. Principal officers of other scientific bodies, industrial research associations and organisations representing both suppliers and consumers will be invited to attend. A form of application for tickets, which are priced at £2 12s. 6d. each, is enclosed in each copy of the *Official Guide*, or may be obtained from the General Secretary. All applications for tickets must be received by 8 March, since it will not be possible to accept late applications.

The Exhibition will be open for the first time on five days and the Committee feels that this arrangement will benefit companies, particularly those in the provinces and overseas wishing to arrange a rota for their technical staff to visit the Exhibition. The hours of opening will be as follows :

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22 March	• •	3 p.m. to 6.30 p.m.
Tuesday 23 March		10 a.m. to 6 p.m.
Wednesday 24 March		10 a.m. to 6 p.m.
Thursday 25 March		10 a.m. to 6 p.m.
Friday 26 March		10 a.m. to 4 p.m.

As in previous years, a stand will be devoted to Technical Education and, on this occasion, it will adjoin the Association's Information Centre in the centre of the Hall. The theme of the Technical Education Stand on this occasion will be *Chemistry and Physics in the Study of Pigment Behaviour.* Already many schools

have accepted invitations to send parties of "A" Level Science students to the Exhibition on the mornings of 23, 24 and 25 March, when they will be given a short introductory talk by members of the Association in a separate Lecture Hall before visiting the Exhibition. The Technical Education Stand will be staffed by representatives from the Association, technical colleges and the trade organisations, and details will also be shown of the courses available in the technology of the industries.

Representatives from 30 overseas countries attended the 1964 Exhibition, and in order to assist the increasing number of both visitors and exhibiting companies, interpreters will again be in attendance, either at the Information Centre or at Stand 82. There will be no charge for admission to the Exhibition, or for copies of the *Official Guide*, which will be available from the Association's Information Centre at the Exhibition or from the Association's Offices prior to the Exhibition.

Amongst the facilities available at Alexandra Palace are two restaurants in the Palace Suite (the Edinburgh Room reserved, and the Alexandra Room unreserved) together with two cafeteria and several bars.

Any company or individual who wishes to receive copies of the Official Guide should write to the General Secretary, R. H. Hamblin, M.A., F.C.I.S., F.C.C.S., Oil and Colour Chemists' Association, Wax Chandlers' Hall, Gresham Street, London, E.C.2 (MONarch 1439, Ext. 3).

ALPHABETICAL LIST OF EXHIBITORS

† Albright & Wilson (Mfg.) Ltd. Allied Colloids Ltd. Amalgamated Oxides (1939) Ltd. Amoco Chemicals Corporation
* Anchor Chemical Co. Ltd. Bakelite Ltd.
* Beckman Instruments Ltd. Berk, F. W., & Co. Ltd. BIP Ltd. Blagden, Victor, & Co. Ltd. Boehm, Fredk., Ltd. Boulton, William, Ltd. British Celanese British Oxygen Co. Ltd. British Resin Products Ltd. British Titan Products Co. Ltd. Bush, Beach & Segner Bayley Ltd. *Byk Gulden Lomberg GmbH Carless, Capel & Leonard Ltd. *Chemische Industrie Synres, NV Chemische Werke Huels AG (Continued overleaf)

(Exhibitors—continued) *Churchill Instrument Co. Ltd. Ciba (ARL) Ltd. Ciba Clayton Ltd. **†Coal Products** †Cole, R. H., Ltd. *Compagnie Francaise Goodyear *Copley, F., & Co. [†]Cornelius Chemical Co. Ltd. †Cox's Machinery Ltd. Cray Valley Products Ltd. *Craymer Inventions (Eng.) Ltd. Croxton & Garry Ltd. D. H. Industries Ltd. *Dispersions Ltd. **Dunlop Chemical Products Division** *Elcometer Instruments Ltd. Farbenfabriken Bayer AG Ferranti Ltd. *Gebruder Netzsch Geigy Co. Ltd., The Grampian Press Ltd. Greeff, R. W., & Co. Ltd. Hardman & Holden Ltd. Hercules Powder Co. Ltd. [†]Hoechst Chemicals Ltd. Imperial Chemical Industries Ltd. †Imperial Smelting Corporation Ltd. *Industrial Colours Ltd. †Isopad Ltd. Johns-Manville Co. Ltd. Kemisk Vaerk Køge A/S Kingsley & Keith (Chemicals) Ltd. Kronos Titanium Pigments Ltd. Kunstharsfabriek Synthese NV Laporte Titanium Ltd. Lennig Chemicals Ltd. *Little, J. H., & Co. Ltd. *Livingston Laboratories Ltd. *Marfleet Refining Co. Ltd. *Maschinenfabrik Heidenau V.e.b. *Meyer's, Rudolph, Incorporated †Mill Room Accessories & Chemicals Ltd.

