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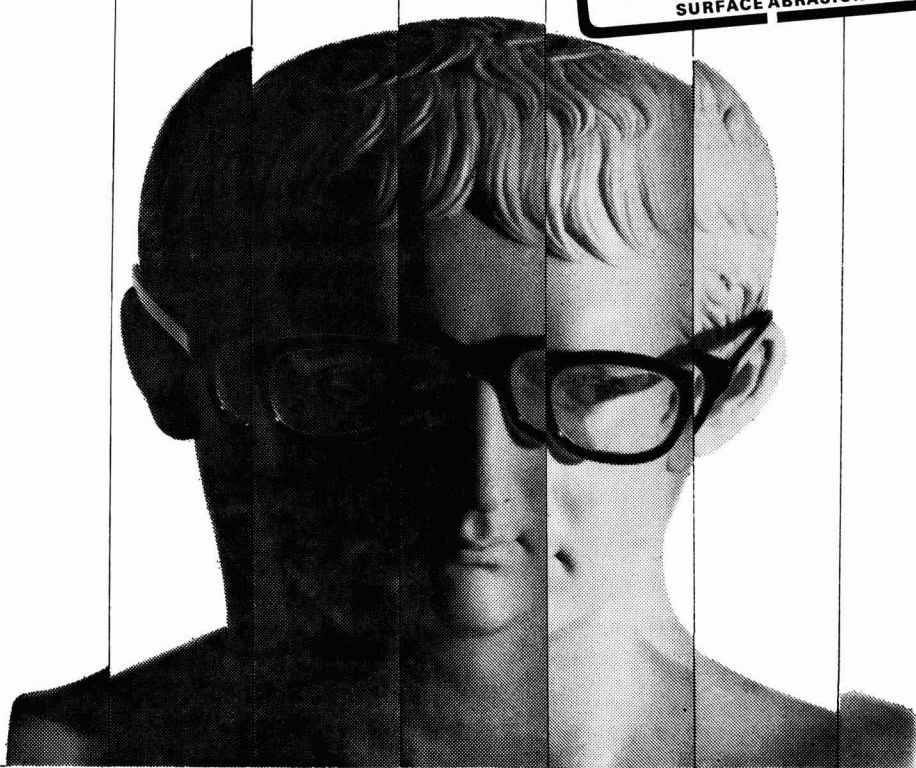without discolouring binder
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The motif for 1976, designed by Robert Hamblin, continues the theme of the last two Exhibitions—in which attention was drawn to the heavy participation by exhibitors and visitors from overseas—by showing the target for 1976 as London, where all the previous Association annual Exhibitions have been held

TRAVEL ARRANGEMENTS

Since the Association's last Exhibition at Alexandra Palace, travel arrangements have been improved by the addition of the Victoria Line to the Underground system, which now links Victoria Station to the Piccadilly Line at Finsbury Park. The building of the extension of the Piccadilly Line from Hounslow to the Airport terminal at Heathrow is now well advanced and when this is completed in due course it will give a direct link with Turnpike Lane Station on the Piccadilly Line. In the meantime, a bus service operates from Heathrow Airport to Hounslow West Station. Those travelling by the Piccadilly Line should alight at Turnpike Lane Station and the Association will be running a bus shuttle service from this station to and from Alexandra Palace. The journey from central London to Turnpike Lane takes approximately 18 minutes

Reproductions are available of the two-colour map shown on page 394. Members of the Association may obtain copies by sending a stamped self-addressed envelope, marked "Alexandra Palace map" in the top left-hand corner, to the Association's offices at the address on the Contents page opposite

JOURNAL of the
OIL AND COLOUR CHEMISTS' ASSOCIATION

Contents   **Vol. 58 No. 10**   **October 1975**

Transactions and Communications————————————————————————

Prediction of the corrosion protective properties of paint films by permeability data *

By H. Haagen and W. Funke

Forschungsinstitut fuer Pigmente und Lacke e.V., Stuttgart, Germany

Summary

The possibility of replacing long term exposure tests for assessing the corrosion resistance of coatings by shorter term permeability measurements is considered.

It is found that, in general, the permeability of coatings by water is sufficiently great to provide all the water necessary for corrosion to proceed at the substrate/film interface, but that this is not always the case with oxygen, whose permeation rate may be lower than needed for the corrosion process.

The permeability of anions, such as Cl^- and $(SO_4)^{2-}$ is extremely low, so that underfilm corrosion caused by them must be due to contamination of the surface prior to coating and not to their diffusion through the film.

Keywords

Process and methods primarily associated with service or utility

   diffusion

   electrolytic corrosion

   underfilm corrosion

Properties, characteristics and conditions primarily associated with dried or cured films

   permeability

La prédiction des caractéristiques anti corrosives de feuils de peintures à l'aide des données sur leur perméabilité

Résumé

On considère la possibilité de remplacer les essais de vieillissement à long terme pour apprécier la résistance à la corrosion de peintures au moyen des mesures de la perméabilité à relativement court terme.

On trouve, en général, que la pénétration de l'eau en les peintures est suffisante d'en fournir la quantité nécessaire à amorcer la corrosion à l'interface entre le subjectile et le feuil, mais ce n'est pas le cas où il s'agit de l'oxygène dont le taux de pénétration est insuffisante pour favoriser le processus de corrosion.

La pénétration des anions tels que Cl^- et $(SO_4)^{2-}$ est extrêment faible, de sorte que toute corrosion provoquée par ces ions sous le feuil doit être due à la contamination de la surface du subjectile avant l'application du feuil et non pas à leur diffusion à travers le feuil.

Voraussage der Korrosionsschutzeigenschaften von Anstrichfilmen auf Grund von Permeabilitätswerten

Zusammenfassung

Es wird die Möglichkeit betrachtet anstelle von langdauernden Bewitterungsversuchen den Korrosionsschutzwiderstand von Anstrichen durch kürzere Permeabilitätsmessungen zu ersetzen.

Die Untersuchungen ergaben, dass die Wasserdurchlässigkeit von Beschichtungen meist ausreicht, um die Korrosion des Untergrundes wie bei ungeschützten Substraten ablaufen zu lassen.

Die Durchlässigkeit für Sauerstoff kann aber geringer sein als für den normalen Ablauf der Korrosion notwendig ist.

Ionen wie Cl^- oder $(SO_4)^{2-}$ können Anstrichfilme, wenn überhaupt, nur in sehr geringem Masse durchdringen. Eine Beeinflussung der Unterrostung durch Ionen dürfte daher auf bereits am Anstrichuntergrund vorhandene Verunreinigungen zurückzuführen sein und weniger durch eine Diffusion durch den Film.

Introduction

Refs. 1-7

It has been known since Mayne[1], Kittelberger and Elm[2] about thirty years ago applied the electrochemical theory of corrosion to paint coatings, that a paint coating must prevent anodic and cathodic reactions on the metal surface. The extent to which corrosion may occur under paint films containing no anticorrosive pigments will be governed essentially by the permeation rates of corrosive agents, such as oxygen, water and ions, to the metallic substrate and of corrosion products, such as iron and hydroxyl ions, to the film surface, as well as by the adhesion being maintained under exposed conditions.

Preceding any corrosion reaction adhesion must be lost, at least locally, due to the action of corrosive agents. As will be referred to later, water has been shown to be primarily responsible for this. It would be expected therefore, that the permeation rate of water can be correlated with the rate of adhesion loss during the exposure of a paint film to corrosive media.

For the corrosion reaction to occur to a substantial extent, a supply of both water and oxygen is necessary and the corrosion rate should be governed by the slowest of these permeation processes.

Finally, the corrosion products, as ferrous and hydroxyl ions, may influence the corrosion reaction by mass action and local polarizing corrosion effects.

The concentration of these ionic corrosion products will depend on whether or not they can leave the interfacial space at all, and if so, how fast they can leave compared with the rate of possible sequential reactions involving oxygen, water and other ions present. The fate of the corrosion products has

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*Paper presented at the Association's Biennial Conference held at the Grand Hotel, Scarborough, Yorks, England, from 17 to 21 June 1975.

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another important implication; the corrosion protective properties of paint films are still mainly evaluated in practical tests by visual examination of the paint samples after a chosen time of exposure to a corrosive medium. This means that the "practical" criteria, according to which corrosive protective properties of paint films are commonly classified, do not apply to the corrosion reaction itself, but to the reactions and processes subsequent to it, which may depend on still other parameters. This may provide a key to the understanding of why in the literature, permeation rates are not unanimously considered to be very useful criteria for estimating the corrosion protective quality of a paint film. Only recently have some results been reported, which are sufficiently promising to anticipate a deeper insight into the mechanism really relevant to the problem of corrosion testing of paint films[3,4]. The authors current work aims to supply additional and more extended experimental data in order to see whether the rate at which corrosion takes place under a paint film, can be related to the transport of water, oxygen and ions through the film. If this can be done, as anticipated, the advantage would be that coatings could be evaluated more reliably and more quickly, since it would not be necessary to rely only on time-consuming exposure tests, and so forth. Another important reason for this work is that at present it is not known for certain whether the common anticorrosive pigments, like red lead and chromates, will be restricted in use or even banned completely in the future, for reasons of toxicity. Then it would be necessary to concentrate even more on the properties of the paint films for which so-called "active pigments" are not needed. If no active pigments are used, paint films can protect metallic substrates from corrosion only if the substances necessary for corrosion are prevented from reaching the film/substrate interface. Paints meeting this requirement fortunately seem to be feasible.

A large amount of data can be found in the literature on the permeability of water and water vapour through paint films. These data normally cannot be compared, however, since they are obtained under different conditions. As far as permeability for ions[5] and oxygen[4] is concerned, much less

reliable data are available. Futhermore, the data mentioned have been obtained with free films and the question arises whether these data apply to films attached to a substrate. There are some indications from literature[6,7] that the structure of paint films adjacent to the phase border differs from that present in the bulk material. Apart from this difference, the method used to remove the film from the substrate may also have some effect. The influence of these factors on corrosion must be considered when judging the results of permeability measurements.

Permeabilities

Refs. 3, 8

Permeability to water

Baumann[3] has calculated the amount of water which must be present on a metal surface to allow corrosion at a certain rate. A comparison of the calculated values with data obtained by measurement shows that in the majority of paint films penetration of water occurs faster than is needed for corrosion. Table 1 shows the calculated and measured data for some paint coatings.

The fact that in many cases more water can be supplied by permeation than is needed for corrosion to occur shows that water permeability in most cases cannot be the rate determining step in this process. This does not mean that either the role of water in corrosion or its destructive action on paint coatings should be underrated. As has been demonstrated by Walker[8], water penetrating to the substrate can reduce or even destroy adhesion of a paint coating. Therefore, water may well be the rate determining factor in the loss of adhesion. With some paint coatings, adhesion may be regained to some extent on drying out, especially if the films are not too old and the process is not repeated too often. Loss of adhesion under wet conditions, however, does not necessarily mean accelerated corrosion. There are paint coatings which rapidly lose adhe-

Table 1
Permeability of paint films for water vapour at 23–25°C[3,9,10]

Vehicle base	Pigment Type and PVC	Film thickness (μm)	Permeability (mg/cm²/day)	Gradient of relative humidity (%)
Acetylcellulose[9]	—	100†	36	100/ 0
Epoxy-polyamide[10]	Fe₂O₃ 35%	100†	5	100/ 0
Cellulose nitrate plasticised[9]	—	100†	4.8	100/ 0
Melamine resin water soluble[9]	—	100†	4.8	100/ 0
Chlorinated rubber[10]	Fe₂O₃ 35%	100†	3.5	100/ 0
Alkyd resin[10]	Fe₂O₃ 15%	100†	2.3	100/ 0
Alkyd resin[10]	Fe₂O₃ 35%	100†	2.1	100/ 0
Polyurethane[9]	—	100†	1.4	100/ 0
Polyester resin[9]	—	100†	1.3	100/ 0
Phenolic resin[9]	—	100†	1.1	100/ 0
Epoxy-coal tar[10]	—	100†	1.1	100/ 0
Chlorinated rubber[9]	—	100†	1.0	100/ 0
Alkyd resin[3]	Pigm.	100†	0.6†–1.7†	90/ 0
Cellulose nitrate combination[3]	Pigm.	100†	3.42–5.48	90/ 0
Polyvinylchloride, chlorinated[3]	Pigm.	100†	0.39–0.78†	90/ 0
Alkyd-amine-resin[3]	Pigm.	100†	1.4†–3.8†	90/ 0
Water necessary for corrosion at a rate of 0.02–0.35 mg Fe/cm²/day (according to Baumann)		—	0.003–0.06	—

†Permeabilities calculated or interpolated for 100μm film thickness

sion under moist conditions. However, if not mechanically stressed or damaged they do not show substantial corrosion even after several months' weathering. Fig 1 shows to what extent humidity impairs adhesion. According to these results, even coatings with very strong initial adhesion may exhibit a considerable decrease with increasing relative humidity, especially from approximately 60 to 70 per cent upwards.
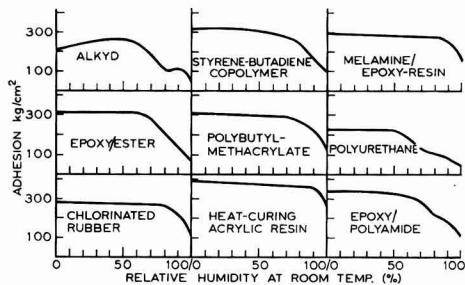
Fig. 1. Influence of humidity on the adhesion of paint films on steel (after P. Walker[8])

Permeability to oxygen

In addition to water, oxygen is prerequisite for atmospheric corrosion. Here again the amount necessary for corrosion at a certain rate can be calculated. A comparison of the calculated values with the data obtained from measurements appears significantly different from those obtained in the case of water. Whilst it is true that permeability data available for oxygen at present are limited, nevertheless, they show that the permeability of some of the coatings examined is either below the calculated threshold or within the range of oxygen supply necessary for corrosion to occur; in a few cases only is the permeation rate of oxygen higher (Table 2). Moreover, these data show that the temperature and to some extent the humidity prevailing during the measurement may influence permeability.

As would be expected, film thickness is another important factor. Experiments with the same types of chlorinated rubber and vinyl films at various film thickness and dried under the same conditions have shown that the permeation coefficient is not constant. Thinner films have shown lower permeability coefficients than thicker ones. However, if the thicker films

are dried longer under more severe conditions they approach the permeability coefficient of the thinner films. The most likely reason for this is that volatile substances, such as solvents, escape under given drying conditions more easily from thinner than from thicker films.

With air drying and stoving enamels a somewhat smaller, but similar tendency was noted. Here permeability to oxygen is again a complex matter. Therefore, permeability data of various films may be compared only if the measurements have been carried out under exactly the same conditions with films having the same prehistory.

Permeability to ions

The question arises whether data on the permeability of paint films to water and oxygen alone are sufficient to predict results which agree with those obtained by a phenomenological classification of the corrosion resistivity of a paint system, in connection with usual practical tests, such as salt spray, the Kesternich test, and the blister box. This includes also the question whether salt spray and similar tests are really indicative of corrosion protective properties or whether corrosion is due simply to the porosity or other small imperfections of the paint film. It has been mentioned that the phenomenological criteria depend on the formation of rust, blisters and so forth, where ions, like Fe^{++} and OH^-, are involved. Since other ions (for example chloride and sulfate) can be present on a surface and may stimulate corrosion, they have to be taken into account, no matter how the corrosion protective properties are judged.

Permeability to chloride ions

Ref. 5

Conductivity measurements using a conductivity cell have shown that, at least within the range 40 to 60 μm film thickness, no detectable amount of chloride ions[5] penetrate intact films, except with highly hydrophilic and swellable ones, such as those based on acetyl cellulose. On the other hand, this means that chlorides present on the substrate before paint application (for example, from atmospheric pollution), cannot leave the interface film/substrate on exposure to humidity or water. These ions will be retained under the film, and in the presence of moisture, will continue their destructive reactions if not neutralised, for example by anticorrosive pigments. Films become permeable to chloride ions only if highly pigmented, that is, in the range of the critical PVC.

Table 2

Permeability to oxygen of paint films of 100μm film thickness. Partial pressure of oxygen = 2 × 10³ N m²

Vehicle base	Permeability for oxygen at various temperatures (10^{-3} mg/cm²/day)				
	10°C	20°C	30°C	40°C	50°C
Cellulose nitrate	69.3	105.8	149.8	237	318
Alkyd melamine resin	4.2	10.3	19.2	30.3	50.6
Epoxy resin	4.7	7.3	8.6	17.8	33.7
Vinylchloride/vinylacetate 87/13 copolymer,					
(i) air dried	3.9	7.5	10.5	29.9	37.5
(ii) force dried at 60°C	4.1	4.9	6.4	10.7	19
Chlorinated rubber	1.1	2.2	3.4	4.3	15.9
				(45°C)	(60°C)

Calculated[4] amount of oxygen necessary: from 8 to 150 × 10^{-3} mg/cm²/day for a corrosion rate ranging from 0.02 to 0.35 mg Fe/cm²/day

The films tested included plasticised cellulose nitrate, a vinylchloride-vinylacetate copolymer, an alkyd/melamine resin stoved at 140°C for 30 minutes, a water dispersible acrylic ester polyester stoved at 140°C for 30 minutes, a water thinnable short oil (castor oil) alkyd resin and a water soluble melamine resin stoved at 140°C for 30 minutes. In addition, two pigmented electrocoating primers were used, one of which was based on an oil-modified polycarbonic acid neutralised with an aliphatic amine and the other on an epoxy/phenolic resin, neutralised with an aliphatic amine.

The tests were continued for up to 100 hrs. The limit of sensitivity of conductivity measurements was 8.5×10^{-5} mg/cm² day.

Permeability to sulfate and hydroxyl ions

Ref. 5

It may be assumed safely that there is no substantial difference between the chloride and these anions[5]. Investigations are continuing. The observation that cathodic blisters are highly alkaline provides strong evidence that paint films are impermeable also to hydroxyl ions.

Permeability to iron ions

On exposure of coated steel panels to condensed water at approximately 35°C, no iron ions, which could have permeated through the film even at a film thickness as low as 20 to 30 μm could be detected. Again, the films become permeable only if pigmented in the range of the critical PVC. It is commonly agreed that apart from steric hindrance, another reason for the barrier function of a paint film towards negative ions might be its negative charge. However, this does not explain why there is also a barrier function against positive ions, such as iron ions. As far as iron ions are concerned, they could be precipitated from solution by oxidation in cases where oxygen penetrates fast enough to react with them before they can leave the film. This point will have to be elucidated by further investigations.

Conclusions

At high relative humidities, many coatings allow more water to penetrate to the metal surface than is needed for the corrosion reaction to proceed. A comparison of permeability data with the amount of water necessary for corrosion

occurring at normal rates as calculated by Baumann and supported by the authors' results, supplies strong evidence for the validity of this statement. Permeability to water, therefore, in many cases cannot be the rate determining step of corrosion. It can, however, determine the speed with which a paint coating loses adhesion and also the extent of loss of adhesion.

Further evidence shows that for a number of paint films, the permeability to oxygen is lower than that required for corrosion, which is in accordance with the results of Baumann. The relative humidity of the atmosphere above a paint film of a given thickness and still more so the temperature, influences the amount of oxygen which penetrates a unit area of film in a given time.