London Section

Ladies' Night

Two hundred and eighty-six members and guests made their way to the Criterionin-Piccadilly on Friday 20 November for the London Section Ladies' Night, where they were received officially by the Chairman, Mr. M. R. Mills, and Mrs.

Mitchell, L. A., Ltd. †Mitchell, W. A., & Smith Ltd. National Adhesives Ltd. Nederlandse Castoroliefabriek Necof NV *Norsk Spraengstofindustri A/S [†]Norwegian Talc, A/S Novadel Ltd. *Optica United Kingdom Ltd. Paint, Oil & Colour Journal Paint Research Station *Pfizer Ltd. Plastanol Ltd. Premier Colloid Mills Ltd. *Pye, W. G., & Co. Ltd. †Reeves & Sons Ltd. Research Equipment (London) Ltd. Sawell Publications Ltd. Scado-Archer-Daniels NV Sheen Instruments (Sales) Ltd. Shell Chemical Co. Ltd. **†Shellac Export Promotion Council** Silverson Machines Ltd. Spelthorne Metals Ltd. Steele & Cowlishaw Ltd. *Sterling Colour Co. Ltd. Styrene Co-Polymers Ltd. Surface Coating Synthetics Ltd. †Swada (London) Ltd. Titanium Intermediates Ltd. Torrance & Sons Ltd. †Translation & Technical Information Services *Unicam Instruments Ltd. †United Coke & Chemicals Co. Ltd. Vinyl Products Ltd. *Weyburn Engineering Co. Ltd. Winkworth Machinery Ltd. *Denotes exhibitors who have not shown at previous

Exhibitions. †Denotes exhibitors who did not show at the 1964

Exhibition.

Mills, and the President of the Association, Dr. J. E. Arnold, and Mrs. Arnold. When all had arrived, and been called to dinner by the toastmaster, Mr. Edwin Spencer, the assembled company received the Chairman, the President, the guest of honour, Dr.



Photo by courtesy

Paint, Oil & Colour Journal

(From left to right) Mrs. Harrison, Dr. V. G. W. Harrison (Guest of Honour), Mrs. Mills, Mr. M. R. Mills (Chairman, London Section), Mrs. Arnold, Dr. J. E. Arnold (President)

V. G. W. Harrison, Director of PATRA, and their ladies with acclamation. This year the dinner had an Italian flavour, and the linguists amongst us were kept busy interpreting *Rombo Garibaldi* and *Pollo alla Montanina*—and very good it was too.

Dr. Harrison said that he had had a happy association with the London Section for 27 years. He had a twofold connection with the Association, through the printing ink industry and as a do-ityourself home decorator. Considerable technical progress had been made with both printing inks and paints, and such advances resulted from the existence of a technical centre for the industry operating at a high scientific level. OCCA was one of the most vigorous and prosperous technical societies of the industries, with a valuable journal and an extremely successful annual exhibition, which together contributed towards the technical development of the industries they served. The strength of the Association depended on the activity and prosperity of its Sections and Branches, and he had great pleasure in proposing the toast of "The London Section of the Oil and Colour Chemists' Association."

Mr. Mills responded to the toast on behalf of the members, thanking Dr. Harrison for his kind remarks. The Association had always been grateful for the support received from PATRA. OCCA was founded some 50 years ago, and was supported by the industries, both directly by advertisements in the Journal and by participation in the Exhibition, and indirectly by the encouragement given to technical staff to present papers. The strength of an association was measured by the enthusiasm of its members, and we were fortunate to have a number of enthusiastic volunteers for office. He was glad to welcome one of the most distinguished, Dr. Arnold, together with Mrs. Arnold. He was also

glad to welcome the President-elect, Dr. S. H. Bell, who had undertaken a large amount of work for the Association in the past and would wear the presidential insignia with distinction. Mr. Mills also welcomed the past Chairman of the London Section, Mr. A. T. S. Rudram, who was present as the Association's Research and Development Officer, and Mrs. Rudram, the Chairman of the Scottish Section, Dr. D. Atherton, and Mrs. Atherton, the Chairmen of the Southern and Thames Valley Branches, Mr. C. A. Carev and Mr. W. J. Arnot. and their ladies, and the General Secretary, Mr. R. H. Hamblin, and Mrs. Hamblin. Mr. Mills had a special word for the ladies, who added colour and charm to the gathering, and revealed that he had recently attended a lecture on feminine charm. He then asked the members to rise for the toast of "The Guests and the Ladies."

Mrs. C. A. Carey, responding on behalf of the ladies and guests, confessed to having had feelings of apprehension,

Manchester Section

Visit to Ford Motor Company, Halewood On the morning of 26 November, 42 members of the Section visited the modern car manufacturing plant at Halewood. This visit was arranged following a successful visit in September, when the number of members was limited.

Scottish Section

Essay competition

The Scottish Section has decided to run its essay competition for the session 1964-65 with modifications which, it is hoped, may go some way to enhance its appeal.

In the first place, the candidate is now left free to choose his own subject. It is felt that this should do much to encourage participation, in that the student will be able to write on a subject in which he has which had been removed by the kind words from the Chairman. She referred to a recent speech by the Director of the British Colour Council, in which the ladies were said to lack ability in home decor and management. This was untrue ; the ladies always had to do the planning beforehand. On this occasion, however, it was very enjoyable to be spared this task, and to be able to sit down to the delightful Italian menu-without pasta! Having sat in the body of the hall on many previous occasions, she knew that the members and guests would be wanting to get on with the dancing, and therefore had pleasure in proposing thanks on behalf of the ladies and guests.

After a short interval, the Chairman and Mrs. Mills led the dancing, which continued until 1 a.m. to the accompaniment of George Fierstone and his Orchestra, with Mr. Edwin Spencer acting as MC. Once again a very enjoyable social occasion had come to an end.

V. T. C.

The visit followed the same lines as that previously recorded in the November issue of the *Journal*, although on this occasion car bodies painted with the new metallic acrylic finishes were also noted with interest.

W. F. MCD.

a special interest or on work on which he has been actively engaged.

The second innovation lies in the fact that the prize offered is a visit to the Association Conference at Torquay, from 25 to 29 May, 1965, the subject of which is "Recent Progress in Coatings Technology." All reservations will be made and all expenses paid by the Scottish Section, who feel that this should provide a significant inducement to candidates.

The competition is strongly commended to Junior Members of the Scottish Section Student Group and the cooperation of all Chief Chemists and Technical Directors is sought in encouraging their staff to participate.

Competition Rules

1965

- (i) Eligibility : The competition is open to all who are bona fide Junior Members of the Scottish Section Student Group at the closing date of the competition (Monday, 1st March, 1965).
- (ii) Subject : The candidate is left free to choose his own subject, the only obvious limitation being that it must fall properly within the field of the surface coatings industry. If he has any doubt as to the suitability of his subject, he should consult the Scottish Section Committee, either directly or through his Chief Chemist. Sub-

ject matter may either be a record of work done or comment on some aspect of the industry or its technology. In the former case, permission to submit the essay should, clearly, be obtained from the candidate's employer.

- (iii) Length : The length of the Essay is not critical, but should be in the region of 4,000 to 5,000 words.
- (iv) Closing Date : All entries should be submitted to the Hon. Education Officer of the Section by Monday, 1 March, 1965.
 - (v) Assessment: The judging of the entries will be in the hands of the Committee of the Scottish Section. whose decision will be final.
- (vi) Prize : One prize will be awarded, this being an "all-expenses-paid" visit to the Association Conference at Torquay, from 25 to 29 May, 1965.

West Riding Section

Chairmen's golf trophy competition

In the early part of 1964 the West Riding Section Committee decided to organise a knock-out golf competition for members of the Section only. A suitable silver cup was presented to the Section jointly by Dr. K. Hargreaves (present Chairman) and Mr. C. Allsop (immediate past Chairman), to be competed for annually, and it was decided to call the event "The Chairmen's Golf Trophy Competition."