Considering the limits of sensitivity of the detecting device used for chloride ions, several of the paint films tested could be considered impermeable to chloride ions. There is some evidence that this is true also for hydroxyl and sulfate ions, in the case of clear films or those not too highly pigmented, for example, top coats. Consequently, chloride and sulfate ions will penetrate neither through the film from outside to the substrate nor from the substrate through to the outside. It may be concluded, therefore, that in case of a non-contaminated substrate, the behaviour of such a coating might be predicted by measuring the permeability rate for water and oxygen. Since on practical exposure, the climatic conditions may vary considerably, the data will have to be determined under selected conditions of humidity and temperature similar to the anticipated environmental conditions of the applied coating.

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Discussion at Scarborough Conference

DR J. E. O. MAYNE said that it was now 28 years since he had pointed out that an unpainted mild steel specimen exposed to the industrial atmosphere at Sheffield consumed 74 micrograms/oxygen/cm²/yr. It could be seen from Table 2 that the majority of films were permeable to 10 per cent or more of this amount. It followed that if the diffusion of oxygen were the controlling factor, the majority of films in Table 2 would reduce corrosion by 90 per cent or less. In fact during the early stages of exposure, no corrosion was detectable and protection had to be due to some other factor.

It was suggested that this factor was the high electrolytic resistance of the film. This resistance might slowly diminish, in the case of immersed specimens, by a process of ion exchange, or under conditions of atmospheric exposure by slow degradation of the polymer, which was brought about by radiation.

DR HAAGEN thanked Dr Mayne for his comments; he had shown how difficult was this subject. The properties of a paint film, including permeability, depended very much on the conditions during aging of the film and it was often for this reason that it was not possible to compare data from two or more experiments.

As far as the electrical resistance of the films was concerned, Dr Haagen agreed that this was very high and thought this could account for the reason why even unpigmented films prevented corrosion for quite a long time. On the other hand, some water and oxygen had to be present on the surface of the substrate for corrosion to occur and it was, therefore, very important to consider the practical conditions during application of the films when discussing permeability data.

In addition, what one was judging visually did not always relate explicitly to the initial corrosion reaction. What was

being studied by visual inspection was the condition of the film under rusting and the amount of corrosion products formed. The amount and nature of the products would depend not only on the starting conditions but also on such factors as how long the film was exposed to dry periods or wet periods or the frequency between these. Nevertheless there always had to be some permeability to water and oxygen. What they were trying to achieve was some form of rating for different paints so that it would be possible to say, for example, that paint X would have a better chance of survival under such-and-such conditions than paint Y and the rating for a new paint could then be determined much more quickly than by either a natural weathering or accelerated weathering test.

PROF. FUNKE commented that they had found the diffusion coefficients to depend on the conditions of the film, and the coefficients were variable in that respect. In preference, they had used diffusion rates in the paper and found these, generally, to be more useful.

DR K. M. OESTERLE asked whether the measurements had been carried out on free films or films attached to a substrate. This would make a great difference because as soon as a film was detached from its substrate its inner structure was changed.

DR HAAGEN answered that it was true that the values were different for supported and unsupported films. He had mentioned that if the supported films reached absorption equilibrium in the time period concerned, then they would absorb less moisture than the free films; if, on the other hand, they did not reach absorption equilibrium, the amount of moisture they absorbed was much higher than in the case of free films. This additional moisture must have come from the film/substrate interface. There was some substrate effect.

PROF. FUNKE commented that the old method used to measure this property was simply to immerse the coatings in water, take them out after a period of time and dry them with blotting paper. The authors found that this method gave some guidance but a disadvantage was the length of time—usually about seven days—necessary to show some measureable change. With the newer method of using an electric balance which the authors had employed, it took a much shorter time—say one or two hours—to show some effect.

MR L. J. BROOKS said that his point was probably of only elementary experimental detail, but he wondered how the authors dried the oxygen. To put this another way, he wondered how the authors differentiated between the effect of oxygen and moisture, bearing in mind the dominant role of the latter.

PROF. FUNKE remarked that the results had shown some effect of water content on oxygen permeability although not so much as if the water had acted as a plasticiser upon the binder material. It was not to be expected that the water would react with the polymer as a normal plasticiser would do, and so the results were perhaps not so surprising.

MR A. F. SHERWOOD reminded the authors that they had stated that the permeability to sulfate ions was low, so that any corrosion due to sulfate had to arise from contamination of the surface before painting. The paint films were probably permeated by sulfur dioxide, however, and this might then react with the oxygen which had permeated through the film, in the presence of water on the substrate surface, to form sulfate ions.

PROF. FUNKE replied that this possibilty should be considered, especially if the SO_2 concentration were considerably higher than that to which the substrate had been exposed before coating.

MR A. N. MCKELVIE commented that when there was sunshine in this country there were often showers of rain at the same time and steel structures exposed to sunlight, even for example motor cars, could attain very high surface temperatures and after a shower of rain could be seen steaming with water vapour. It was very appropriate, therefore, that the authors were studying permeability at high temperatures. The elevated temperatures were still in line with conditions which might pertain in practice, and the increased oxygen permeability at these temperatures was certainly above the threshold limit for initiating corrosion in the presence of water.

MR P. FINK-JENSEN asked whether the loss of adhesion was in fact a prerequisite of corrosion. He thought it possible that the process could start with corrosion, soon followed by loss of adhesion caused by the osmotic activity of the corrosion products.

PROF. FUNKE replied that this was like the very old question of which came first the chicken or the egg. It was his view that for any supported paint film, there were points of adhesion and points where there was no adhesion and that the corrosion reactions were initiated at the points where the adhesion had been lost. It was also a question of the temperature because above the glass transition temperature, this model would not be accurate and it would probably be truer to say that the interface consisted of a dynamic equilibrium of points of adhesion, alternately being created and destroyed. In this range, the influence of water might be much more drastic than when the film was below its glass transition temperature.

MR G. W. ROTHWELL said that firstly he would like to confirm from recent first-hand experience that a dark coloured painted surface could quite easily reach a temperature of 40°C or more when exposed in direct sunlight.

Secondly, he pointed out that in practical situations much corrosion took place from the inadequately protected edges of steel components and he asked how the authors had protected their panels during the immersion absorption tests. The efficiency of this protection could have affected the results for water absorption of supported films.

DR HAAGEN explained that care was taken to ensure the edges were painted and in addition tests were carried out with films on non-corroding steel.

PROF. FUNKE added that the samples were dip-coated and checked for adequate thickness at the edges to ensure there was no interference from these. They did not have any interference from the edges in their experiments, although it had to be admitted that extra care had to be taken in this regard when using the electric balance because of the smaller size of the test samples.

MR A. R. H. TAWN (Session Chairman) said that the paper seemed almost a report of two separate relationships: first of all there was the variation of oxygen permeability with temperature; in the second place there was the study of permeability to water vapour. He would have expected some inter-relation between these two. At high relative humidity, for example, the film would be in a swollen state due to its high moisture content and this might be expected to increase its permeability to oxygen.

DR HAAGEN replied that this had been studied and they had been surprised to find that so far as oxygen permeability was concerned, there was little difference between the dry and swollen films. The influence of temperature on oxygen permeability was much stronger than the influence of water.

MR C. E. HOEY returned to an earlier comment by Professor Funke when he remarked that adhesion changed at the glass transition point. Mr Hoey was aware of a change in mechanical properties at the glass transition temperature, which might influence the nature of failure manifested by some adhesion tests, but he was not aware that the forces

of adhesion were radically altered by small changes in temperature around the glass transition point.

PROF. FUNKE said that he considered the adhesion of the film as a whole to be in some way dependant on the individual points of adhesion at the interface. Below the glass transition temperature, these points were in a "steady state", whereas above the glass transition temperature, there was a dynamic situation and it was far easier for agents, such as water, to interfere with the adhesion of the film. As to small changes in temperature around the glass transition temperature, it would be better to speak of the glass transition temperature range over which the more important changes could be expected.

DR OESTERLE wondered whether the authors had made any measurements of the variation of adhesion with time during diffusion of moisture: there should at first be a small increase in adhesion when the water at the interface was only a mono-molecular layer, or at least only a few molecules thick, and then a decrease.

PROF. FUNKE said that Dr Oesterle could only speculate on this increase of adhesion and there was no means whereby the effects could be measured at the present time. It could be said that there was no significant decrease in adhesion during the early stages of water absorption, but it could not be stated with assurance that the adhesion increased. This might be the case since with two glass slides: for example, the adhesion between them could be increased by a small drop of water at the interface, but it could not be stated with certainty that there was a similar effect at the interface between a paint film and its substrate.

Problems encountered in testing paint films*

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Summary

This paper reports on an investigation carried out to determine the relative importance of various atmospheric and application factors in relation to the performance of paint on steel. The experimental technique, the results obtained and the validity of the conclusions drawn are all discussed.

Quelques problèmes éprouvés en faisant des essais sur feuils de peintures

Résumé

Cet exposé est un compte rendu d'une investigation en vue de déterminer l'importance relative de diverses variations dans l'atmosphère ambiante et dans les modes d'application à l'égard du rendement des peintures sur acier. On discute la technique expérimentale ainsi que les résultats obtenus et la validité des conclusions formulées.

Probleme bei der Prüfung von Lackfilmen

Zusammenfassung:

Diese Arbeit berichtet über eine Untersuchung zur Bestimmung der relativen Wichtigkeit von verschiedenen atmosphärischen und anwendungstechnischen Faktoren mit Bezug auf das Verhalten von Anstrichfarben auf Stahl. Die technologischen, experimentellen Methoden, die erhaltenen Resultate und die Gültigkeit der gezogenen Schlüsse werden alle besprochen.

Introduction

The interest in the experiment to be described lies not so much in the influence of certain factors on paint performance since, in qualitative terms at least, these were well known, but rather in two other aspects particularly relevant to the subject of this Conference. These are, on the one hand, the unusual methodology used and, on the other hand, some unexpected and otherwise hidden results that this approach brought to light and that have important implications for paint testing.

The testing of paint films is an activity which has occupied very many man-years since the dawn of the scientific age, and it continues so to do. Investigations are mainly carried out to determine which type of paint performs best under various exposure conditions and, as the number of paint types increases, so too does the number of investigations required to establish their properties. Because of the amount of testing to be carried out, considerable effort has been spent, over the years, in developing laboratory-based rapid test methods to replace the traditional and time-consuming atmospheric exposure method of testing paint films. The salt spray test in its various forms, accelerated weathering apparatuses, and the numerous sulfur dioxide tests are all products of these efforts and, with careful and intelligent use, these techniques can give useful indications of the performance of various systems. The breakdown of the paint films in these tests, however, is brought about by the exaggerated action of a very small number of atmospheric factors (usually one or two), and this makes the interpretation of the results and their application to practical conditions very difficult and in some cases impossible. This is because the breakdown of paint films under practical conditions is the result of a wide range of atmospheric factors and these can interact with one another to give effects quite different from those obtained in a laboratory-based test.

In addition to atmospheric factors during exposure, the performance of a given paint film will be affected by other factors, such as the nature and condition of the substrate, the total thickness of the paint film and the atmospheric conditions during the application and drying of the paint film. It is obvious that each of these factors will have an effect, be it large or small, on the performance of a paint film and this paper describes an investigation carried out to measure the effect of these factors and of interactions between them on two types of paint film and in two different sets of atmospheric conditions. Details of the experimental design and of the results obtained after 6.5 years' exposure are given; both of these are discussed in light of the problems encountered.

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*Paper presented at the Association's Biennial Conference held at the Grand Hotel, Scarborough, Yorks, England, from 17 to 21 June 1975.

Experimental

Conditions investigated

The technique used to investigate the effects and interactions of these factors was a series of experiments in accordance with the traditional "factorial" design. This is a technique where two distinctly different levels of each factor are chosen (for example, good surface preparation and bad surface preparation, good quality paint and bad quality paint). Using these levels, experiments are then carried out with all the possible combinations of the high and low levels. The factors investigated were as follows:

(i)   surface preparation,

(ii) type of paint,

(iii) thickness of paint,

(iv) weather conditions during painting,

(v)   weather conditions during drying,

(vi) weather conditions during exposure.

Surface preparation

The obvious high and low levels of this factor would be shot blasted steel on the one hand and rusted steel on the other. It was felt, however, that to use such vastly different surfaces would mask the influence of other factors and, secondly, it would lead to a very much longer experimental period with the shot blasted steel. For these reasons it was decided to use two different rusted steel surfaces for the main experiments and to introduce additional experiments to measure the effect of shot blasting. Consequently the high (good) level of surface preparation was chosen as a wire-brushed rust film produced during three months' exposure at Leatherhead, Surrey, which is an urban site, and the low (bad) level of surface preparation was chosen as a wire-brushed rust film formed during nine months' exposure at Battersea, London, an industrial site.

Type of paint

The high level of this factor was represented by red lead (type B of BS 2523), which is recognised as suitable for application over rust. The low level was represented by a specially formulated material, red oxide in a varnish medium, which would not normally be considered for use over a rust film. Both of these primers were to be overcoated using the BISRA Grey Finishing Paint No. 404.

Thickness of paint

The high and low levels of this factor were to be achieved by aiming to apply the same thickness of paint per coat and to use a three-coat system for the high level and a two-coat system for the low level.

Weather conditions during painting

After considerable thought, it was decided that the only effective way of achieving two significant levels of this factor at the one time of year was to use conditions indoors at the laboratories as the high level and conditions outdoors on the laboratory roof as the low level.

Weather conditions during drying

Exactly the same conditions were used for this factor as for the previous one.

Weather conditions during exposure

The two sites used to produce the rust films for the "surface preparation" experiment were used to give different atmospheric exposure conditions. Leatherhead, with its lower levels of pollution, was chosen as the "good" site and Battersea, the industrial site, was chosen as the "bad" site.

Analysis of the results of all the experiments in the factorial design allows the effect of the various factors to be quantified. In addition, it allows the effect of interactions between those factors to be quantified, that is, the extent to which the effect of one factor is affected by a variation in the value of others. Table 1 shows the high and low levels of each condition.

Additional factors were investigated outside the main factorial experiment. These were shot blasting of the steel surface and spray application of the paint film.

Experimental design

Table 2 shows all the possible combinations. Each combination has been allotted an experiment number. Two specimens were allocated to each experiment. The "factorial experiment proper" ends at experiment No. 64; experiment Nos. 65 to 72 are those introduced to check, on a limited scale, the effects of shot blasting and of applying the paint by spraying.

Specimen preparation

Seventy-two specimens of the unpainted metal were weathered at Leatherhead for three months and a further 72 at Battersea for nine months. They were removed from the sites on 27 February 1967 and were then wire-brushed and weighed. The painting programme was divided up into four sections, namely:

(a) specimens to be painted indoors and dried indoors,

(b) specimens to be painted indoors and dried outdoors,

(c) specimens to be painted outdoors and dried indoors,

(d) specimens to be painted outdoors and dried outdoors,

Tables 3, 4A and 4B give the dates of painting and weights of paint used for these four sections. Analysis of the rust remaining after wire-brushing was carried out and the results are given in Table 5.

Table 1

Levels of conditions used in the factorial experiment

No.	Condition	Low level	Value used for regression	High level	Value used for regression
1	Surface preparation	Wire-brushed - rust formed at Battersea	0	Wire-brushed - rust formed at Leatherhead	0
2	Type of paint	Red oxide varnish		Red lead (BS25 23) (Type B)	
3	Thickness of paint	2 coats		3 coats	
4	Weather conditions during painting	Outdoors	0	Indoors	1
5	Weather conditions during drying	Outdoors	0	Indoors	1
6	Exposure conditions	Exposed at Battersea	1	Exposed at Leatherhead	0

Table 2

Layout of the factorial experiment

Experiment Number / Conditions	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
Surface Preparation	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B
Quality of Paint	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RO	RO	RO	RO	RO	RO	RO
Thickness (No. of Coats)	2	2	2	2	2	2	2	2	3	3	3	3	3	3	3	3	2	2	2	2	2	2	2	2
Weather at Time of Painting	In	In	In	In	Out	Out	Out	Out	In	In	In	In	Out	Out	Out	Out	In	In	In	In	Out	Out	Out	Out
Weather at Time of Drying	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out
Weather During Exposure	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L
Specimen Nos.	20 21	22 23	101 9	71 108	2 4	109 1	29 30	25 28	44 12	45 13	103 107	15 148	5 6	7 3	33 34	31 32	19 18	17 16	11 10	61 56	70 144	131 74	73 114	46 47

Experiment Number / Conditions	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48
Surface Preparation	B	B	B	B	B	B	B	B	L	L	L	L	L	L	L	L	L	L	L	L	L	L	L	L
Quality of Paint	RO	RO	RO	RO	RO	RO	RO	RO	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL	RL
Thickness (Number of Coats)	3	3	3	3	3	3	3	3	2	2	2	2	2	2	2	2	3	3	3	3	3	3	3	3
Weather at Time of Painting	In	In	In	In	Out	Out	Out	Out	In	In	In	In	Out	Out	Out	Out	In	In	In	In	Out	Out	Out	Out
Weather During Drying	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out
Weather During Exposure	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L
Specimen Numbers	43 42	40 41	65 51	59 62	134 133	118 86	49 8	49 50	336 338	339 337	358 308	354 355	298 305	307 306	369 368	299 310	300 370	289 301	396 397	384 297	321 312	390 373	344 341	294 311

Experiment Number / Conditions	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72
Surface Preparation	L	L	L	L	L	L	L	L	L	L	L	L	L	L	L	L	Sb	Sb	Sb	Sb	B Sp	B Sp	L Sp	L Sp
Quality of Paint	RO	RO	RO	RO	RO	RO	RO	RO	RO	RO	RO	RO	RO	RO	RO	RO	RL	RL	RL	RL	RO	RO	RO	RO
Thickness (Number of Coats)	2	2	2	2	2	2	2	2	3	3	3	3	3	3	3	3	2	3	2	3	3	3	3	3
Weather at Time of Painting	In	In	In	In	Out	Out	Out	Out	In	In	In	In	Out	Out	Out	Out	In	In	In	In	In	In	In	In
Weather During Drying	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out	In	In	Out	Out	In	In	In	In	In	In	In	In
Weather During Exposure	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L	B	L
Specimen Numbers	285 340	377 348	323 296	309 314	379 328	322 343	333 335	290 342	365 362	378 295	291 286	361 360	326 303	304 327	331 325	332 334	453 457	450 452	456 454	451 455	37 38	39 27	387 374	292 288

Key:

B: Exposed at Battersea, wire brushed before painting
RL: Red lead
In: Painted or dried indoors
Sb: Shotblasted

L: Exposed at Leatherhead, wire brushed before painting
RO: Red oxide
Out: Painted or dried outdoors
Sp: Sprayed

F

Table 3

Paint details for "in/in" and "in/out" batches of factorial experiment*

Spec. No.	Experiment No.	Date of Priming	Type of Paint	Wet Wt. of Paint g	Dry Wt. of Paint g	Thickness microns	Date of 1st Top Coat	Wet Wt. of Paint g	Dry Wt. of Paint g	Thickness microns	Date of 2nd Top Coat	Wet Wt. of Paint g	Dry Wt. of Paint g	Thickness microns	Original Weathering Site	Where Painted	Where Dried
20	1		RL	9.6	9.251	102		5.5	4.895	55					B	In	
21	1		RL	8.6	7.850	86		5.3	5.041	55					B	In	
22	2		RL	8.6	8.139	89		5.7	5.236	58					B	In	
23	2		RL	8.8	8.285	91		6.1	5.519	61					B	In	
44	9		RL	9.1	8.659	94		5.1	4.648	51	13-3-67	4.9	4.352	48	B	In	
12	9		RL	8.5	7.971	86		8.1	6.666	75	13-3-67	3.8	3.499	38	B	In	
45	10		RL	8.5	7.879	86		5.0	4.555	51	13-3-67	4.4	3.936	43	B	In	
13	10		RL	9.4	8.923	99		6.8	6.157	70	13-3-67	3.7	3.528	41	B	In	
19	17		RO	1.9	2.068	36		5.6	5.147	58					B	In	
18	17		RO	3.7	2.016	33		4.9	4.575	53					B	In	
17	18		RO	3.0	2.272	38		5.8	5.277	58					B	In	
16	18		RO	3.4	2.609	43		3.8	3.760	43					B	In	
43	25		RO	3.0	2.284	38		4.7	4.431	51	13-3-67	4.2	3.680	41	B	In	
42	25		RO	2.7	1.890	33		4.4	4.178	46	13-3-67	3.1	2.755	30	B	In	
40	26	28-2-67	RO	2.6	1.843	33	6-3-67	5.0	4.568	51	13-3-67	2.7	2.588	28	B	In	In
41	26		RO	2.8	2.171	36		4.4	4.136	46	13-3-67	2.8	2.491	28	B	In	
336	33		RL	7.8	7.768	86		4.9	4.720	53					L.	In	
338	33		RL	6.6	6.100	66		4.8	4.393	48					L.	In	
339	34		RL	7.8	7.150	78		5.5	4.555	51					L.	In	
337	34		RL	7.6	7.041	76		4.0	3.744	43					L.	In	
300	41		RL	7.1	6.634	75		5.0	4.628	51	13-3-67	3.7	3.341	38	L.	In	
370	41		RL	7.6	6.975	76		4.6	4.066	46	13-3-67	4.3	3.729	41	L.	In	
289	42		RL	8.4	7.188	78		5.3	4.549	51	13-3-67	3.9	3.560	41	L.	In	
301	42		RL	8.3	8.122	89		3.7	3.469	38	13-3-67	4.9	4.829	53	L.	In	
285	49		RO	2.6	1.886	33		4.6	4.090	46					L.	In	
340	49		RO	2.1	1.520	25		3.9	3.782	43					L.	In	
377	50		RO	2.5	1.656	27		3.4	3.188	35					L.	In	
348	50		RO	2.6	1.663	28		3.4	3.071	35					L.	In	
365	57		RO	2.9	2.089	36		4.1	3.867	46	13-3-67	3.9	3.505	38	L.	In	
362	57		RO	2.9	2.057	36		5.5	4.512	51	13-3-67	4.4	4.099	46	L.	In	
378	58		RO	2.5	1.939	33		4.9	4.463	51	13-3-67	4.3	4.874	53	L.	In	
295	58		RO	2.1	1.419	23		3.8	3.493	38	13-3-67	3.2	2.968	33	L.	In	
101	3		RL	7.9	7.512	81		5.4	5.007	55					B	In	
9	3		RL	10.1	9.317	102		5.3	4.805	53					B	In	
71	4		RL	9.4	9.096	100		7.9	7.155	78					B	In	
108	4		RL	8.9	8.476	91		5.7	5.254	58					B	In	
103	11		RL	9.7	9.006	100		5.8	5.231	58	14-3-67	3.0	2.892	33	B	In	
107	11		RL	9.9	9.204	102		6.0	5.687	64	14-3-67	4.4	3.985	46	B	In	
15	12		RL	10.3	9.826	107		4.6	4.230	48	14-3-67	3.5	3.394	38	B	In	
148	12		RL	10.5	9.690	107		4.1	4.091	46	14-3-67	3.6	3.381	38	B	In	
11	19		RO	2.9	2.264	38		4.6	4.207	48					B	In	
10	19		RO	3.1	2.305	38		4.2	4.144	46					B	In	
61	20		RO	3.9	2.908	48		4.5	4.075	46					B	In	
56	20		RO	2.7	2.156	36		4.6	4.369	48					B	In	
65	27		RO	3.5	2.657	46		5.0	4.521	51	14-3-67	3.9	3.422	38	B	In	
51	27		RO	3.1	2.467	41		3.6	3.591	41	14-3-67	3.4	3.028	33	B	In	
59	28	2-3-67	RO	2.9	2.016	33	8-3-67	4.2	4.173	46	14-3-67	3.6	3.354	38	B	In	Out
62	28		RO	3.1	2.400	41		4.5	4.158	46	14-3-67	3.8	3.461	38	B	In	
358	35		RL	6.6	6.110	66		5.3	4.961	55					L.	In	
308	35		RL	7.4	6.992	76		5.8	5.511	61					L.	In	
354	36		RL	5.8	5.547	61		5.6	5.164	58					L.	In	
355	36		RL	6.7	6.318	68		5.1	4.681	53					L.	In	
396	43		RL	8.9	8.062	89		4.9	4.456	51	14-3-67	4.8	4.287	48	L.	In	
397	43		RL	7.6	7.152	78		5.1	4.779	53	14-3-67	4.1	3.825	43	L.	In	
384	44		RL	7.6	7.148	78		5.6	5.174	58	14-3-67	3.9	3.732	41	L.	In	
297	44		RL	7.2	6.707	75		4.2	3.755	43	14-3-67	3.5	3.287	35	L.	In	
323	51		RO	2.4	2.158	36		4.6	4.092	46					L.	In	
296	51		RO	2.6	1.801	30		3.6	3.601	41					L.	In	
309	52		RO	3.3	2.232	38		4.0	3.725	41					L.	In	
314	52		RO	2.9	2.261	38		4.5	4.064	46					L.	In	
291	59		RO	2.3	2.015	33		4.6	4.157	46	14-3-67	5.7	4.915	55	L.	In	
286	59		RO	2.3	1.710	28		3.8	3.535	41	14-3-67	3.4	3.137	35	L.	In	
361	60		RO	2.3	1.745	30		4.1	3.823	43	14-3-67	3.9	3.458	38	L.	In	
360	60		RO	2.9	2.152	36		3.7	3.600	41	14-3-67	3.3	3.167	35	L.	In	

*Symbols as in Table 2   1 mil $= 25.4\mu$m

Table 4A

Paint details for "out/in" and "out/out" batches of factorial experiment*

Spec. No.	Experiment No.	Date of Priming	Type of Paint	Wet Wt. of Paint g	Dry Wt. of Paint g	Thickness microns	Date of 1st Top Coat	Wet Wt. of Paint g	Dry Wt. of Paint g	Thickness microns	Date of 2nd Top Coat	Wet Wt. of Paint g	Dry Wt. of Paint g	Thickness microns	Original Weathering Site	Where Painted	Where Dried
2	5		RL	12.8	11.875	130		4.3	3.954	43					B	Out	
4	5		RL	11.0	10.027	110		6.2	5.699	64					B	Out	
109	6		RL	10.5	9.618	104		8.0	6.161	68					D	Out	
1	6		RL	12.7	12.019	132		5.9	5.315	58					B	Out	
5	13		RL	10.3	9.630	104		6.2	5.748	64	14-3-67	4.4	4.061	46	B	Out	
6	13		RL	10.3	8.684	81		6.8	6.113	68	14-3-67	4.6	4.068	46	B	Out	
7	14		RL	11.0	11.006	122		6.5	6.166	68	14-3-67	5.0	4.668	51	B	Out	
3	14		RL	13.0	11.997	132		6.3	5.729	64	14-3-67	3.9	3.377	38	B	Out	
70	21		RO	4.1	2.956	51		6.2	5.820	66					B	Out	
144	21		RO	3.8	2.818	48		4.8	4.417	48					B	Out	
131	22		RO	2.9	2.350	41		4.8	4.525	51					B	Out	
74	22		RO	3.9	2.782	48		5.8	5.325	58					B	Out	
134	29		RO	3.2	2.450	41		5.0	5.548	64	14-3-67	4.1	3.799	43	B	Out	
133	29		RO	4.2	3.501	58		4.4	3.432	38	14-3-67	4.1	3.616	41	B	Out	
118	30		RO	3.0	2.400	41		4.4	4.339	48	14-3-67	3.7	3.262	36	B	Out	
86	30	2-3-67	RO	2.9	2.297	38	8-3-67	4.9	4.335	48	14-3-67	3.8	3.244	36	B	Out	In
298	37		RL	6.8	6.398	71		5.6	5.152	58					L.	Out	
305	37		RL	9.9	9.267	102		5.0	4.413	48					L.	Out	
307	38		RL	7.3	6.974	76		6.0	5.702	64					L.	Out	
306	38		RL	6.7	6.426	71		5.2	4.886	53					L.	Out	
321	45		RL	9.3	8.216	89		4.1	3.732	43	14-3-67	4.5	4.175	46	L.	Out	
312	45		RL	6.7	6.223	69		4.3	4.032	46	14-3-67	5.4	4.936	53	L.	Out	
390	46		RL	8.8	7.893	86		4.7	4.407	48	14-3-67	3.5	3.044	33	L.	Out	
373	46		RL	7.0	6.526	71		3.6	3.479	38	14-3-67	4.2	3.802	43	L.	Out	
379	53		RO	2.8	1.960	33		6.1	4.603	51					L.	Out	
328	53		RO	2.7	2.026	33		6.2	5.804	64					L.	Out	
322	54		RO	2.8	1.975	33		5.4	5.066	55					L.	Out	
343	54		RO	2.3	1.942	33		4.9	4.489	51					L.	Out	
326	61		RO	2.9	1.993	33		5.4	5.149	55	14-3-67	3.5	3.304	38	L.	Out	
303	61		RO	2.3	2.032	35		4.5	4.197	48	14-3-67	3.0	2.710	30	L.	Out	
304	62		RO	3.5	2.730	46		5.0	4.699	53	14-3-67	3.9	3.387	38	L.	Out	
327	62		RO	3.6	2.408	41		7.1	6.604	75	14-3-67	4.2	3.900	43	L.	Out	
29	7		RL	10.0	9.015	99		6.2	5.945	66					B	Out	
30	7		RL	10.2	9.502	104		5.7	5.359	61					B	Out	
25	8		RL	8.6	8.044	89		6.1	5.673	64					B	Out	
28	8		RL	10.0	8.951	99		4.9	4.640	51					B	Out	
33	15		RL	11.6	10.579	114		5.2	4.974	55	14-3-67	4.7	4.339	48	B	Out	
34	15		RL	9.9	9.204	102		6.3	5.765	64	14-3-67	5.6	4.936	55	B	Out	
31	16		RL	10.6	9.461	104		6.3	6.018	68	14-3-67	5.9	5.246	58	B	Out	
32	16		RL	8.2	7.790	86		7.4	6.899	76	14-3-67	4.3	3.944	43	B	Out	
73	23		RO	4.3	3.038	51		5.3	5.115	58					B	Out	
114	23		RO	4.1	2.860	48		6.0	5.621	64					B	Out	
46	24		RO	4.0	2.966	51		7.0	6.521	75					B	Out	
47	24		RO	3.5	2.919	48		4.1	3.811	43					B	Out	
48	31		RO	3.9	2.908	48		6.1	5.815	64	14-3-67	4.2	3.741	43	B	Out	
8	31		RO	4.7	3.070	51		6.5	6.824	76	14-3-67	3.9	3.609	41	B	Out	
49	32		RO	4.0	3.347	55		6.0	5.275	58	14-3-67	4.0	3.615	41	B	Out	
50	32		RO	4.0	3.180	53		5.9	5.248	58	14-3-67	4.0	3.766	43	B	Out	
369	39	1-3-67	RL	6.7	6.457	71	7-3-67	6.3	5.708	64					L.	Out	Out
368	39		RL	8.1	7.300	81		5.2	4.863	53					L.	Out	
299	40		RL	7.6	7.199	79		6.5	5.974	66					L.	Out	
310	40		RL	10.3	9.659	107		4.4	4.216	48					L.	Out	
344	47		RL	7.4	6.977	76		6.4	6.128	68	14-3-67	4.2	3.971	43	L.	Out	
341	47		RL	6.8	6.173	69		6.6	6.438	71	14-3-67	4.1	3.735	41	L.	Out	
294	48		RL	8.0	7.373	81		6.7	6.149	68	14-3-67	4.8	4.412	51	L.	Out	
311	48		RL	6.2	5.568	61		5.7	5.162	58	14-3-67	5.2	4.911	55	L.	Out	
333	55		RO	3.2	2.345	41		6.4	5.715	64					L.	Out	
335	55		RO	3.3	2.541	43		6.0	5.291	58					L.	Out	
290	56		RO	3.6	2.647	46		5.9	5.546	64					L.	Out	
342	56		RO	3.5	2.958	51		5.9	5.477	61					L.	Out	
331	63		RO	3.8	2.936	51		5.3	4.967	55	14-3-67	4.2	4.175	46	L.	Out	
325	63		RO	4.3	3.098	51		6.0	5.206	58	14-3-67	4.0	3.636	41	L.	Out	
332	64		RO	2.7	2.099	33		4.9	4.800	53	14-3-67	4.2	3.558	41	L.	Out	
334	64		RO	3.6	2.333	41		5.3	4.844	53	14-3-67	3.7	3.394	38	L.	Out	

*Symbols as in Table 2   1 mil = 25.4μm

Table 4B

*Additional experiments to check the effects of (1) shotblasting and (2) spraying**

Spec. No.	Experiment No.	Date of Priming	Type of Paint	Wet Wt. of Paint g	Dry Wt. of Paint g	Thick-ness microns	Date of 1st top coat	Wet Wt.of Paint g	Dry Wt. of Paint g	Thick-ness microns	Date of 2nd top coat	Wet Wt. of Paint g	Dry Wt. of Paint g	Thick-ness microns	Surface Condi-tion	Method of Paint Appli-cation	Where Painted	Where Dried
457	65	10-3-67	RL	5.8	5.627	61	14-3-67	4.7	4.040	46					Sb	Br	In	In
453	65	10-3-67	RL	8.2	7.645	84	14-3-67	4.7	4.004	46					Sb	Br	In	In
452	66	10-3-67	RL	5.7	5.518	61	14-3-67	4.0	3.483	38	15-3-67	4.7	4.030	46	Sb	Br	In	In
450	66	10-3-67	RL	6.4	5.365	58	14-3-67	3.4	3.261	35	15-3-67	3.6	3.213	35	Sb	Br	In	In
454	67	10-3-67	RL	5.8	5.454	61	14-3-67	4.7	4.115	46					Sb	Br	In	In
456	67	10-3-67	RL	6.8	6.477	71	14-3-67	4.4	4.033	46					Sb	Br	In	In
455	68	10-3-67	RL	7.4	6.850	76	14-3-67	4.3	4.009	46	15-3-67	4.4	4.153	46	Sb	Br	In	In
451	68	10-3-67	RL	8.2	7.876	86	14-3-67	4.8	4.746	53	15-3-67	4.1	3.894	43	Sb	Br	In	In
38	69	9-3-67	RO		2.254	38	13-3-67		3.508	38	16-3-67		2.481	28	R	Sp	In	In
37	69	9-3-67	RO		1.835	30	13-3-67		4.045	46	16-3-67		2.133	23	R	Sp	In	In
27	70	9-3-67	RO		1.998	33	13-3-67		3.438	38	16-3-67		2.106	23	R	Sp	In	In
39	70	9-3-67	RO		2.136	36	13-3-67		3.422	38	16-3-67		1.873	21	R	Sp	In	In
374	71	9-3-67	RO		1.930	33	13-3-67		3.153	35	16-3-67		1.789	20	R	Sp	In	In
387	71	9-3-67	RO				13-3-67		6.373x		16-3-67		1.605	19	R	Sp	In	In
292	72	9-3-67	RO		2.427	41	13-3-67		4.040	46	16-3-67		1.472	16	R	Sp	In	In
288	72	9-3-67	RO		0.979	19	13-3-67		5.269	58	16-3-67		2.069	23	R	Sp	In	In

Two coats: 1 primer and 1 top coat　　　*Symbols as in Table 2 with addition of: R = rusted; Br = brushed

Table 5

Details of rust analysis

Spec. No.	Exposure Site	Wt. of Adherent Rust, g/m^2	Wt. of SO_2 in Adherent Rust, g/m^2	SO_4 in Adherent Rust, %	Wt. of Cl in Adherent Rust, g/m^2	Cl in Adherent Rust %
315*	Leather-head	133.5			0.384	0.274
329*	Leather-head	126.5			0.690	0.52
119*	Battersea	360.0			0.384	0.107
14*	Battersea	347.0			0.230	0.066
367	Leather-head	132.2	0.52	0.39		
67	Battersea	311.0	2.21	0.71		

*The results of the sulfate analyses on specimens 315, 329, 119 and 14 were so low that it was assumed that there was some error in this part of the analysis

Table 6

Air pollution and weather conditions during painting

	INDOORS															OUTDOORS											
Date	Daily Pollution Smoke µ g/m³/day	SO₂ µ g/m³ day	Time of reading hrs. GMT	RH %	Temp °C	Wind	Time of reading hrs. GMT	RH %	Temp °C	Wind	Time of reading hrs. GMT	RH %	Temp °C	Wind	Daily Pollution Smoke µ g/m³ day	SO₂ µ g/m³ day	Time of reading hrs. GMT	RH %	Temp °C	Wind	Time of reading hrs. GMT	RH %	Temp °C	Wind	Time of reading hrs. GMT	RH %	Temp °C	Wind
28.2.67	56	132	15.00	50	20	-	16.30	55	20	-																		
1.3.67															72	314	11.15	72	9.5	Light	12.15	60	10	Light				
2.3.67	88	167	15.00	45	20	-	16.00	45	20	-	16.45	45	20	-	96	339	10.00	60	6	Strong	11.15	53	10	Strong	12.00	52	10	Strong
6.3.67	88	89	14.30	56	21	-	16.15	56	21	-																		
7.3.67															46	118	14.30	74	8.5	Moder-ate	15.30	74	8	Moder-ate	16.15	75	8	Moder-ate
8.3.67	102	92	14.30	50	21	-	15.15	50	20.5	-					46	120	10.00	77	8	Very Light	11.15	63	9	Very Light				
10.3.67	96	110	16.30	50	18	-																						
13.3.67	68	124	15.30	53	21	-																						
14.3.67	94	106	16.15	48	22	-									78	209	11.00	60	12.5	Very Light								

Weather conditions

During painting and drying, the temperature, relative humidity, and sulfur dioxide and smoke concentrations were measured in the laboratory and on the laboratory roof, where the painting was carried out.

Table 6 shows how these conditions varied from the beginning to the end of each painting period. Table 7 shows how the conditions varied during the drying period. The red lead (type B) paint required one or two days to dry and the red oxide paint dried in about 12 hours.