During the spring and summer the various rounds were played and R. J. Hardy and J. M. Shields emerged as the finalists. The final was fixed for 20 September at the Oakdale Golf Club, Harrogate, and it was decided to hold a Stableford competition for the other members at the same time.

The weather on the day of the final could only be described as "typically British." Heavy showers, glorious sunshine and high winds did not help towards low scoring, and one competitor commented that he was changing in and out of waterproof clothing more frequently than members of the fair sex are reputed to change their minds. In spite of the conditions, everyone had a most enjoyable afternoon, and J. M. Shields won the trophy after a hard fought match, and C. Butler won the Stableford competition.

After the golf a number of members' wives joined the players in an excellent meal at the clubhouse; there were a number of very informal speeches, and the trophy was presented to the winner by Mrs. C. Allsop. It was duly "christened" in the time-honoured fashion and the event came to a close. Everyone voted the entire competition a great success and there will no doubt be an even larger entry in 1965.



Fourth Congress of the Scandinavian Federation

A technical session in progress

The Fourth Congress of the Scandinavian Federation of Paint and Varnish Technicians was held in Helsinki, Finland, 1-3 October. Among the 200 guests from 11 countries was Dr. H. A. Hampton (President 1961-63), who represented the President of the Association.

After an opening address in four languages by the Scandinavian Federation President, Mr. H. Furuhjelm, in which he spoke of the feeling of affinity that was felt by the Federation towards OCCA, the technical sessions started.

On the Thursday three papers were delivered (the session concluded with a panel discussion on "Testing Methods"); these were : (i) "Hiding Power and Film Thickness," by Dr. S. Wilska, (ii) "Notes on the Hardening Procedure of Melamine/ Alkyd Resin Combinations," by Mr. L. Augustsson, (iii) "The Paint Research Station : its Work on Media," by Dr. L. Valentine.

On the Friday further papers were presented : (i) "Viscosities by Very High



Dr. H. A. Hampton reading his paper to the Congress

and Low Shearing," by Mr. P. Fink Jensen, (*ii*) "Urethanes in Surface Coatings," by Dr. H. A. Hampton, (*iii*) "Development in pva Dispersions for Paint," by Dr. K. Gauslaa. Also during the morning session there was a discussion on "Education of Technicians in the Paint and Varnish Industry," which was preceded by four short papers.

The social side of the Congress was not overlooked and was typical of Finnish hospitality. Thursday evening consisted of a small informal dinner given by Mr. Furuhjelm to the principal guests and a trip to the theatre for some other delegates. On Friday there was a sightseeing tour of Helsinki, and in the evening the gala dinner was held at the Brando Casino. In his speech, Mr. Furuhjelm expressed his hopes for the continuance of the close co-operation between the Scandinavian Federation and OCCA. At the close of his address he wished his successor, Mr. Helge Meyer, of Sweden, well in his term of office.

Dr. Hampton conveyed greetings from the Association and thanked Mr. Furuhjelm for his kind words. Later a telegram from the Association's President, Dr. J. E. Arnold, was read to the assembled guests.

British Joint Corrosion Group

As already mentioned in the December issue of the *Journal* (page 1022), the Association has been notified of the formation of a British Joint Corrosion Group under the auspices of the Iron and Steel Institute, the Society of Chemical Industry, the Institute of Metals and the Institute of Metal Finishing. The Organising Committee of the Joint Group has now decided that, in addition to the four sponsoring societies, interested bodies should be invited to become Co-operating Societies ; and accordingly the Oil and Colour Chemists' Association has been so invited.

Members of the Association will, therefore, be given equal rights to join the Joint Group without charge, to attend meetings and to subscribe to the *British Corrosion Journal* at a reduced rate. The reduced rate will be for the 18-month period July 1965-December 1966, £3 5s., with future annual subscriptions at the rate of £2 10s. The invitation to the Association to become a co-operating society will continue for a trial period of 12 months and will then be the subject of review by the Organising Committee.

Any members of the Association wishing to be registered as a member of the British Joint Corrosion Group should apply for the registration form to F. J. Griffin, Esq., O.B.E., Secretary, British Joint Corrosion Group, 14 Belgrave Square, London, S.W.1, and in completing the form under item 2 should state that they are members of this Association.