Table 7

Air pollution and weather conditions during drying

| | INDOORS | | | | OUTDOORS | | | | | | | | | | |
Date	Smoke μg/m³/day	SO₂ μg/m³ day	Average Temp °C	Average R.H.%	Smoke μg/m³/day	SO₂ μg/m³/day	Time of readings hrs. GMT	Temp. °C	R.H.%	Time of readings hrs. GMT	Temp. °C	R.H.%	Time of readings hrs. GMT	Temp. °C	R.H.%
28-2-67	56	132	16	55	58	245	03.00	9	96	13.00	10	50	23.00	5	76
1-3-67	70	178	16	55	72	314	04.00	4	84	13.00	10	56	23.00	6	78
2-3-67	88	167	16	60	96	339	06.00	6	83	13.00	12	50	23.00	6	80
3-3-67	64	213	17	55	96	405	04.00	6	90	13.00	11	61	23.00	8	64
4-3-67	-	-	16	55	-	-	06.00	7	66	13.00	11	60	23.00	7	77
5-3-67	-	-	16	55	-	-	06.00	7	90	14.00	12	76	23.00	8	78
6-3-67	-	-	16	55	-	-	06.00	7	92	12.00	13	78	23.00	8	88
7-3-67	88	89	17	58	46	118	06.00	6	92	14.00	8	70	23.00	7	90
8-3-67	102	92	17	58	46	120	06.00	6	92	13.00	10	53	23.00	6	92
9-3-67	66	106	17	60	80	146	06.00	9	92	13.00	9	72	23.00	6	72
10-3-67	86	110	15	60	58	186	07.00	6	85	13.00	11	47	23.00	5	84
11-3-67	-	-	12	65	-	-	06.00	4	83	16.00	7	46	23.00	3	72
12-3-67	-	-	12	63	-	-	06.00	2	76	13.00	8	44	23.00	3	84
13-3-67	-	-	15	60	-	-	06.00	3	84	17.00	8	54	23.00	5	66
14-3-67	68	124	17	56	78	209	06.00	7	88	14.00	14	50	23.00	7	85
15-3-67	94	106	17	53	68	186	06.00	7	84	15.00	13	42	23.00	6	61
16-3-67	58	183	17	50	65	152	06.00	4	84	13.00	12	50	23.00	7	72
17-3-67	52	110	15	56	56	114	04.00	5	84	13.00	10	72	23.00	9	68
18-3-67	-	-	15	64	-	-	07.00	7	81	13.00	12	50	23.00	6	74
19-3-67	-	-	16	64	-	-	06.00	7	83	14.00	13	44	23.00	7	62
20-3-67	-	-	17	60	-	-	06.00	5	78	12.00	13	42	-	-	-

Table 8

Condition of specimens after 6.5 years' atmospheric exposure

Spec. No	Experiment No	% Breakdown FRONT	BACK	Spec. No	Experiment No	% Breakdown FRONT	BACK
20	1	E. T/C	-	18	17	10R	15R
21		E. T/C	-	19		5R	10R
22	2	-	-	16	18	10R	5R
23		-	-	17		60R	5R
101	3	E. T/C	-	10	19	7R	15R
9		E. T/C	-	11		7R	15R
71	4	-	0.05R	56	20	75R	20R
108			-	61		25R	20R
2	5	E. T/C	-	70	21	-	2R
4		E. T/C	-	144		2R	2R
109	6	30 Bl	-	131	22	40R	15R
1		-	-	74		10R	5R
29	7	E. T/C	-	73	23	0.1R	10R
30		E. T/C	-	114		7R	1R
25	8	-	-	46	24	2R	1R
28			-	47		15R	1R
44	9	-	-	42	25	-	-
12		-	-	43		-	0.5R
45	10	-	-	40	26	7R	0.2R
13		-	-	41		0.5R	0.2R
103	11	-	-	51	27	-	5R
107		-	-	65		-	0.5R
15	12	-	-	59	28	1R	0.5R 30 Bl
148		10 Bl	-	62		1.5R	0.2R
5	13	-	-	133	29	0.05R	2R
6		-	-	134		0.05R	5R
7	14	-	-	86	30	1R	0.2R
3		-	-	118		5R	0.2R
33	15	-	-	8	31	-	0.05R
34		-	-	48		0.5R	0.2R
31	16	-	-	49	32	4R	5R 30 Bl
32		-	-	50		2R	0.2R

Table 8—continued

Spec. No	Experiment No	% Breakdown FRONT	BACK	Spec. No	Experiment No	% Breakdown FRONT	BACK
336	33	E. T/C	-	285	49	15R	25R
338		E. T/C	-	340		25R	20R
339	34	0.05R	-	377	50	100R	50R
337		2R	-	348		90R	40R
358	35	E. T/C	-	323	51	10R	5R
308		E. T/C	-	296		20R	25R
354	36	0.01R	-	309	52	50R	2R
355		-	-	314		17R	5R
298	37	E. T/C	-	379	53	2R	5R
305		E. T/C	-	328		20R	0.01R
307	38	0.01R	0.05R at edge	322	54	75R	1R at edge
306		0.01R	-	343		80R	1R at edge
369	39	E. T/C	-	333	55	4R	0.05R
368		E. T/C	-	335		0.25R	5R
299	40	0.01R	0.5R at edge	290	56	25R	2R
310		0.01R	0.5R at edge	342		25R	1R at edge
300	41	-	-	365	57	0.05R	0.5R
370		-	-	362		-	10R
289	42	-	-	378	58	30R	1R at edge
301		-	-	295		40R	1R at edge
396	43	-	-	291	59	-	-
397		-	-	286		0.15R	-
384	44	-	-	361	60	1.5	-
297		0.01R	0.1R at edge	360		50R	2R at edge
321	45	-	-	326	61	0.05R	-
312		-	-	303		5R	-
390	46	-	-	304	62	40R	2R at edge
373		-	-	327		0.25R	2R at edge
344	47	-	-	331	63	-	-
341		-	-	325		4R	-
294	48	-	-	332	64	0.05R	1R at edge
311		-	-	334		30R	1R at edge
			ADDITIONAL EXPERIMENTS				
453	65	E. T/C	-	37	69	1R	1R
457		E. T/C	-	38		0.1R	-
450	66	-	-	39	70	10R	2R
452		-	-	27		5R	2R
456	67	0.05R in Brush marks	-	387	71	5R	1R at edge
454		0.05R in Brush marks	-	374		15R	2R at edge
451	68	-	-	292	72	1R	-
455		-	-	288		2R	-

R = rust;   Bl = blisters;   E. T/C = erosion of top cast

Exposure

The painted specimens were exposed at 45° to the vertical facing south at Leatherhead on 21 March and similarly at Battersea on 22 March 1967.

Calculations of paint thickness

Paint constants were not available for all the paints used and it was necessary to determine the dry film thickness of the coatings from the weight of paint applied. The method used

is shown in Part 1 of the Appendix and it was checked as shown in Part 2.

Results

During the design stage of the investigation, it was felt that the best way to achieve a quantitative measure of the performance of the system used in each experiment was to record the number of days' exposure required to produce a certain degree of breakdown, for example, 1.0 per cent rust or 1.0 per cent blisters. In the event, however, it did not prove possible to maintain this approach, mainly because of the very slow rate of breakdown exhibited by a large number of the specimens. Because of this, it became impractical to maintain the frequency of inspection necessary to collect the detailed information required. As was mentioned earlier, it was also intended to use blistering of the paint film as a measure of breakdown but this again proved impossible. This was a result of the type of surface preparation selected for use in the investigation.

The specimens exposed at the Battersea site for nine months developed much coarser rust films than those exposed at Leatherhead and, after wire-brushing and painting, the appearance of the Battersea specimens was similar to that of a "blistered" paint film. It was possible in the early stages to make allowances for this but, as time went on, it became impossible to make a reliable assessment of the percentage of the specimen surface area affected by real blistering.

For these reasons, only periodic examinations of the specimens were made on site and in July 1973, after approximately 6.5 years' exposure, it was decided to remove all the specimens to the laboratory for a detailed examination. Table 8 gives the results of this examination. These results were used for statistical analysis using a regression technique designed to cope with discontinuous variables.

In advance of the statistical analysis, the following general conclusions were drawn on the basis of visual observations:

Type of paint

The red oxide paint was very definitely inferior to the red lead paint under all circumstances. Some of the three-coat red oxide systems exposed at Battersea, however, were in a surprisingly good condition.

Surface preparation

Systems applied over Leatherhead rust were generally worse than systems applied over Battersea rust. This was a surprising result and was thought to be partly due to the fact that there was a tendency for the Battersea rust to pick up more paint than the Leatherhead rust. This was subsequently borne out by the statistical analysis which showed a fair degree of correlation (correlation coefficient = 0.605) between the type of rust and the thickness of the primer coat. Because of the almost total lack of breakdown on the specimens painted with red lead, the effect of shot blasting the steel prior to painting cannot be readily observed, except on the underside where the shot-blasted specimens are superior.

Thickness of paint film

In all cases the three-coat system performed better than the two-coat system on the same primer, but the two-coat red lead system performed better than the three-coat red oxide system.

Weather conditions during painting

There is some evidence that painting indoors was detrimental, but this again can almost certainly be explained by the different paint film thicknesses achieved indoors compared with those outdoors. The correlation between paint film thickness and place of painting was found to be high (correlation coefficient = 0.715).

Weather conditions during drying

This did not appear to have any effect, except possibly with the two-coat red oxide system on Leatherhead rust exposed at Leatherhead, where drying indoors seemed to be slightly detrimental.

Site of exposure

Where there was any sign of breakdown, the Leatherhead site was found to be the more aggresive. The exceptions to this were with erosion of the top coat, which was quite severe on some specimens at Battersea, and on the undersides of the specimens, which showed slightly more breakdown at Battersea than at Leatherhead.

Method of application of the paint film

It is difficult to make a significant comparison because of the small number of specimens, but it would appear that the main effect of the spray application of paint films has been to reduce the paint film thickness and consequently reduce the paint performance.

Statistical analysis of results

As already mentioned, the degree of breakdown on specimens coated with red lead primer was very slight and, therefore, the entire set of red lead specimens was omitted from the statistical analysis. Regression analysis was carried out on the results from the 32 experiments in the factorial set which involved the red oxide primer. The regression was carried out using a technique capable of assessing the effect of discontinuous independent variables. The calculations were carried out on an ICL 1904 computer and the results are given in Table 9. This method gave the following relationship between the percentage of paint breakdown and the six independent variables set out in Table 1:

$$\text{Percentage breakdown} = (0.87 - 0.09R - 0.005T - 0.03P + 0.08D - 0.29S) \times 100$$

where R = type of rust over which the paint applied. Condition No. 1 (see Table 1).

T = total thickness of paint applied (μm), Condition No. 3

P = place of painting, Condition No. 4

D = place of drying, Condition No. 5

S = site of exposure, Condition No. 6

The value given to the two levels of the discontinuous variables, D, R, P and S are given in Table 1.

Table 9
Results of statistical analysis

Variable	Coefficient	Standard error of Coefficient
Rust	−0. 09006	0. 03117
Paint	−0. 02639	0. 03467
Dry	0. 08357	0. 03343
Site	−0. 28726	0. 03527
Thick	−0. 00514	0. 00077
Intercept =		0. 87318
Standard Error =		0. 01440
Coefficient of Determination =		0. 64925
Error Degrees of Freedom =		58. 00000

Key: Rust = site of pre-rusting
Paint = place of painting
Dry = place of drying
Site = site of exposure
Thick = total paint film thickness (μm)

The correlation coefficient for the regression was 0.81, showing that about 65 per cent of the variation is accounted for by this equation. By substituting the appropriate values in the above equation the following conclusions can be drawn. Paint film breakdown is decreased by:

(i) applying the paint over a Battersea rust rather than a Leatherhead rust,

(ii) applying the paint indoors,

(iii) allowing the paint to dry outdoors,

(iv) exposing the paint film at Battersea rather than Leatherhead,

(v) increasing the amount of paint applied.

The conclusions agree quite well with the earlier conclusions based on visual observations. The statistical analysis, however, does throw some additional light on two important effects, (i) the effect of the rust film and (ii) the effect of painting indoors. The statistical results demonstrate that the Leatherhead rust has a detrimental effect independent of the paint film thickness and that, at equal paint film thicknesses, applying the paint film indoors as opposed to outdoors is beneficial.

Discussion

Three of the five effects so far observed in this investigation are not difficult to understand. Application of the paint film indoors will reduce the contamination of the paint by atmospheric pollution and airborne dusts, paint films allowed to dry outdoors, sheltered from direct rain, will dry more slowly which will usually result in a more stable film and, clearly, thicker paint films will provide more protection than thinner ones.

The other two effects, however, that of the rust film and that of the site of exposure, are more difficult to understand. With both effects, contrary to expectations, Leatherhead has proved to be more aggresive than Battersea.

It can be seen from Table 5 that, chemically, the Leatherhead rust provided less of a problem than the Battersea rust. It is possible, however, that the morphologies of the rusts were such that the adhesion of the paint to the Battersea rust was sufficiently strong, by comparison with the Leatherhead rust, to overcome the adverse effect of rust composition. Further investigation will be required to check this possibility and it will, of course, be interesting to see if the same effect manifests itself with the red lead set.

The adverse effect of exposure at Leatherhead compared with Battersea is completely against expectation. This is the first investigation of paint performance carried out at Leatherhead and it had been assumed, because of its lower levels of atmospheric pollution, that it would be a less aggressive site than Battersea. This is clearly not the case and, looking beyond atmospheric pollution for the cause, the most likely candidate seems to be the humidity. The site is within 50 metres of the River Mole and is surrounded on three sides by trees, which provide substantial shelter, whereas the site at Battersea, being on the roof of a four-storey building, is completely unsheltered. This explanation fits in with the results for the undersides, which were sheltered. Those of the Battersea specimens have deteriorated slightly more than the undersides of the Leatherhead specimens.

Further investigation of the effects of humidity and the time during which the paint is in contact with water (time of wetness), and their relationship with sulfur dioxide, on paint performance is required. These are clearly very important factors when considering paint performance on the undersides of bridges.

There is one final observation which has emerged during this investigation which must be commented upon and that is the degree of protection provided by what would be described in commercial terms as "inferior systems", particularly bearing in mind that all these films were applied over rust. Specimens such as specimen No 70, with as little as 117μm of an inferior paint applied over an "industrial" rust film and exposed in an industrial atmosphere for 6.5 years, are still in almost perfect condition. The specimens painted with red lead, which are showing virtually no breakdown at either site, are again many times better than would normally be expected. These results must raise fundamental questions about the test methods being used and the applicability to large structures of results obtained with small specimens. Further investigations must be carried out to establish the relationship between large-and small-scale tests.

This investigation was very carefully designed to give a quantitative measure of the effect of certain factors on paint performance and to determine how these factors interacted with the type of paint films used. For a number of reasons this has not yet proved possible. The main reason for this is the incredibly good performance given by systems which, in normal commercial use, would almost certainly have failed after one, or at most, two years. This has not only made the investigation a much more time-consuming one but, more importantly, it raises the question of the validity of any observations which might be made about other factors. There must obviously be a reason for this difference in performance and there is need for further investigations in this area. For example, it must be established whether or not this difference

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- apparatus construction

Technical Data:

(on pre-treated aluminium)
Gloss (Gardner)	approx. 95—100%
Pencil hardness	H
Impact test	approx. 80 inch · lbs
T-bend-test	T 0-2 depending on the type
Salt spray test	min. 400 hours, unaffected

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is due to specimen size. It is quite likely that this factor will interact with atmospheric conditions (for example, temperature/humidity) to produce quite different micro-climates depending upon the "bulk" of the steel substrate.

Final conclusions and recommendations

This investigation has shown that:

(a) Paint performance on shot-blasted steel, as opposed to rusted steel, is better, although longer exposure periods will be required fully to evaluate this effect.

(b) Factors other than the chemical compositions of the rust must be taken into account, for example the profile of the rust film. Also, thicker rust films pick up thicker paint films during brush application.

(c) The red lead is much superior to the red oxide paint, giving almost total protection to pre-rusted steel for over 6.5 years of exposure. Even the red oxide paint gives a high degree of protection to pre-rusted steel under certain circumstances.

(d) Paint performance is improved if the paint is applied indoors, provided care is taken to build an adequate paint film thickness.

(e) Paint performance is improved by allowing the paint to dry outdoors, sheltered from direct rain.

(f) Paint performance is improved by applying thicker paint films, Consequently, paint which is thinned and spray applied, may not perform so well as brush-applied paint.

(g) It is likely that damp sites can be more aggressive towards paint films, despite having lower levels of industrial pollution.

It is recommended that further work be carried out to investigate (i) the relationship between results obtained with small test pieces and either large test pieces or permanent structures, (ii) the relationship between humidity, time of wetness, atmospheric pollution levels and paint performance with a wide range of paints and (iii) the difference between various rust films in terms of the physical properties, which may make them suitable or otherwise for paint application.

[Received 6 March 1975

Appendix

Determination of paint film thickness

Part 1

Method of determination of paint film thickness from dry density

The aim is to determine the volume of dry paint film given by a known weight of paint. From this the thickness of the dry paint can be calculated, using the formula:

$$T = V/A$$

Where A = Surface area in cm²

V = Volume of dry paint film in cm³

T = Thickness of dry paint film in cm

For the determination, a steel specimen 5cm square is weighed first in air and then in water. The specimen is then painted and allowed to dry.

It is then reweighed in air and in water.

Let x = weight of specimen in air (g)

y = weight of specimen in water (g)

x^1 = weight of specimen and paint in air (g)

y^1 = weight of specimen and paint in water (g)

Volume of specimen = $(x - y)$ cm³

Volume of specimen and paint = $(x^1 - y^1)$ cm³

∴ Volume of paint = $(x^1 - y^1) - (x - y)$ cm³

From this $(x^1 - x)$ g of paint on 25cm² gives a dry film thickness,

$$T = \frac{(x^1 - y^1) - (x - y)}{25} \text{ cm}$$

∴ $\left(\frac{(x^1 - x)}{25} \times 315 \right)$ g of paint on a $15 \times 10 \times 0.3$cm specimen

will also give

$$T = \frac{(x^1 - y^1) - (x - y)}{25} \text{ cm}$$

∴ 1g of paint (dry weight) on a $15 \times 10 \times 0.3$cm specimen will give

$$T = \frac{(x^1 - y^1) - (x - y)}{315 (x^1 - x)}$$

The paint film thickness on a $15 \times 10 \times 0.3$cm specimen can now be obtained by multiplying the dry weight of paint applied by

$$\frac{(x^1 - y^1) - (x - y)}{315 (x^1 - x)}$$

The figures obtained for the three paints used were as follows:

Red lead, type B: 1g per $15 \times 10 \times 0.3$cm specimen gives a thickness of 10.8μm

Red oxide: 1g per $15 \times 10 \times 0.3$cm specimen gives 16.7μm

Grey finishing paint: 1g per $15 \times 10 \times 0.3$cm specimen gives 11μm

Part 2

Method of determination of paint film thickness from wet paint constants

The thickness given by 1g of grey finishing paint on a $15 \times 10 \times 0.3$cm specimen was also calculated using the formula:

$$T = \frac{W}{A} \left(\frac{1}{G} - \frac{V}{100g} \right)$$

Where T = Thickness in cm

W = dry weight of paint, in g

A = Surface area of specimen, in cm²

G = Specific gravity of the paint

V = Per cent volatile content of the paint

g = Specific gravity of the thinners

For this calculation $W = 1$g, $A = 325$ cm², $G = 2.39$, $V = 5.5$ and $g = 0.78$

From this formula it was calculated that 1g of grey finishing paint on a $15 \times 10 \times 0.3$cm specimen gives a thickness of 10.5μm. This experimental figure is in sufficiently close agreement with the one obtained by the method based on dry densities to warrant the use of the experimental method when the paint constants are not known.