Seminar on Fatty Acids

A seminar on fatty acids will be held at the Regional Research Laboratory, Hyderabad-9 (India), during the first week of February 1965. The seminar will be conducted in two broad sessions—academic and industrial. In the academic session research papers will be presented covering the latest advances in the field of fatty acids and their derivatives. The industrial session will consider various aspects of manufacture and utilisation of fatty acids as well as their standards and specifications. Further particulars may be obtained from the director of the laboratory.

News of members

Mr. W. P. Jenkins, an Ordinary Member attached to the Newcastle Section, has recently retired from the position of Chief Chemist to British Paints Ltd., being the first full-time works chemist employed by the Company. He was a founder member of the Newcastle Section and served as its Honorary Secretary from 1942-46, then as its Chairman from 1946-48, and subsequently as a Vice-President of the Association from 1948-49.

His other activities include a long membership of the Technical Panel of BISRA and the chairmanship of the Paints Panel of the Research Association of British Paint, Colour and Varnish Manufacturers.

Following the Annual General Meeting of the British Colour Makers' Association, it has been announced that Sir Christopher Cowan, J.P., an Ordinary Member attached to the London Section, has been appointed Chairman for 1964-65. The other officers are Vice-Chairman, Mr. J. Smethurst, an Ordinary Member attached to the Manchester Section, and the Honorary Treasurer, Mr. G. E.

W. Pollard

It is noted that the death has occurred of Mr. William Pollard, F.R.I.C. Mr. Pollard, an Ordinary Member attached to the Manchester Section, was Technical Director for the last 15 years of W. W. Hillier, an Ordinary Member attached to the London Section. Other members of OCCA elected to the Council are Mr. A. K. Ames, an Ordinary Member attached to the Manchester Section, Mr. C. M. Beavis, an Ordinary Member attached to the Bristol Section, Mr. W. B. Cork, an Ordinary Member attached to the Hull Section, and Mr. C. L. Lewis, an Ordinary Member attached to the Manchester Section.

Mr. J. Wright, an Ordinary Member attached to the London Section, has been awarded the City and Guilds of London Institute Insignia Award for a thesis entitled "The Development of Surface Coatings Resistant to Chemical, Thermal and Climatic Stress." We congratulate Mr. Wright on this award.

Hill, Son & Wallace Ltd., which Company he had joined as a Laboratory Assistant in 1924. A member of various panels of the Research Association of British Paint, Colour and Varnish Manufacturers, he was travelling to Teddington for a meeting when he died.

Register of Members

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

Ordinary Members

AMARNATH, ANGIAH SAHASRANAM, B.SC., A.R.C.S.T., C/O MESSTS. Alexander, Fergusson & Co., 50 Ruchill Street, Glasgow, N.W. (Scottish)
 ARTHUR, JOHN REID STUART, B.A.(CANTAB), Wing Cottage, St. Leonards Dale, Winkfield Road, Windsor, Berkshire. (London)
 ASHTON, BRIAN LESLIE, L.R.I.C., 15 Cherrywood Court, Bundora, Victoria, Australia. (Victorian)
 BEATON, CAMERON, 709 Moss Park Drive, Cardonald, Glasgow, S.W.2. (Scottish)
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BORTHWICK, REGINALD, 49 Fitzwilliam Road, Vaucluse, New South Wales, Australia. (New South Wales)

BOWDEN, JOHN CAMPBELL, L.R.I.C., 21 Merrick Gardens, Glasgow, S.W.1. (Scottish) BRAAGAARD, PETER L., Skovgaardsvej 31, Charlottenlund, Denmark. (Overseas) BRAY, JOHN MARMION, B.SC., 51 Serrell Street, East Malvern, Victoria, Australia. (Victorian)

CRAIG, ROBERT, Meldorf, 35 Willoughby Drive, Whitley Lodge Estate, Whitley Bay, Northumberland. (Newcastle)

FAIRBAIRN, PETER FORBES, A.S.T.C., 52 Eileen Street, Balgowlah, New South Wales, Australia. (New South Wales)

FRIIS-HANSEN, JØRGEN, M.SC. CHEM. ENG., 15 Prebens Vaenge, Kgs. Lyngby, Denmark. (Overseas)

KENT, NOEL RICHMOND, B.SC., A.R.I.C., 10 Boxhill Way, Strood Green, Betchworth, Surrey. (London)

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MEYER, HELGE HOLGER, M.SC., Goteborgsvagen 103, Savedalen, Sweden. (Overseas) MILES, ALAN KENNETH, B.SC., 18 Tottenhall Road, Palmers Green, London, N.13.

(London)

MYATT, BERNARD EDWARD, 79 Endhill Road, Kingstanding, Birmingham, 22. (Midlands)

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Notice Board

(The following information concerning conferences of other organisations, which may be of interest to members, is taken from the DSIR" Forthcoming International Scientific and Technical Conferences." For further information contact the General Secretary at the address shown on the front cover)

1965

20 January

Society of Dyers & Colourists' 12th London Lecture "The Nature of Colour"

8-12 February

Meeting of the American Society for Testing and Materials.

20-26 February

16th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy.

21-25 February

Annual Meeting for TAPPI.

21 April-2 May

36th International Congress of Industrial Chemistry.

10-12 May

TAPPI Coating Conference.

19-21 May

11th Congress of the International Federation of Associations of Textile Chemists and Colourists.

31 May-2 June

5th Australian Spectroscopy Conference.

9-11 June

International Rubber Conference.

16-20 August

38th Congress of the Australian and New Zealand Association for the Advancement of Science (ANZAAS).

21-23 September

16th Testing Conference (TAPPI).

10-13 October

20th Engineering Conference (TAPPI).

10-16 November

3rd International Congress and Exhibition on Measurements and Automation (Interkams).

Forthcoming Events

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

Monday 4 January

Hull Section. "Linseed Putty, a Simple System," by R. R. Davidson, at the Royal Station Hotel, Hull, at 7.15 p.m.

Thursday 7 January

London Section. Symposium, "Paint Versus Corrosion," at the Imperial College, South Kensington, S.W.7, at 3 p.m.

Newcastle Section. "Recent Developments in Thermosetting Acrylic Resins," by P. V. Robinson and K. Winter, at the Royal Turks Head Hotel, Newcastle, at 6.30 p.m.

Friday 8 January

Bristol Section. "Printing and Coating on Plastic Films," by T. Sutcliffe, at the Royal Hotel, College Green, Bristol, at 7.15 p.m.

Monday 11 January

London Section—Southern Branch. "Possibilities with Emulsions in the Paint Field," by Dr. P. M. Troll, at the Queen's Hotel, Southsea, at 7.30 p.m.

Hull Section. Discussion Evening at the Queen's Hotel, George Street, Hull, at 7.15 p.m.

Tuesday 12 January

West Riding Section. "Furoic Acids in Alkyds," by V. F. Jenkins, at the Great Northern Hotel, Leeds, at 7.30 p.m.

Wednesday 13 January

Scottish Section—Eastern Branch. "Developments in Water Thinnable Resins," by J. R. Berry, at the North British Hotel, Princes Street, Edinburgh, at 7.30 p.m.

Thursday 14 January

London Section—Thames Valley Branch. "Design of Experiment," by A. R. H. Tawn, at the Royal White Hart Hotel, Beaconsfield, at 7 p.m. Refreshments at 6.30 p.m.

Midlands Section—Trent Valley Branch. (Provisional.) Open Discussion (Joint Meeting with NFMP and D) at British Railways School of Transport Lecture Theatre, London Road, Derby, at 7.30 p.m.

Scottish Section. "A General Review of Driers," by J. W. H. Turner, at More's Hotel, Glasgow, at 7.30 p.m.

Friday 15 January

Midlands Section. "Raw Material Trends," by A. Mott, at Birmingham Chamber of Commerce, at 6.30 p.m.

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

Saturday 16 January

Scottish Section—Student Group. Tutorials: (1) "Alkyd Resins," by W. Maxwell Young, (2) "Emulsion Paints," by A. McLean, (3) "Driers," by D. Atherton at More's Hotel, Glasgow, at 10 a.m.

Wednesday 20 January

London Section—Junior Group. "The Rheology of Printing Inks," at London College of Printing, Elephant and Castle, London, S.E.1. Lecturer and time to be advised.

Friday 22 January

Manchester Section. "New Organic Pigments—The Chemistry and Properties of the Derivatives of 4, 5, 6, 7 Tetrachloro 180-indolene 1,-one" by A. Pugin at the Manchester Literary and Philosophical Society, George Street, Manchester, at 6.30 p.m.