Discussion at Scarborough Conference

MR P. WHITELEY asked whether the panels were hot- or cold-rolled steel. In smaller and not statistically controlled experiments at the Building Research Station at Garston many years ago, fairly clean cold-rolled steel panels had produced no corrosion under various primers after more than ten years, whereas the same paints had shown failures of the normal time scale on rusty hot-rolled steel.

MR KILCULLEN said that these results were very interesting, but he wondered whether the size of the panel was the significant factor, rather than whether the steel was hot or cold rolled. The larger panels would be more representative of a practical structure, for example a bridge, in terms of such factors as the temperature and relative humidity cycles which affected the surfaces.

MR WHITELEY thought that this was certainly an important factor. Small panels hanging in free circulating air and getting plenty of sunshine were a lot drier than usually supposed.

MR KILCULLEN said that work he had done on temperature cycles indicated that the temperature changes in small test panels very closely followed atmospheric conditions, whereas this was certainly not the case with a large steel girder.

MR WHITELEY added that there tended to be a lot more rust on bridge structures than on test panels and this was another factor to be considered.

MR S. L. DAVIDSON commented that he could easily believe the long lives of test coatings reported in this paper. In New Jersey, they had a test panel with four coats on it which had been exposed at 45° facing south for 18 years and still showed no sign of failure.

MR K. JULYAN DAY said that the figures given for percentage breakdown on page 373 seemed to indicate that the site of exposure was the factor of over-riding importance.

MR KILCULLEN agreed that at first sight this was the most important term in his equation, but it had to be remembered that the term S in the equation representing the site of exposure took a value of either zero or one; this was also true of the other terms except for T the thickness of the film which varied between a minimum of approximately 80 and a maximum of about 150. As a result, by far the most significant term in the equation was the thickness of the film. He agreed that there was nothing new in this: it only went to prove the validity of the equation.

DR M. L. ELLINGER accepted this point, but added that a higher grade of paint applied in a thinner layer would often give better results than thick coats of an inferior grade of paint.

The use of more sophisticated synthetic binders and pigments made it possible to build up protective systems of lower film thickness than those of the old conventional (usually oil based) paints, using pigments of higher specific gravity.

In the case of a specific system, the thicker multi-layer systems would no doubt increase the protective value, since it was unlikely that weak spots or film faults in consecutive layers would coincide.

MR KILCULLEN replied that these were very valid points, although in a practical situation it might be more economical to use one of the older paint systems rather than the newer more sensitive type and to apply a thicker coat in order to achieve the protection required.

MR F. G. DUNKLEY was interested in the difference between the results for panels exposed at Battersea and at Leatherhead. British Rail experience was that panels exposed on the roof of a centrally heated building failed less rapidly than those exposed on an adjacent site on the ground (for example, in a field).

MR KILCULLEN confirmed that one exposure site was at ground level and the other at the top of a four-storey building.

MR DUNKLEY thought that this could explain, at least in part, the unexpected difference in performance at the two stations.

DR J. B. HARRISON said that if he were involved in a similar set of experiments and found surprisingly good performance, he would be very sceptical and would wish if possible to repeat the experiments from the beginning.

MR DAVIDSON remarked that in America they had found that films exposed in a rural atmosphere would fail faster than in an industrial atmosphere because of the greater concentration of dust and other contaminants in the atmosphere.

MR KILCULLEN said that one of the things which was noticeable from their set of experiments was the cleanliness of the panels exposed at Battersea compared with the ones at Leatherhead.

Returning to Mr Harrison's point, however, he thought that the main question to be answered now was whether the results reported in this paper meant anything at all in terms of paint testing and how relevant were the tests on small panels—at any site—in relation to the situation pertaining in reality to large structures, such as bridges.

Implications of the paint film contraction theory for comparisons of accelerated and natural weathering results*

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Summary

Alkyd paints pigmented with a range of titanium dioxide pigments have been subjected to tests by a number of methods after exposure for various periods and the results are compared. A modified BSS 3900 accelerated weathering cycle has been used and weight loss measurements, together with loss of gloss, TNO chalking rating, and red reflectance measurements on blue tinted panels are used to provide a consistent picture of changes during weathering, based on a "contraction model". Some methods for interpreting the detail in the results are illustrated, with special consideration given to chalking.

Similar comparisons are made with natural weathering results (at Nettleton, UK). The differences between natural and accelerated weathering are discussed, especially in terms of the penetration of ultraviolet light. The advantages of the control possible with artificial weathering methods are emphasised.

Keywords

Raw materials:
binders (resins, etc)

  alkyd resin

prime pigments and dyes

  titanium dioxide

Equipment primarily associated with analysis, measurement or testing

  glossmeter
  reflectometer

Properites, characteristics and conditions primarily associated with dried or cured films

  chalk resistance
  film erosion
  gloss retention
  shrinkage
  weather resistance

L'importance de la théorie de contraction des feuils de peintures en faisant les comparaisons des résultats des essais de vieillissement accéléré avec ceux de l'exposition aux intempéries

Résumé

Après de diverses périodes d'exposition des peintures alkydes pigmentées avec une gamme de pigments de dioxyde de titane ont été soumises aux essais de divers types et on fait comparer les résultats. On se servait d'une modification du cycle de vieillissement accéléré selon la norme BS3900 et on a utilisé les chiffres de la perte de poids, ainsi que ceux de la perte de brillant, le taux de farinage TNO, et les mesures de la lumière rouge reflétée par les éprouvettes revetues d'une peinture bleue, pour assurer une image rationnelle, basée sur un "modèle à contraction", des altérations se produisant lors du vieillissement. On explique certaines méthodes pour inter-préter en détail les résultats, et on considère en particulier, le farinage.

On fait de pareilles comparaisons à l'égard des résultats de l'exposition aux intempéries effectuée à Nettleton, RU. On discute les différences entre le vieillissement naturel et accéléré. surtout au point de vue la pénétration de la lumière ultra violet. On souligne les avantages du contrôle plus précis que l'on peut atteindre dans le domaine des méthodes de vieillissement artificiel.

Die Auswirkung der Lackfilm-Kontraktionstheorie auf Vergleiche von mit beschleunigten und natürlichen Bewitterungsprüfungen erzielten Resultaten

Zusammenfassung

Nach Exponierung von verschieden langer Dauer wurden mit einer Anzahl von Titanweissen pigmentierte Alkydharzemaillen verschiedenartigen Prüfmethoden unterzogen, und die Ergebnisse verglichen. Ein modifizierter BSS 3900 Kurzbewitterungstest wurde benutzt, ebenso Glanzverlust, TNO Kreidungsbewertung; und Rot-Reflektionsmessungen auf blau gefärbten Prüftafeln werden verwandt, um ein konsistentes, auf einem "Kontraktionsmodell" basierendes Bild der bei Verwitterung vor sich gehenden Veränderungen zu geben. Unter besonderer Berücksichtigung des Kreidens werden einige Methoden zur Interpretierung der Einzelheiten illustriert.

Ähnliche Vergleiche werden mit Resultaten natürlicher Bewitterung (in Nettleton UK) gemacht. Die Unterschiede zwischen natürlicher und Kurzbewitterung werden, besonders hinsichtlich des Eindringens von ultraviolettem Licht, besprochen. Die Vorteile der mit Hilfe von Kurzbewitterungsmethoden möglichen Kontrolle werden betont.

Introduction

Refs. 1, 2

In order to assess the performance of a surface coating, an objective measurement or assessment of some kind is required. As regards the durability properties, it is accepted that the main surface changes which must be measured are loss of gloss and appearance of chalk. The authors believe that weight loss measurements, whilst not necessarily directly related to obvious surface parameters, provide a fundamental measure which is very useful when used in conjunction with the other test methods.

Alkyd paint systems pigmented with TiO_2 have been used for the work described in this paper. On the basis of interference microscope measurements combined with weight loss determinations, a model based on the importance of appreciable contraction of the film during weathering has

been proposed[1]. This "film contraction" model has been used to explain many effects, which are summarised later in this paper. The contraction theory is used also to interpret the data presented in this paper.

Large numbers of weathering results from various accelerated cycles and from natural weathering sites have been compared in the literature. In many cases appreciable differences have been found, but little attempt has been made to interpret these differences. Work up to 1971 on paints containing titanium dioxide pigment has been thoroughly reviewed by Sullivan[2].

The aims of the authors' current work are:

1. To examine in detail (by frequent measurements) the changes in gloss, weight loss and chalking properties of a particular system under controlled conditions (accelerated weathering) using a limited range of TiO₂ pigments.

2. To illustrate methods of assessing these detailed results and to show how they can be interpreted in terms of changes in the film (using the contraction model).

3. To demonstrate relationships between the test methods for the system used and to provide evidence that the weight losses reported previously occur well before loss of pigment by chalking.

4. To compare these measurements with natural weathering results and to attempt an explanation of the differences observed. Since a general correlation is to be expected, it is the detail of the differences which is important.

5. To consider the practical implications of these ideas on durability testing and measurement.

Paint film contraction and its consequences

Ref. 1

It is well known that rutile TiO₂ strongly absorbs ultraviolet (UV) radiation and so has a protective effect on the binder (a photochemically oxidised binder, such as the typical alkyd used in this work). However, the pigment also assists the degradation by a photo-catalytic mechanism. Weight loss provides a fundamental measurement of the total degradation of the binder (photochemical plus photo-catalytic). All the evidence available indicates that weight losses equivalent to several microns of thickness occur on weathering before there is any loss of pigment[1], and this cannot be explained by erosion. No substantial "clear layer" exists to be eroded.

Starting with a concept that breakdown of the binder and the resulting weight loss is proportional to the damaging energy reaching each depth within the film, a picture is developed in which the greatest rate of weight loss is at the surface, gradually diminishing to zero within the film. As the products of breakdown of the medium are lost, the paint film contracts to take up the volume of the degraded material.

The effect of differential contraction is to cause pigment particles to move within the film, resulting in a progressive increase in the surface pigment volume concentration (PVC) with exposure. The contraction of the film and the resulting increase in surface PVC are factors which have not been appreciated in the past; they logically explain a large number of phenomena associated with weathering, and these include:

1. The shape of the curves of weight loss versus weathering time.

2. The effect of variations in initial PVC on the curves for gloss and weight loss.

3. The instances of increase in gloss during weathering.

4. The failure of various mathematical treatments of weathering phenomena.

5. The behaviour of paints containing photo-stable binders.

A further aspect which is also explained is that longer milling of an aggregated pigment, or the use of more easily dispersed pigments, improves the gloss retention, but does not essentially affect the weight loss. This is because contraction in the presence of an aggregated pigment leads to much greater roughness of the surface and consequent loss of gloss. The UV penetration may also be affected.

Pigments may show good gloss retention, therefore, either because their fundamental photo-catalytic activity is low or because they disperse well. By plotting durability results in the form of gloss against weight loss, the effect of dispersion can be distinguished since on this basis all similarly dispersed pigments give sensibly identical curves, independently of the various weathering times involved.

Preliminary comments on the chalking region

Ref. 3

In the current work, this kind of comparison is extended to chalking. There are problems in defining what is meant by "chalking", since different types of measurement necessarily define it in different ways. Clearly, as contraction continues, pigment particles eventually can no longer pack beneath the surface and become exposed—the nature of the chalking surface will depend on the relative photo-activity of the pigment and photo-stability of the medium. At this stage, however, the pigment particles are still strongly attached to the film. As weathering proceeds they become less well bound to the coherent surface and eventually they become completely loose. It is very difficult, therefore, to judge precisely what moment corresponds to the "beginning of chalking".

The term "free chalking" is used in this paper to describe the stage at which an appreciable amount of pigment becomes detached from the film so that it can be removed by minimal washing or gentle rubbing. For the system discussed here, using surface treated rutile pigments, this occurs when the surface PVC has increased to a virtually "close packed" state and, therefore, cannot increase further. The film surface can then change only by the build-up and removal of loose chalk. As degradation continues, removal of surface pigment enables UV light to penetrate further into the film, and the nature of the chalking surface remains essentially unchanged.

An adhesive tape measurement of chalking will clearly determine the amount of pigment that can be pulled out of the surface by the tape used. The importance of the effect of pigment adhesion on chalk ratings has been pointed out recently[3]. A reflectance measurement of chalking, on the other hand, will depend on the area of the surface on which uncovered pigment is visible. Both of these types of measurement are likely to indicate some "chalking" before any pigment is inherently free. One aspect of this study was to compare these types of measurement, and relate them to gloss and weight loss changes in terms of the contraction model.

Comparison of test methods

Refs. 4, 5, 6, 1

Experimental design

The measurements selected for comparison were the 45° gloss before and after washing, weight loss, and two measurements of chalking: the TNO adhesive tape test[4], and reflectance measurements (especially red reflectance) on blue tinted paints[5]. All these methods are used for assessing durability. It was also decided to use both natural and accelerated weathering.

For this work the carbon arc Marr artificial weathering apparatus operated closely to BSS 3900/F3 requirements was used for the accelerated testing. This cycle is in common use, especially in the United Kingdom, and is generally believed to correlate reasonably well with behaviour in temperate climates[6]. Panels were also exposed at Nettleton, Lincolnshire.

Four TiO_2 pigments known to have different durability characteristics were selected for this detailed study. All are surface treated rutile grades and are marketed internationally by different manufacturers. Pigments *1* and *4* are sulfate process grades; pigment *2* is a high durability and pigment *3* a relatively low durability chloride process grade. The same alkyd medium was used as in the previously reported work[1], but a constant initial PVC of 18 per cent was chosen for this comparison.

Four sets of stainless steel panels were prepared for each pigment. Duplicate panels were used for most of the tests:

A   Gloss and TNO chalking for panels not washed (gloss after washing, on half the panel for natural weathering).

B   Gloss after washing and weight loss.

C   Red, green and blue reflectances for panels not washed (blue tinted panels).

D   Red, green and blue reflectances after washing (blue tinted panels).

Experimental details

The paints were prepared using conventional methods and a standard formulation. The blue tinter used was a phthalocyanine blue at a concentration of 1.2g per 100g TiO_2. The paints were all ball-milled for 48 hours. Several 10cm × 15cm stainless steel panels were selected, cleaned, weighed and paints appropriate to the programme designed were applied by spinning. The panels for natural and accelerated weathering were prepared at the same time and treated in the same way.

The panels for test *B* (weight loss) were carefully cleaned in the conventional manner, all paint being removed, except that in the required area, which was then measured. Other panels were cleaned as appropriate (back and bottom strip for accelerated weathering, top strip also for natural), and they were all conditioned at 40°C for 13 days.

After the initial measurement, the panels for accelerated testing were inserted in a carbon arc Marr apparatus. They were removed and measured weekly for a total of about 1 400 hours and then fortnightly, taking a working day for measurement. They were always placed in a random order on the top rack of the Marr apparatus. All panels, except those for pigment 2, were withdrawn after about 2 200 hours, and

panels for pigment 2 after 2 900 hours (a total of 18 measurement stages).

Before each measurement, the panels for tests *B* and *D* were washed and the unpainted areas of the test *B* panels were then cleaned with acetone. The washing procedure consisted, generally, of gentle unidirectional downward wipes of the inverted panel with a wet J-cloth under running demineralised water, followed by thorough rinsing. Surplus water was shaken off and the panels allowed to dry under standard conditions.

The red, green and blue reflectances were measured on a Colormaster, Model IV. Measurements were made at two positions, one at each end of the panel, the instrument being set on the standard after every two panels. The gloss was measured on an EEL 45° glossmeter, in five positions on each panel, but when TNO chalking measurements were made at the top end of the panel, only four positions were used. "Top" in this context refers to the panel orientation in the Marr weatherometer.

The mean values of the gloss, the weight loss per unit area (mg/10cm²) and the reflectances were calculated. The accelerated weathering results are discussed below.

The panels for natural weathering were exposed at Nettleton on racks at 45° facing south, commencing 24 November 1971. They were withdrawn at three-monthly intervals (12 weeks, with one week for measurement), for 18 months, and then at six-monthly intervals up to 30 months: a total of 8 measurement stages.

They were examined in the same way as the accelerated panels, except that they were washed initially in warm detergent solution to remove attached dirt (except for measurements of TNO chalking and the reflectances of unwashed panels).

Accelerated weathering results and discussion

The results from test *A* are given at each weathering stage in Table 1. The other results are presented graphically. The symbols used for the four pigments are consistent throughout all the graphs.

TNO chalking

From Table 1 and Fig. 1, in which the chalk ratings are plotted against the weathering time, the clear differentiation between pigment *2* (low chalking) and pigment *3* (high chalking) is evident. Pigments *1* and *4* are similar in the chalk test, although the gloss retention is much better with pigment *1*.
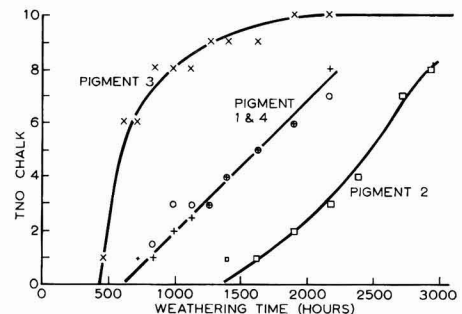

Fig. 1. TNO chalk rating versus accelerated weathering time

Table 1
Mean gloss values for panels not washed and TNO chalk ratings (accelerated weathering)

Time in Marr (hours)	Pigment							
	1		2		3		4	
	Mean gloss	TNO chalk	Mean gloss	TNO chalk	Mean gloss	TNO chalk	Mean gloss	TNO chalk
0	96	—	97	—	95	—	93	—
91	81	0	83	0	76	0	72	0
218	68	0	72	0	61	0	54	0
347	57	0	66	0	50	0	41	0
470	51	0	62	0	33	1	36	0
608	45	0	57	0	13	6	30	0
716	42	0	54	0	7	6	30	(1?)
841	39	1–2	54	0	4	8	22	1
991	32	3	48	0	3	8	17	2
1120	29	3	44	0	3	8	13	2–3
1255	24	3	38	0	2	9	10	3
1396	20	4	36	(1?)	2	9	8	4
1625	14	5	29	1	2	9	5	5
1901	8	6	20	2	1	10	3	6
2169	4	7	12	3	1	10	1	8
2390	—	—	8	4	—	—	—	—
2711	—	—	6	7	—	—	—	—
2938	—	—	5	8	—	—	—	—

Gloss for washed panels (test B): comparison with chalk ratings

In Fig. 2, the gloss levels for washed panels are plotted against weathering time. Large differences between the pigments are observed, with good agreement between duplicate panels, and the pigments can be put in the expected order (best first): $2 > 1 > 4 > 3$.
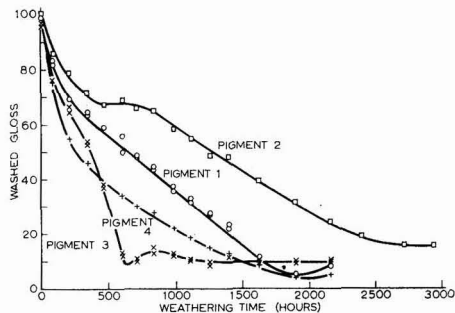
Fig. 2. Gloss versus accelerated weathering time for washed panels

When the gloss has fallen to a low value (< 20), the curves show a pronounced change in direction, and in most cases there is a small but distinct gloss increase. This effect is well known for gloss measurements on washed panels (or "washed gloss" measurements) and is associated with chalking. Visually, it occurs when there is sufficient loose chalk on the panel surface for an appreciable amount to be removed by the washing procedure. It can be associated, therefore, with the beginning of free chalking. Clearly, the removal of the first loose chalk from the surface can expose more uniform and reflective areas, which can contribute to a slightly increased gloss. It is worth while emphasising that the gentle water spray in the Marr apparatus does not remove any chalk up to this stage, and very little is removed later.