Friday 29 January

Bristol Section. "Painting and Coating of Synthetic Wood-Based Boards," by L. E. Akers, at the Royal Hotel, College Green, Bristol, at 7.15 p.m.

Bristol Section—Irish Branch. "Electrophoresis," by A. G. North, at the Dolphin Hotel, Essex Street, Dublin, at 8 p.m.

Monday 1 February

Hull Section. "Colour Matching from an Art to a Service," by J. L. R. Landry, at the Royal Station Hotel, Hull, at 7.15 p.m.

Thursday 4 February

Newcastle Section. "Some Marine Coating Problems," by J. C. Kingcome, at the Royal Turks Head Hotel, Newcastle, at 6.30 p.m.

Friday 5 January

London Section—Thames Valley Branch. Buffet Dance at the Riviera Hotel, Maidenhead, at 8 p.m.

Monday 8 February

London Section—Southern Branch. "Some Applications of Marine Paints," by Dr. T. A. Banfield, at the Royal Hotel, Cumberland Place, Southampton, at 7.30 p.m.

Wednesday 10 February

Scottish Section—Eastern Branch. "Some Aspects of Heating Treatment in the Motor Industry," by J. Davidson, at the North British Hotel, Princes Street, Edinburgh, at 7.30 p.m.

Thursday 11 February

Scottish Section. "Zinc Dust and Flake Pigments," by D. A. Atkinson, at More's Hotel, Glasgow, at 7.30 p.m.

Saturday 13 February

Scottish Section—Student Group. AGM and Members' Discussion Morning, at More's Hotel, Glasgow, at 10 a.m.

Tuesday 16 February

Hull Section. "Colour—A Brief Survey," by W. B. Cork, at the Queen's Hotel, Hull, at 7.30 p.m. (The Section are guests of the HCES.)

London Section—Thames Valley Branch. "The Permeability of Paint Films to Water," by Dr. R. W. Wilson, at the Royal White Hart Hotel, Beaconsfield, at 7 p.m. Refreshments at 6.30 p.m.

Wednesday 17 February

London Section. "Dirt Pick-up of Paints," by G. L. Holbrow, at Manson House, Portland Place, London, W.1, at 7 p.m.

Thursday 18 February

New South Wales Section. Eighteenth Annual General Meeting at the Theaterette, MLC Building, Miller Street, North Sydney, at 8 p.m.

Friday 19 February

Manchester Section. "Technological Education and the Paint Industry." A dialogue between Prof. R. H. Peters and Dr. H. W. Keenan, at the Manchester Literary and Philosophical Society, George Street, Manchester, at 6.30 p.m.

Midlands Section. "Powder Coatings," by C. H. Morris, at Birmingham Chamber of Commerce, at 6.30 p.m.

Thursday 25 February

Midlands Section—Trent Valley Branch. "Some Effects of Pigmentation on Electrodeposition of Paint Films," by L. Tasker and J. R. Taylor, to be read by L. Tasker, at British Railways School of Transport Lecture Theatre, London Road, Derby, at 7.30 p.m.

West Riding Section. Joint Meeting with the Society of Chemical Industry on Pigment Wetting. Introductory Paper, "Pigment Dispersion Agents," by Mr. Schwitzer, and "Pigment Dispersion and Pigment Structure," by R. J. Cole, at the Great Northern Hotel, Leeds, at 7.30 p.m.

Friday 26 February

Bristol Section. "Printing and Coating on Aluminium Foils," by J. R. Green, at the Royal Hotel, College Green, Bristol, at 7.15 p.m.

Thursday 18 March

New South Wales Section. "Physical Chemistry Aspects of Some Paint Problems," by Prof. Alexander, at the Theaterette, MLC Building, Miller Street, North Sydney, at 8 p.m. A DCL tanker taking on supplies of DCL Bisol chemicals. From a nationwide network of factories and depots, DCL's fleet of road and rail tankers provides a prompt delivery service to any part of Britain. Chemicals in bulk or drum, of consistent quality, and backed by technical advice—DCL delivers the goods in full measure.

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As the name indicates, this machine is designed to permit not only 2 different experiments to be carried out simultaneously, but will also take different containers to produce a variety of different mixing effects to suit the work in hand. It is equally suitable for fluids and solids, material in powder and granule form, since apart from using different containers, speed can also be varied within the range of 0 to 240 r.p.m.



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