Approximate times at which this levelling off or slight increase in gloss occurs for the four pigments are given below:

2	1	4	3
2 700 hrs	1 900 hrs	1 900 hrs	600 hrs

Comparison of these times with the chalking data of Table 1 or Fig. 1 indicates that the change always takes place at or very near to chalk rating (6). Hence chalk rating (6) corresponds to "free chalking" under these conditions.

The washed gloss values corresponding to the "beginning of chalking", as defined by chalk rating (1), are indicated below for the four pigments:

1	2	3	4
44	48	37	30

The gloss values for panels not washed ("unwashed gloss" values) show similar significant differences between the pigments over the range 20 to 40. Hence the relationships between gloss and chalking are dependent on the pigment.

Unwashed gloss

Differences between the washed and unwashed glosses indicate a steady build up of dirt on the surfaces, especially for pigment 2. This may be an indication of its uniform surface and good chalk resistance. After free chalking, the unwashed gloss continues to fall in a smooth curve as chalk accumulates, whereas the washed gloss, as expected, reaches an effectively constant level as a steady state is approached.

The difference in gloss reaches a steady ten units for pigment 2. This indicates the effect that dirt build up can have even on accelerated weathering results.

Weight loss: comparison with washed gloss

In Fig. 3, the weight loss per unit area is plotted against weathering time. The curves have the expected shape, with a decreasing rate of weight loss as contraction moves the pigment nearer to the surface and so provides greater protection for the remaining medium. This is followed by an increasing rate of weight loss as pigment, as well as medium, begins to be lost. It is only at total free chalking, that is, at chalk rating (6), that a significant amount of material is removed by washing, and an appreciable change in slope occurs near the corresponding times. Before these times, there are indications that very small amounts of pigment may be lost.
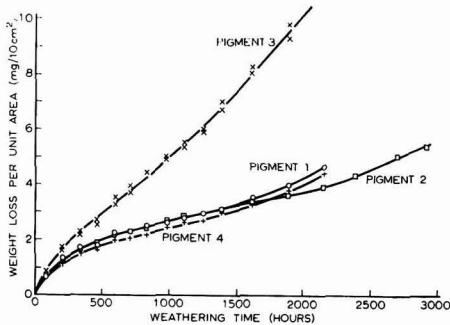
Fig. 3. Weight loss per unit area versus accelerated weathering time

It is clear that pigment 3 is much worse than the other pigments, and rather surprisingly considering the other measurements, pigment 4 has the lowest weight loss. The rate at which weight is lost at the beginning of free chalking appears to be comparable for all the pigments, although a wide range of weathering times is involved.

In Fig. 4, the washed gloss is plotted against the weight loss per unit area. This is the plot that gives information about the dispersion of the pigments. Dispersion in this sense refers to groups of a few primary pigment particles—much smaller than anything visually apparent.
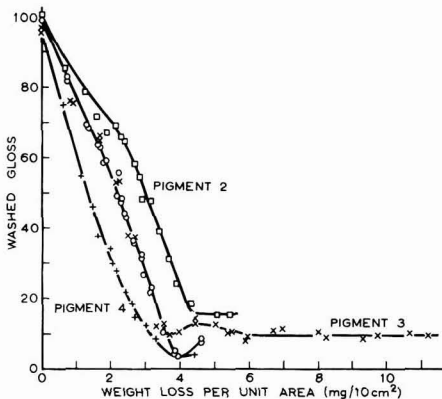
Fig. 4. Washed gloss versus weight loss per unit area

As expected, these curves come much closer together than the glosses plotted against time. The abrupt change in slope takes place at free chalking, which appears to occur after a weight loss in the range 3.5 to 4.5 mg/10cm². This is due to changes in both gloss and weight loss; that is, the rate of weight loss increases and simultaneously the rate of loss of gloss decreases.

After the initial period, all the curves run effectively parallel, with pigments 1 and 3 together. The clear distinction of the other two pigments suggests that pigment 2 is better dispersed and pigment 4 is poorly dispersed (more aggregated) in comparison with the pigments 1 and 3. This would not have been suspected from the gloss figures alone.

Washed and unwashed reflectances: comparison with chalk ratings

Blue reflectances show changes which are not related to chalking behaviour or any of the other measurements; in fact a minimum is observed near 1 000 hours, independent of pigment grade. The red and green reflectances, however, show very significant differences between pigments, and an identical pattern. Hence, only the red reflectance results are discussed here, being presented in Fig. 5 ((a) unwashed; (b) washed). There are large differences between the pigments with the clear order 2 > 1 > 4 > 3 in terms of lower unwashed reflectances: the same as with the gloss measurements.
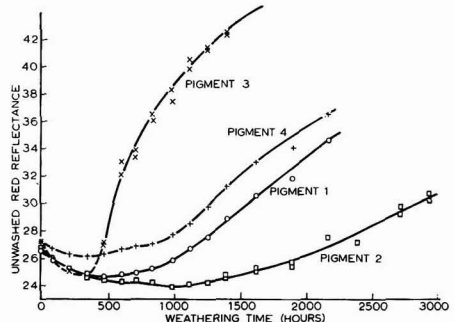

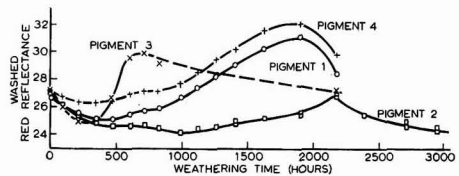**Fig. 5a. Blue tinted panels; unwashed red reflectance versus accelerated weathering time**

Fig. 5b. Blue tinted panels; washed red reflectance versus accelerated weathering time

The red reflectances show an initial gradual decrease, with a slight superimposed increase in the region of 600 to 800 hrs. This applies to both the washed and unwashed values, so cannot be associated with washing effects. The beginning of a rapid rise is then observed at times which roughly correspond to the first appearance of surface pigment, at chalk rating (1) to (2). The exact position is difficult to estimate because of the initial decrease, but there is no doubt about the differences between the pigments.

In this region, as would be expected, the washed and unwashed reflectances are similar (washed slightly higher

because of dirt removal). Later, however, whereas the un-washed values continue to rise as chalk accumulates, the washed values start to fall again as chalk is removed. The approximate times at which this occurs are given below for the four pigments:

2	1	4	3
2 300 hrs	1 900 hrs	1 800 hrs	700 hrs

These times correspond quite closely to "free chalking" (chalk rating (6)). This would also be expected, since it is at this stage that appreciable white surface chalk, which has accumulated to cover more of the natural panel colour, begins to be removed.

Agreement between duplicate panels and measurements on each panel are generally very good, and smooth curves are obtained on the graphs. When pigment 3 was chalking appreciably, variable washing removed different levels of chalk, and affected the washed values. This was done deliberately in order to assess the effect. The values for pigment 3 after free chalking are, therefore, not all shown.

An alternative representation of these relationships with chalking is shown in Fig. 6, where washed and unwashed red reflectances are plotted against chalk rating (from the last zero rating). Pigment 2 is slightly different, but the general relationship obtained is virtually independent of pigment, especially after allowing for different starting points. Thus the unwashed reflectances show a continuous increase, and the washed values (initially slightly higher) show a maximum at rating (6).
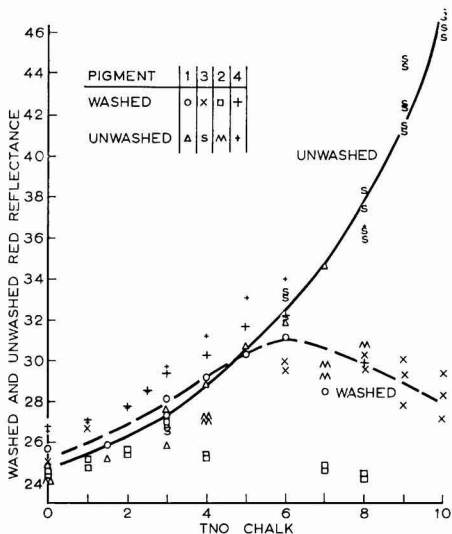
Fig. 6. Blue tinted panels; red reflectance versus TNO chalk rating

Chalking and weight loss: differences between pigments

In Fig. 7, the chalk rating (from the last zero rating) is plotted against weight loss. The pattern obtained is relatively inde-pendent of pigment. There is, however, a tendency for chalk-ing to commence at different points on the weight loss axis,

so that, at least for the early chalking stages, pigment 2 chalks at greater weight losses, and pigment 4 at slightly lower weight losses than the other two pigments. It is also worth noting that the washed glosses corresponding to the beginning of TNO chalking are slightly higher for pigment 2 and are lowest for pigment 4.
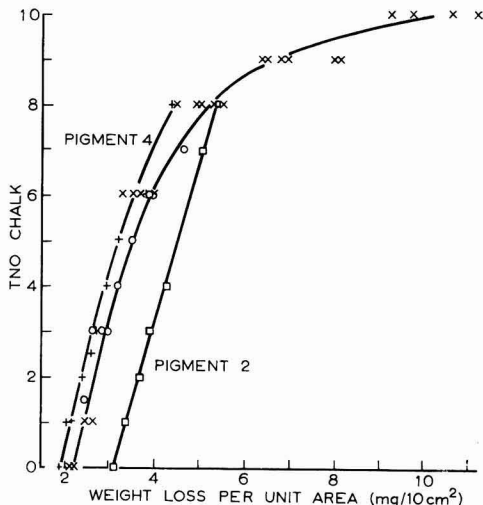

Fig. 7. TNO chalk rating versus weight loss per unit area

These effects can be related to the comments previously made about gloss and aggregation. Thus, a more finely dispersed pigment, for a given amount of weight loss and resulting contraction, may start chalking later and display a lower chalk rating than a more aggregated pigment. The gloss could also be higher at the same chalking level because of the more uniform surface. Thus, well dispersed pigments with an overall smaller and more uniform particle size and distribution would be expected to approach the surface more slowly and more uniformly.

Relative to its weight loss, pigment 2 shows a low gloss and very low chalk ratings before free chalking. This can be explained by the good micro-dispersion shown by this pigment. The low chalk ratings up to free chalking are then due to the firm retention of the pigment on and under the paint film surface.

After free chalking has commenced, a well dispersed pigment is likely to display a much more uniform chalking surface than an aggregated pigment. The free chalk should, therefore, be easier to remove by washing, leading to a smoother and more closely packed surface, and to higher gloss. Thus, pigment 2 shows the highest washed gloss value during free chalking. The maximum red reflectance for the washed tinted panels (Fig. 5b) appears to occur at a slightly lower chalking grade for pigment 2, and this maximum value is not as high as that for the other pigments. These effects can also be explained by the easier removal of free chalk, and support the contention that pigment 2 is well dispersed.

Pigment 4 shows a higher gloss loss and slightly higher chalk ratings than the other pigments relative to weight loss (the effect on gloss being most pronounced). Aggregation explains both of these effects. In addition, the washed gloss level at free chalking is lowest for pigment 4, suggesting that this pigment has the most uneven chalking surface and, therefore, provides

support for the presence of more micro-aggregates. In comparison, the other pigments *1* and *3* show intermediate dispersion/aggregation behaviour; it is the high weight loss of pigment *3*, and the resulting rapid onset of chalking, that makes it the worst of these four pigments.

Chalking measurements and surface changes on the contraction theory

The interpretation of the various measurements in terms of changes in the paint film surface can now be summarised. As weathering proceeds, material is lost from the medium and, as a consequence, contraction of the film occurs. The rate at which gloss is lost depends on both the contraction and the dispersion of the pigment. The red reflectance shows a slow decrease not related to the pigment.

Chalking occurs (TNO measurement) when some pigment reaches the surface and can be plucked out with the adhesive tape. This surface pigment appears first, other things being equal, with an aggregated pigment; hence a film containing such a pigment "chalks" first, and at a lower gloss value. Under the weathering conditions used, for a constant weight loss, the direct effect of aggregation on gloss retention appears to be greater than the effect on chalking.

As the amount of surface pigment increases, the red reflectance begins to rise (chalk rating (1) to (2)). This pigment is not removed on washing, but dirt removal causes washed gloss and red reflectances to be consistently slightly higher than the unwashed values. If the TiO_2 is of low photo-activity, the increasing surface PVC provides more protection for the medium and the rate of weight loss is reduced. Before any pigment is lost, the gloss values continue to fall, red reflectance values increase, and higher chalk ratings are obtained.

When the free chalking stage is reached, the surface layer of pigment becomes relatively loose, and can be removed by gentle washing, revealing a pigment layer of a high PVC still attached to the paint film. Hence at total free chalking there is an increase in the rate of weight loss and the washed gloss becomes constant, or shows a slight increase. Washed red reflectances start to fall again after free chalking, whereas unwashed values continue to increase (and unwashed gloss values continue to fall) as chalk accumulates. "Total" free chalking implies that the condition is uniform; there may be limited areas of free chalk, especially for a more aggregated pigment, at an earlier stage.

Weight loss (pigment and medium) continues until complete failure of the film. Chalk ratings increase as more pigment becomes loose, but the washed gloss rating reaches a steady value. The final steady value of the washed gloss may give an indication of the micro-dispersion; the more aggregated pigment will have a rougher chalking surface, and hence a lower gloss.

General comments on accelerated weathering comparison

The results and the inter-relationships between the test methods have been interpreted using the contraction model of the weathering process. The development of that model was based on the measurement of changes in the film before free chalking (loss of pigment). The current results provide evidence that, under the conditions employed, pigment is not lost until after the gloss has fallen appreciably (to below 20).

Dirt retention even on accelerated weathering can lead to significant differences between washed and unwashed gloss

values. This demonstrates the importance of washing panels for gloss measurement even when they are not exposed to an external environment.

It must be emphasised that the detailed relationships between the test methods, especially the measures of chalking, are applicable only to the type of medium and the weathering conditions used. Other relationships may well apply with other media. What has been demonstrated is a method of interpreting such differences in terms of surface changes in the weathering film.

Differences between natural and accelerated weathering parameters

Ref. 5

Accelerated weathering has a number of advantages over "natural" weathering, in addition to the acceleration in the testing time. These mainly relate to the control which it is possible to exercise over the weathering parameters of an accelerated weathering cycle.

A naturally weathered panel is exposed to:

1. A variation in light intensity on both a diurnal and annual basis.

2. A variation in the wavelength distribution of the light (on both a diurnal and annual basis).

3. A regular variation in the incidence angle of the light (the height reached by the sun also varies throughout the year). There is often also an appreciable and variable amount of diffuse radiation.

4. A variation in the amount and distribution of the rainfall, relative humidity, temperature etc.

5. The possibility of panel damage and the deposition of appreciable dirt on the surface.

6. Different weathering parameters from another "natural" weathering site.

Natural weathering at sites in the UK is very variable in the short term, none of the important parameters can be controlled and measurement of these is difficult since their exact nature is ill-defined. There are sites, of course, where the weather can be predicted, but results at such sites are relevant only to the particular climate involved and because of this, the data obtained need careful interpretation. Attempts have also been made to eliminate some of the variables, for example, by the use of variable angle racks[5]. However, the more such techniques are employed, the more artificial is the environment. By definition, natural weathering is always subject to lack of control of some variables.

In contrast, accelerated weathering can be controlled to provide a constant and reproducible cycle of weathering parameter changes. With a constant intensity, wavelength distribution and angles of incidence of the illumination, measured parameters can be meaningfully plotted against weathering time; where changes are rapid, the measurements can be made more frequently. This is an important advantage of accelerated weathering, where more than a simple comparison is required.

"Natural" weathering can be regarded, therefore, as the exposure of panels in an artificial environment (even the use of 45° racks introduces some elements of artificiality),

without any control of weathering parameters or easily observed changes in a reasonable time.

The importance of seasonal variations in UV light

Refs. 1, 7, 8

It has already been emphasised[1] that one important factor controlling weathering changes is the amount of UV radiation absorbed at various depths within the film, that is, the UV penetration. If more absorption occurs (and hence more degradation of the medium) near to the surface of the film, then relative to the amount of weight loss, surface changes in the film (loss of gloss and appearance of some chalk) will be more rapid.

It is very important to note that as well as seasonal variations in the total amount of UV light present in sunlight, the relative proportion of the UV wavelengths changes. As would be expected, measurements at the Nettleton exposure site show that the proportion of the shortest UV wavelengths is much smaller in winter than in summer. Typical values are given in Table 2 for three narrow band

Table 2
Relative UV radiation : winter and summer at Nettleton exposure site

		Filter		
		Short UV	Mid-UV	Visible
Peak transmission				
wavelength (nm)		316	350	544
Band width at half height (nm)		8	5	14
Winter month total	(a) 1973	37	60	98
radiation	(b) 1974	40	66	112
Summer month total	(a) 1973	825	709	811
radiation	(b) 1974	625	548	586

filters, two in the UV and one in the visible range, for comparison. The relative amounts of the shortest UV radiation (316nm filter) in mid-winter are less than half the summer values. It is probable that the shorter the wavelength, the more important is the difference. Wavelengths shorter than 310nm have a most pronounced effect on paint films, as has been recently demonstrated for accelerated weathering by the use of UV filters[7, 8].

Relative to the total received, the penetration of UV radiation into an exposed paint film is more in winter than a similar panel receiving UV light in summer. Winter degradation, therefore, occurs at a much lower rate than summer degradation, and it is also less localised on the surface. Identical panels exposed initially at different seasons should, therefore, show some differences. This is currently being investigated.

This also means that when pigments of differing photocatalytic activities are compared by initial exposure at the same time, seasonal fluctuations will reach the panels at different stages of the contraction process; the surface PVCs will be different when the shortest, more surface damaging wavelengths are present in the summer, and the UV penetration will be affected. This is relevant to the results of the present study.

It is clear, therefore, that seasonal variations in UV light intensity have an important effect on the breakdown rate of a paint film. Variations in wavelength distribution also

affect the visible signs of the breakdown. This is, of course, equally relevant to the correct choice of wavelength distribution for accelerated weathering, where the UV distributions can be closely controlled.

Measurement parameters

Panels must necessarily be measured after long periods of exposure. In the UK climate, appreciable dirt can collect on exposed panels between measurements. To remove in-grained dirt, washing needs to be more severe, and this may affect the intrinsic nature of the surface. It may not be possible to clean panels without some polishing effect. Variations over the area of the panel will also make all measurements less reliable.

"Natural washing" of the panels will also vary considerably. Thus heavy rain can remove dirt (or dirt and chalk where appropriate). The combined effect of wind-blown dirt and rain may also have a polishing effect on the surface, especially when little degradation is taking place. "Unwashed" panels will also be greatly affected by variations in natural washing.

Effects on the various measurements are considered in the following sections.

Natural weathering results and discussion

Most of the results are presented in the form of mean values over a period. This enables trends to be observed more clearly. Differences in detail from the accelerated results are summarised and interpreted in a later section.

TNO chalking

Chalking assessments are made on unwashed panels. The reliability of this test is, therefore, strongly affected by the presence of dirt. Over long time intervals, reproducibility is poor. Once pigment can be lost from the film, the effect of natural washing, wind and dust is likely to reduce the chalking values. Hence after free chalking, differences between the pigments are blurred.

The chalking total per panel for seven readings (that is, before free chalking) is given below:

Pigment	2	1	4	3
Chalking total per panel	1	4	4	12

This is the same relationship between the pigments as was obtained from the accelerated results, but the chalk ratings are lower and the differences between the pigments are less pronounced.

Washed gloss

Washing might not completely remove dirt (leading to a low gloss) or may polish the surface (leading to a higher gloss). The results obtained are not very sensitive, therefore, to real changes in the surface. Mean gloss results from both the weight loss and the chalking panels are given in Table 3,

Table 3
Mean gloss levels (natural weathering)

| | Pigment | | | |
Months	2	1	4	3
1 — 6	97	92	94	95
9	81	71	65	56
12 — 30	45	29	23	32

The order at nine months (where the maximum discrimination is apparent) is the same as that shown by the accelerated results. In the later results, however, the free-chalking gloss of pigment *3* is greater than the glosses of pigments *1* and *4*.

The final gloss values show the same order as for accelerated weathering, but the values are all higher and the range is also much greater. The high final glosses obtained probably indicate a surface polishing effect, with the complete removal of all slightly loose chalk. The development of "free chalking" cannot, therefore, be observed for these panels.

Unwashed gloss

Measurement of "unwashed" gloss on natural weathering does not have much direct meaning. Results taken on the tinted panels show a maximum difference between washed and unwashed gloss at nine months, that is, before chalking helps to remove dirt. There appears to be a slightly greater dirt retention on pigment *2*, with a difference of up to 25 units, but this may simply be a function of its good gloss. The accelerated weathering results showed a similar effect, although the dirt involved was much less.

Weight loss

The weight loss results are presented in Fig. 8. It is clear that seasonal effects are dominant and make interpretation of
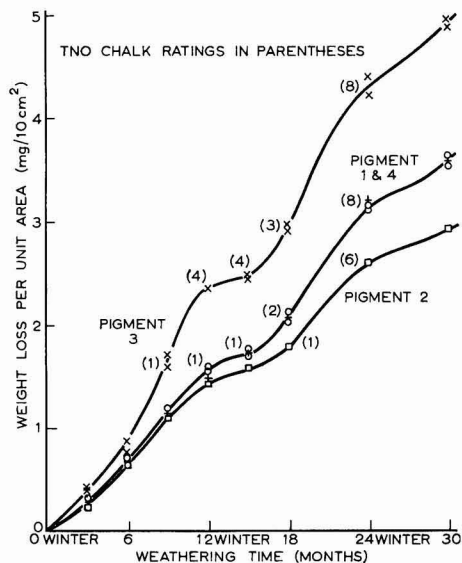
Fig. 8. Weight loss per unit area versus exposure time (natural weathering)

the shapes of the curves difficult. Differences between the pigments, however, are well defined. An advantage of the weight loss measurement is its cumulative nature; provided the washing is uniform, consistent effects are obtained.

Chalk ratings are also indicated on Fig. 8. The beginning of chalking as indicated by the TNO test takes place after essentially the same weight loss for all pigments. This represents a distinct difference from the accelerated results. This weight loss ($\sim 1.5\text{mg}/10\text{cm}^2$) is lower than was observed for any pigment on accelerated weathering. Higher chalk ratings, indicative of free chalking, occur after 18 months' exposure. The corresponding weight losses appear to be significantly lower than those obtained from the accelerated results (3.5 to $4.5\text{mg}/10\text{cm}^2$).

When these results are compared on a basis of gloss versus weight loss, a consistent series of parallel slopes is not obtained. This is probably because these pigments have such a wide range of activities that the effect of seasonal variations in the UV distribution on the pigments at different stages has an important effect. The appearance of removable chalk at an earlier stage, which results in higher final gloss values, also changes the nature of the relationship.

It is clear that very little degradation takes place during the winter periods. This is hardly surprising considering the reduction in the intensity of radiation especially in the UV region (Table 2). The effect of the weather may then be to induce changes in the film other than those due to loss of weight.

Washed and unwashed red reflectances on blue tinted panels

The natural weathering results are much less consistent than the accelerated values. The following points can be noted:

1. Over the initial stages (0 to 9 months), the washed values are greater than the unwashed ones. This is due to the presence of appreciable dirt.

2. After 18 months' exposure, the washed values are less than the unwashed. This must be due to the removal of chalk on washing.

3. The spread of values between the pigments is less for the washed panels than for the unwashed, indicating that chalk and dirt build-up has some differential effect. In general, however, there is much less dependence on the pigment than that displayed in the accelerated results.

4. The order of pigments at nine months and later is $3 > 4 > 1 > 2$ (both washed and unwashed). This is as expected. For the last few measurements, the reflectance of pigment *3* falls below that of pigment *4* due to the removal of appreciable chalk from the surface of the film. Thus the basic results are similar to the accelerated ones, but with fewer measurements and more fluctuations it is not worth considering the data in more detail.

Differences between the natural and the accelerated weathering results

In the results from the panels exposed to "natural" weathering:

1. There is less discrimination between the pigments in terms of both gloss retention and chalking (TNO and red reflectance).

2. TNO chalking begins after a lower weight loss, which is effectively the same for all the pigments. Free chalking also occurs after a lower total weight loss, and the chalking values obtained are generally lower.

3. The final values reached by the washed gloss are higher and there is then a greater spread between the pigments.

4. Differences between washed and unwashed red reflectances are smaller (after chalking).

These effects can all be interpreted on the basis of two postulated differences:

1. A smaller UV penetration on natural exposure, which strongly influences the initial weathering stages.

2. "Natural washing" on exposure to the weather, which has a surface polishing effect. This affects the results after the beginning of chalking.

The effect of UV penetration

Refs. 2, 8, 3

The weight loss results provide the most direct evidence that the UV penetration is less on natural weathering. A more surface localised attack will concentrate the contraction in the surface layers, and surface changes will occur for lower weight losses. This is especially important during the initial stages, where the UV radiation normally penetrates to the furthest depth, and the rate of weight loss is greatest. Differences are not large but are self-consistent.

Thus, pigment reaches the surface and the various stages of chalking occur after smaller weight losses. Some pigment is more easily exposed on the surface and can probably also be removed more easily. Hence, lower chalk ratings are obtained and the commencement of free chalking is less well defined. Dispersion of the pigment appears to have little effect on the beginning of chalking.

Another effect is that the proportion of the weight loss due to the photochemical breakdown of the medium will be increased; the pigments have less chance of displaying their protective capabilities. Discrimination between the pigments in terms of their photo-catalytic activities will be less, and hence differences in gloss and in chalking will be smaller. All of the measurements indicate that the panel surfaces from the different pigments are more nearly the same.

There are several possible reasons why the degradation is more surface localised. Probably a combination of factors is involved:

1. The much greater proportion of diffuse radiation reaching the panels on natural weathering. This will have less in-depth penetration. Even the direct radiation will reach the panels at a much wider range of angles.

2. Although the short wavelength UV distribution of the carbon arc may match an average sunlight, there is a large excess of radiation in the 400nm region[2,8]. This could mean that the carbon arc has a greater relative effect at lower levels within the film.

3. Appreciable dirt may reduce the UV penetration possible on naturally exposed panels.

4. Changes in the absorption of the resin over the long time intervals and temperature cycles involved. Changes in the mechanical properties of the paint film could also hinder contraction and have a similar effect.

A surface-localised contraction, for whatever reason it occurs, will clearly lead to a surface appearance rather similar to that which would be produced by erosion. Scanning electron micrographs of paint films of a similar type exposed to accelerated weathering have been published and interpreted largely in terms of erosion[3]. On the other hand, the features which these micrographs demonstrate are entirely consistent with the effects of contraction.

The effect of natural washing

The variable effect of the weather (wind and rain) is to remove some dirt before chalking, and eventually dirt and chalk. All degradative changes take place so much more slowly, that there is time for the paint surface to be "polished" to some extent by the weather. In the winter periods especially, virtually no degradation occurs and the surface can then consolidate.

Because natural washing removes chalk, the differences between washed and unwashed red reflectances are smaller. The amount of "chalk" on the surface (as determined by both types of measurement) is reduced. In the later stages, the (washed) gloss is increased.

The higher final gloss values are due to the removal of more chalk in the washing procedure, and it is then that dispersion differences become apparent. The better dispersed pigment (pigment 2) has the higher final gloss.

Practical implications

The carbon arc weathering cycle used shows larger differences between pigments than those which appear on natural weathering under the conditions used. This is consistent with much established data. The current work has demonstrated that this is due to more surface-localised degradation upon natural weathering in the UK.

This increase in discrimination on accelerated testing need not always be a disadvantage for testing purposes (for example, comparison of similar materials), provided it is understood. There have been a number of indications that the appropriately filtered Xenon arc, with its closer distribution compared to sunlight, gives better correlation with natural exposure in many cases; it is, therefore, steadily coming into greater general use.

Diffusion of sunlight could also be an important factor, and raises the question whether improvements in some weathering cycles could be made by the introduction of a diffuser. Measurement of the proportions of diffuse and direct radiation on natural weathering could also be useful.

It is easy to see why exposure sites with different climates should give very different results. It is clear that measurements of the total radiation received are irrelevant. The distribution of the UV and especially of the short wavelength UV has the dominant influence and measurements of this are more important. The contraction model provides a simple explanation of these effects within the context of the whole range of durability phenomena.

Conclusions

The contraction model can be used as an interpretive tool for comparing results from various test methods. The use of several different methods enables a picture to be formed of the physical effects of the degradation on the paint film throughout the weathering sequence.

Although not directly important from the point of view of the appearance of the film, the weight loss is the fundamental measure which enables changes in the visible parameters to be interpreted and explained.

Because of the control and accuracy possible, accelerated testing can give more information than natural weathering, especially at a site in a temperate climate, such as the UK.

It is possible to explain differences between results from different natural sites or artificial weathering cycles or between natural and accelerated weathering from a detailed study of the relationships between various measurements for the same systems.

In particular, the wavelength distribution of UV radiation, especially the shorter wavelengths, is of extreme importance in the early stages of weathering. This is because of the relatively low penetration of the shorter wavelengths, which has the effect of concentrating the degradation, and resulting contraction, close to the surface.

Variation in angle of incidence (including diffuse radiation) probably also has a significant effect in promoting the more surface-localised degradation of naturally weathered panels.

Once these effects are understood, accelerated weathering results can be used confidently to predict the effect of various natural exposure conditions.

Acknowledgments

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Discussion at Scarborough Conference

Prof. W. Funke asked whether the authors had any direct evidence for the contraction of paint films due to degradation on weathering.

It was conceivable that weight loss might also be due to the release of volatile material, such as the higher boiling retained solvents.

Mr Colling replied that the evidence for the contraction was basically from the interference microscope measurements and the results from these reported in the authors' paper presented at the last FATIPEC Congress. The only other possible explanation for the amount of weight loss which could be obtained before any loss of pigment was by assuming that there was a large amount of medium at the surface and that this was eroded. This did not seem likely from estimates of the amount of medium that would be required.

Before commencement of the experiments, the panels were "conditioned" for 14 days at 40°C and this was essentially to a minimum weight loss. This indicated that, in effect, almost all the solvent had been released. The weight then began to increase, principally as a result of oxidation.

Prof. Funke replied that his experiments had shown that after storing at even higher temperatures and for longer periods, there could still be a considerable amount of solvent retained in the film.

Mr Colling accepted that this might be so, although panels stored for long periods after conditioning had shown no significant change in weight compared with the exposed panels.

Dr Oesterle said that it was also possible to get a reduction of volume by shrinkage due to water. This happened when there was only a partial correspondence of the functional groups of the pigments and vehicle.

He reminded the authors of the papers by Völz and Kämpf, which had given a comprehensive theory of the onset of chalking due to a reaction between the pigment and vehicle.

He had taken this theory and shown that it was possible to detect a tendency to chalking long before there was any visible sign of chalking or detectable destruction of the surface.

Mr Colling explained that the work of Völz and others was an attempt to present a mechanism of photocatalytic weight loss. The paper presented here did not attempt to do this, but rather it accepted that there was a weight loss and proposed a model for this. The authors were aware that there were several theories for the mechanism of weight loss at the present time. In the systems they were studying, there were two factors in the weight loss (the photocatalytic effect of the pigment and the photo-degradability of the binder), and their model took account of both.

Dr G. de W. Anderson (Session Chairman) said that work done at the Paint Research Station several years ago closely paralleled the results of this paper. These had shown similar weight loss curves to those reported, and also a volume loss equal to that which would be expected. On chemical analysis, it had been found that only about five per cent of pigment was lost when the whole film had been reduced to chalk (that was, for an anatase pigment) and the loss per unit area during accelerated weathering was independent of film thickness, indicating that weight loss was primarily from the surface.

He wondered whether the authors had taken the effect of refractive index into account. The shrinkage would result in increased density and significantly increased refractive index of the paint film, which could account for some of the results. Dr Anderson's colleagues assured him that this would explain some of the unusual gloss change anomalies and also the observed initial fall in red reflection of blue tinted panels, as shown in Figs. 5(a) and 5(b). The rise in refractive index would reduce scattering by the white pigment and this would be accentuated by the reduction of inter-particle distance—with unchanging absorption of the blue dyestuff, the shade deepened; at a later stage as more pigment/air interfaces developed, white scattering would increase and the shade would become lighter.

MR COLLING replied that the increasing refractive index with increasing PVC had been used to explain the increasing gloss which it was possible to get on weathering. This effect was particularly noticeable with well-dispersed pigments. This was a phenomenon which had not previously been explained satisfactorily.

MR J. H. W. TURNER said that in his paper he would be showing that the loss of gloss and chalking of some paints pigmented with three grades of titanium dioxide appeared to be related—though not apparently directly related—to the loss of film weight which occurred under the same exposure conditions. Perhaps by lessening the loss in weight by oxidative degradation, aluminium compounds used as paint driers had reduced the tendency of the film to chalk.

MR WILKINSON said that their work was done primarily as part of a research programme on high durability titanium dioxide pigments and they had been very specific, therefore, in their choice of binder; the work reported here was, in fact, only concerned with one alkyd resin system. Certainly, in any programme such as the one Mr Turner had described, the durability behaviour of different titanium dioxide pigments could be explained by their photocatalytic properties and their state of dispersion in the binder.

MR Z. W. WICKS asked whether the authors had any experimental evidence to support the statement that relative to the total radiation, penetration of UV radiation was more in winter than in summer.

MR COLLING said that the statement was a deduction based on measurements of radiation which had been made at the exposure site. The depth of penetration of the radiation into the film would vary with wavelength, and the general shift in wavelength between the winter and summer periods caused penetration to be more (relative to the total) in winter. The phrase "relative to the total" was important: the total radiation received in summer would obviously be far greater than during the winter period.

DR M. L. ELLINGER remarked that good correlation had been mentioned between the long-term natural exposure and the accelerated laboratory test results in the 2 000 to 3 000-hour range of test period in the weatherometer. The reason which had been given for this was the facility for strict control over the UV content in the irradiating light, so that it was possible to simulate the seasonal ultraviolet variations observed for solar radiation. She wondered whether accelerated exposure tests had been attempted by the authors (for example, to simulate exposure conditions in Arizona) where results could be obtained within a very similar test period and under the local conditions of high UV content in the solar radiation, seasonal changes might not come into the picture during the test.

MR WILKINSON had two points to make concerning the use of accelerated "natural" exposure, such as used in Arizona cycles.

A programme they had sent to Arizona had had poor results due to exposure during a period referred to as a "low Langley period" (little sunshine and, therefore, low exposure period).

In their work, the authors felt they needed their own laboratory control in order to carry out intermediate assessments. In addition, the transport time involved was often too great for the experimental programme.

Next month's issue:

The Honorary Editor has accepted the following papers for publication, and they are expected to appear in the November issue of the *Journal*.

Theories—laboratory investigations—practical performance *by B. Lindberg*

The theory and practice of film formation by co-ordination reactions involving aluminium compounds *by J. H. W. Turner, W. K. H. Lakin and P. Womersley*

Practice makes perfect *by A. E. Claxton*

Short Communication——————————————————————

High-speed photographic studies of ink filamentation

By I. G. Thomson and F. R. Young

Watford College of Technology, Watford, Herts.

Summary

High-speed cinematographic and microsecond still photographic techniques have been employed to investigate ink filamentation between two rotating rollers for a known speed range. Ink film thickness and temperature were standardised. A standard letterpress ink was used. Analysis of the results indicates that the fila-

ment volumes and the maximum filament lengths attained, decreased with increasing speed. The behaviour of the filaments at the low roller speed range used may be less attributable to elastic flow than at high roller speeds.

Keywords

Etudes de la formation des filaments d'encre grâce à la photographie ultra-rapide

Résumé

La cinématographie à vitesse élevée et l'analyse photographique d'images prises à une microseconde d'intervalle sont les techniques que l'on a employées pour étudier la formation des filaments d'encre entre deux rouleaux rotatifs sur une plage de vitesses connues. La température et l'épaisseur de la pellicule d'encre étaient standardisées. On a employé une encre typographique

standard. L'analyse des résultats indique que les volumes de filaments et les longueurs maximales de filaments obtenues diminuaient en fonction de l'augmentation de la vitesse. L'écoulement élastique est sans doute un facteur qui influence moins le comportement des filaments aux vitesses inférieures de rouleaux employées qu'aux vitesses de rouleaux élevées.

Hochschnelle fotografische Untersuchungen der Druckfarben-Filamentation

Zusammenfassung

Hochschnelles Filmen und Methoden für Einzelaufnahmen in Abständen von Mikrosekunden wurden zur Untersuchung der Druckfarben-Filamentation verwendet, zu der es zwischen zwei über einen bekannten Geschwindigkeitsbereich rotierenden Walzen kommt. Stärke und Temperatur des Druckfarbenfilms waren genormt. Verwendet wurde eine Standard-Druckfarbe. Die Analyse

der erzielten Resultate zeigt, dass das Volumen und die maximal erreichten Längen der Farbfilamente mit gesteigerter Geschwindigkeit geringer wurden. Das Verhalten der Farbfilamente dürfte beim verwendeten geringeren Geschwindigkeitsbereich der Walzen weniger vom elastischen Farbfluss abhängen als bei höheren Walzengeschwindigkeiten.

Introduction

Refs. 1-5

Previous studies of ink filamentation have been made by various workers. Sjødahl[1, 2] used microflash still photography to study the kinematics of ink separation on rotating rollers. He was able to show that splitting of the film is preceded by the formation of thin filaments which elongate and then finally break. Hull[3] considered the effects observed by Sjødahl to be due to visco-elasticity. He based his argument for this on two main points:

(a) The standard white offset ink used by Sjødahl gave filaments of uniform diameter for a considerable part of the filament length.

(b) The absence of filament remnants after rupture.

Smith, Engle et al[4] carried out some high-speed cinematographic studies of ink film separation using an LTF Inkometer. From their general observations, they tentatively concluded that an increase in viscosity increased the filament population density, and that an increase in speed increased the number of filaments formed. Erb and Hanson[5] studied

the kinematics of film separation for a range of polymeric liquids between two metal blocks with parallel flat surfaces and of known mass. They found that in some cases the force tending to hold the blocks together could be attributed to viscous flow, and in other cases could be attributed to elastic storage.

The object of the present work was to make a more detailed study of ink filamentation between two rotating rollers for various roller speeds using a standard letterpress ink.

Experimental

A 16mm high-speed Fastax WF3 camera at maximum framing speeds of 5 500 fps was the instrument principally employed to record ink filamentation in the outgoing nip region between two rotating rollers (Fig 1). This filamentation was investigated for a comparatively low roller speed range of 0.25 to 1.6m s^{-1} at a constant ink film thickness of 19μm and at a temperature of 20°C. The ink used was Mander Kidd "Flash Concentrated Black" CQ99. In addition, microsecond still photographs were taken using an Ashai Pentax Spotmatic SP camera, with a single pulse from a Furzehill F61 strobolyser providing the light source. This
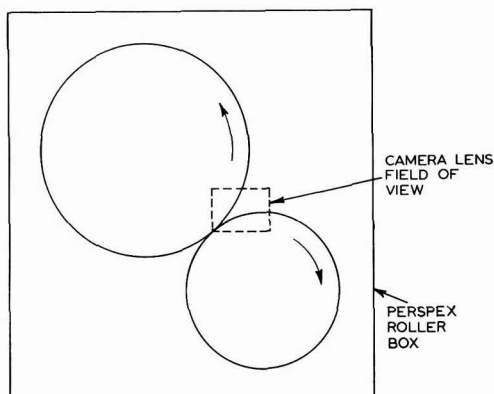
Fig. 1. Schematic diagram of roller arrangement

arrangement was satisfactory at low roller speeds, where speed blurr did not become objectionable for the purposes of analysis. Figs 2 and 3 show examples of the high-speed stills taken at a peripheral roller speed of 0.25m s⁻¹.

Fig. 2. Ink filamentation (× 4.3 magnification)

Five reels of the high-speed cinematographic records (at the following peripheral roller speeds: 0.282, 0.545, 0.797, 0.979 and 1.614m s⁻¹) were suitable for analysis. For two filament lengths common to all five reels, the filament diameters were measured at positions corresponding to 1/5, 1/2 and 4/5 of the length.
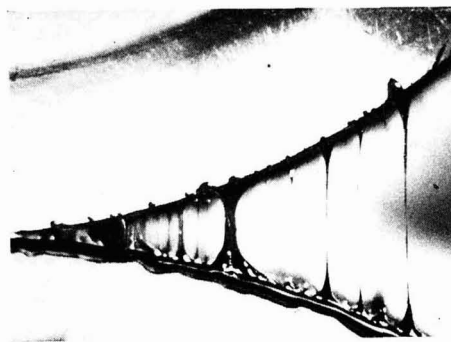
Fig. 3. Ink filamentation (× 4.3 magnification)

Discussion and results

Refs. 1-3, 6

Sjødahl[1, 2] and Hull[3] had proposed that the absence of the ruptured filaments after breaking (less than 5μs), was indicative of elastic behaviour. In the present investigation, it was noted that the ruptured filaments did not fully retract in the time taken for one revolution (approximately 10ms). This suggested that the elastic forces generated were of a smaller degree of magnitude. A factor contributing to this was the relatively low roller speeds, compared with those used in previous investigations.

The results indicate that the filament volumes decrease with increasing speed and, similarly, that the maximum filament lengths attained decrease with increasing roller speed. In conclusion, it can be stated that the behaviour of the filaments at these low roller speeds may be less attributable to elastic flow than at high roller speeds. More details are given in reference 6.

Acknowledgment

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Correspondence

Traffic paint compositions

SIR—After reading the article "Some traffic paint compositions" by Kamel *et al.* (*JOCCA*, 1975, **58**, 191-195), I was chagrined to say the least.

Having been the one-time chairman of the Traffic Paint subcommittee of the Technical Committee of Cleveland Society for Paint Technology, I had fond hopes of finding some new information for laboratory testing of traffic paints. I might add that the purpose of our subcommittee was to develop a series of laboratory tests for traffic paint, which would correlate with traffic paint performance and obviate the necessity for time consuming highway test stripes. Whilst the subcommittee never was able to conclude its work, several basic conclusions were drawn:

1. Weather resistance was as important as traffic resistance.

2. *Wet* abrasion resistance, rather than dry, was critical.

3. The degree of flexibility and adhesion over a metal substrate is meaningless.

4. Adhesion to concrete and asphalt is mandatory.

5. Freeze/thaw ($-40°C$ to $+100°C$) tests were more important than flexibility tests.

6. Most laboratory tests for paints would *not* correlate with traffic paint performance: that is, with respect to hardness, scratch resistance, gloss, flexibility, etc.

Whilst I am well aware of the fact that conditions in Egypt do not parallel those in the United States, there are basic matters which should be taken into consideration. I have no quarrel whatever with the authors' chemistry, and I think they should be commended for this portion of their work. I do take issue with them on *all* of their tests, and for my part, the project is only half done. Now that these new paints have been made, why not run *real* traffic paint tests on them, and let's see what really has been developed. Then, if any of the paints survive these tests, let's do an economic study!

If the authors are interested in pursuing this project, I will be glad to work with them on the traffic paint tests.

Yours faithfully,

J. H. BOATWRIGHT

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28 July 1975.

Reviews

Water-based paint formulations

By E. W. Flick

Park Ridge, New Jersey: Noyes Data Corporation 1975. Pp. xvi + 396. Price US$28.00

The author in his introduction, states that he has compiled 350 formulations of water-based paints from information derived from 2 000 sources. If, at the not inconsiderable cost of $28, a reader is seeking a collection of 350 trade technical data sheets, supplied in the main by United States raw material companies, then this book may be of some interest to him.

The ingredients are presented as trade-named materials and reference must be made to another part of the book for the suppliers and some indication of their nature. Some attempt is made to describe the raw materials under the (often misleading) heading of "Chemical description" where the reader will frequently find such unhelpful information as, for example, "Proprietary water-thinnable vehicle (40 per cent solids in water)"—hardly a chemical description and of little use in finding an alternative should it be necessary.

The formulations are divided into three sections covering exterior paints for trade sales, interior paints for trade sales and speciality paints and coatings. The physical constants and key properties of all the paints are given.

The book may provide an interesting conspectus of the US water paint scene, but it is not comprehensive in relation to the UK or European market. It is doubtful if it would be of much value to technical or managerial personnel outside the marketing areas of the named suppliers.

A. T. S. RUDRAM

Powder coatings technology

By M. W. Ranney

Park Ridge, New Jersey and London: Noyes Data Corporation 1975. Pp. xii + 426. Price US$36.00

Readers who are familiar with the Noyes Data Corporation Chemical Technology Reviews, based exclusively on the US patent literature, will know whether they will be of value to them. This present work consists of the reproduction of substantial parts, in particular the "Examples", of about 270 US patents on powder coatings. They are arranged in reasonable order, but there is no attempt at a critical survey or an overall summary.

For a field in which American invention has not exactly led, and in view of the slowness of appearance of US patents, this work alone could give a very misleading impression of the state of the art; it is essential that it be read in conjunction with other surveys covering European publications. Some people would, of course, prefer a wider bibliography, with the opportunity to obtain detailed patents later if required.

The book is arranged in two parts. The first covers coating compositions, divided into sections covering the particular polymers used: epoxy, vinyl, acrylic, fluorocarbon, polyester, polyurethane and polyamide. The second deals with industrial applications (namely to electrical components, pipes, glass and metals), and the powder coating processes (fluidised bed and spray application). There are company, inventor and US patent number indexes, but no subject index.

L. A. O'NEILL

Information Received

Burrell/Du Pont agreement

Du Pont (UK) Ltd and Burrell & Co. Ltd, of London have entered into an agreement under which the latter will contract manufacture "Krolor" chrome yellow and molybdate orange pigments for sale in the United Kingdom and Europe.

Pigments made by Burrell will be marketed by Du Pont (UK) through established European distributors. These pigments, prior to contract manufacture by Burrell, were made by E. I. du Pont de Nemours and Company for the European market at Newark, New Jersey, USA, and have been sold by European distributors since 1966. Du Pont's other pigments will continue to be exported from the United States and sold through the same distributors.

The decision to establish contract manufacture of "Krolor" in the United Kingdom was made to improve service to European markets. Du Pont will continue to make these products for the US and other non-European markets at its Newark plant.

C-Cure Coatings

P. W. Hall Ltd, the Scottish-based manufacurer of plastic coatings and colourings for the plastics industry, has announced the formation of a new company, C-Cure Coatings Ltd, to manufacture a range of high performance protective coating systems for buildings. The establishment of the new company is part of a large-scale diversification programme by P. W. Hall, which will involve a phased expansion by 25 per cent of the existing 2 322m² manufacturing facilities at the company's Kirkintilloch factory.

ICI to acquire interest in German paint manufacturer

Agreement has been reached in principle for the acquisition by ICI of a 70 per cent interest in the German paint manufacturer Hermann Wiederhold KG of Hilden, near Dusseldorf. As soon as the formalities are completed, the operations of ICI's existing German paint interests in Spangenberg Werke near Hamburg will be integrated with Wiederhold.

Wiederhold is the largest independent German paint producer and its paint capacity is nearly half that of ICI in the UK. It was founded in 1867 and now employs about 2 000 people in factories at Hilden, Nuremburg, and Hoorn, Holland. It is particularly strong in the fields of industrial paints, printing inks, wood finishes, can coatings and can sealants. ICI has owned Spangenberg Werke since 1962 and its strength lies in the decorative paint field, particularly with regard to white gloss paints.

As well as complementing its product range by the merger, ICI will gain a broader base in industrial paint technology, which should have relevance to the company's activities world-wide.

Middle East contract for Boulton

William Boulton Limited, Burslem, Stoke-on-Trent, manufacturer of ceramic and process machinery, has made a major breakthrough in the Middle East. On 17 August in Baghdad, the company signed a contract with the Iraqi Government to supply plant and machinery worth £100 000. The contract is the first step by Iraq to establish ceramic factories in the territory and includes provision for training two Iraqi technicians at William Boulton's Burslem plant. The firm will also carry out trials on ceramic materials from Iraq.

New company in Ireland

To give its customers in Ireland a comprehensive technical and manufacturing service, Swale Chemicals has formed a joint company with Gibbon Inks to manufacture liquid inks and lacquers: the new company has been named Swale-Gibbon Ltd.

Queen's Award to Industry for Allied Colloids Ltd

Allied Colloids Ltd, Bradford, has received the Queen's Award to Industry for its export achievement.

Since March 1966, Allied Colloids has adopted a deliberate policy to promote export business. In that year exports totalled £129 000. By 1971 the figure had risen to £1.43 million and that achievement won for the company its first Queen's Award. Today the figure is running at an annual rate of about £6.5 million.

New products
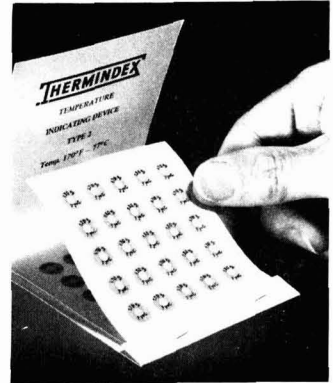
The new Thermindex single dot temperature indicating disc, which changes colour if a particular temperature has been reached or exceeded

Aqueous paint dispersant

The Marchon Division of Albright & Wilson Ltd has announced a new pigment dispersant for use in the manufacture of a wide variety of water-based paints.

It is claimed that the new product, called "Empicryl APD", when used in emulsion paints enables a high gloss to be imparted to the finished paint work, and the company explains that this is due to the outstanding dispersant properties of "Empicryl APD", which has a narrow distribution of molecular weight—one of the desirable features of a pigment dispersant.
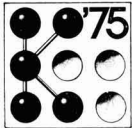
Process batch mixer

A new type of conical orbital screw mixer, specially developed for quick, high intensive mixing of solids, pastes and heavy emulsions, has been introduced to the UK and Eire by Delmec Engineering Ltd of Park Road Industrial Estate, Bacup, Lancs. Called the "Type S-KV", it is thought to be particularly suitable for process industry batching operations.

The mixing is implemented by a rotating screw attached to an orbiting arm, both of which are driven from the top of the mixer. The Type S-KV is available in 15 sizes covering batch sizes ranging from 1 000 to 25 000 litres. The mixer is manufactured in Zelham, Holland, by Machinefabriek Vrieco and distributed in the UK and Eire by Delmec, the sole agents for Vrieco's range of conveying, mixing and processing equipment in this area.
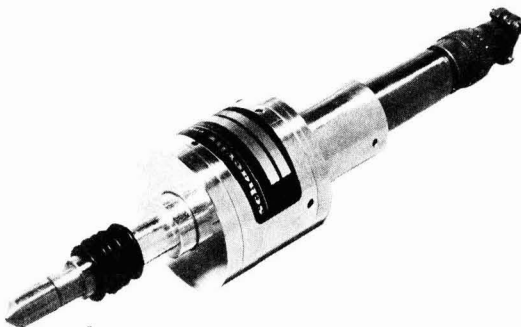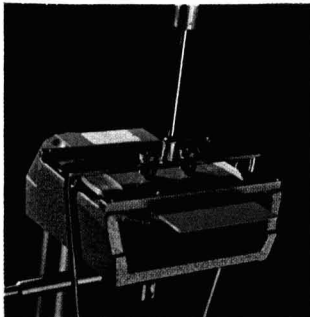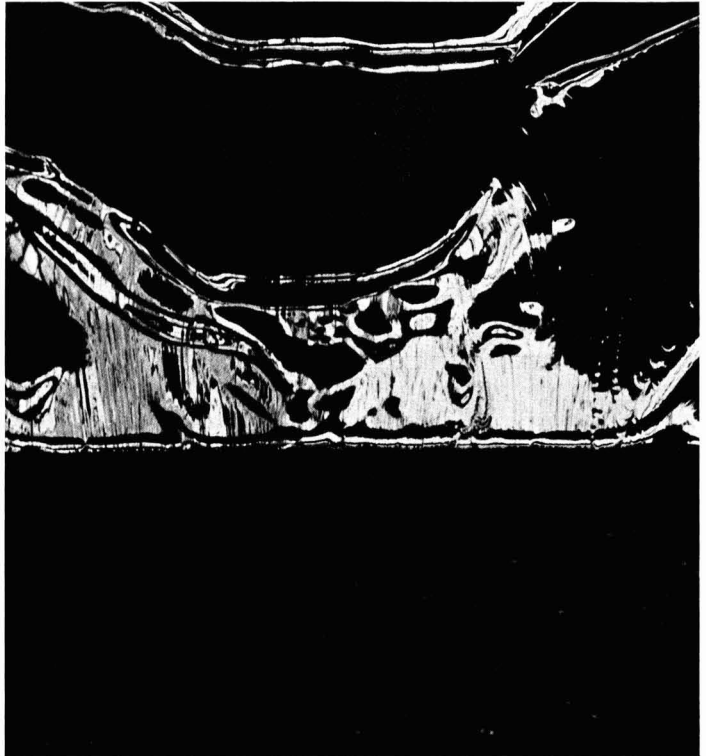

The new series PPA-230 non-contacting gauge head recently announced by Schaevitz EM Ltd, specially designed for thickness measurements on delicate materials or where accuracy is effected by the use of a contact head

Strengthened hose for high pressure spraying

Exit SA has recently introduced a new hose for high pressure (airless) paint spraying. It is claimed that the "Exitflex" hose will overcome all the problems which users have previously experienced with flexible type hoses for this application.

Thixotropic agent

Nynas Petroleum, a member of the Stockholm-based Axel Johnson Group, has announced a new product, designated "Fluosil", for the control and improvement of viscosity and thixotropy in a variety of applications, such as adhesives, fertilisers, lubricating oil, paints, plastics and printing inks.

Among the characteristics of "Fluosil" are its extremely low particle size and large specific surface, and the ability of its aggregates to form a three-dimensional network.

Low temperature curing varnish

Swale Chemicals has released a newly-developed coating for use on varnishing machines. Designated "KV 4453," this high gloss varnish system has been formulated to cure at temperatures as low as 60°C using traditional equipment, and is claimed eliminates the difficulties previously associated with catalysed varnishes. The cured film is fully glueable, the varnish may be used for up to six weeks after addition of catalyst, and the solvent is alcohol, which has no adverse effect on roller systems and does not involve hazards from obnoxious fumes.
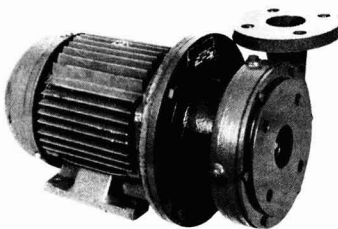
A new centrifugal model has been added to the range of industrial pump units manufactured by Stuart Turner Ltd, Henley-on-Thames; the new Type-160 pump shown above is designed to full metric specifications

Powder flow rate

A new instrument to measure the flow properties of powders intended for electrostatic application has been introduced into Great Britain by Binks-Bullows Ltd. The "AS 100" flowmeter is intended to help powder manufacturers and users achieve optimum flow characteristics, and is recommended for automatic electrostatic powder coating.

Conferences, courses etc

Colour technology

Instrumental Colour Systems Ltd will hold a three-day course on colour technology from 12 to 14 November 1975 at the company's premises in Newbury, Berkshire. The course is intended to cover the whole subject in some depth, with options for

those participants wishing to specialise in certain aspects of colour measurement etc. The registration fee is £89.00.

Paint testing

A two-day seminar on paint testing will be held on 11 and 12 November 1975 at the Paint Research Station, Teddington, Middx. The seminar will be a survey of the latest standard test methods and will include a comprehensive review of the application of the basic techniques of paint testing and the principles of operation of the various test methods employed.

Printing machines and ink transference

The annual lecture of the Technical Training Board for the printing ink and roller making industry, entitled "Printing machines and ink transference", will be given at the Royal Institution, London W1 on Monday 6 October 1975, commencing at 6.15 p.m. The speaker will be Mr P. J. Ogden, the executive chief engineer—research and development of Crabtree Vickers Ltd.

Literature

Carbon black pigments for paints

The British Standards Institution Sub-Committee PVC/1/9—Black Pigments has issued a draft for public comment of its specification for carbon black pigments for paints (BS 284, 286). The latest date for receipt of comments is 31 October 1975.

Pigments for cement

The British Standards Institution has announced that copies are now available of BS 1014: 1975—Pigments for Portland cement and Portland cement products.

Notes and News

OCCA-28 Exhibition

The continuous dialogue
between suppliers
and manufacturers
in the surface coating industries

The preparations for OCCA-28 to be held at Alexandra Palace London, 23-26 March 1976 are now well advanced and companies intending to exhibit at this unique technical Exhibition are reminded that the closing date for applications is 1 October 1975.

The Exhibition, which is known as the international forum for technical display and discussion in the surface coatings industries, is the most important annual event of its kind in the industries and it offers an unparalleled opportunity for a continuous dialogue on technical advances and problems between suppliers and manufacturers in an informal atmosphere.

Many young technologists and scientists who visited the earlier Exhibitions have now risen to high positions within their organisations and the advantage to exhibitors of keeping in constant contact with their counterparts in the manufacturing industries needs hardly to be stressed.

In recent years the Exhibitions have been visited annually by representatives from 50 countries and bringing all these interested parties together at a regular meeting is in itself invaluable when exhibitors consider the cost in time and travel which would be entailed in making personal contact with these visitors annually.

Alexandra Palace was the venue for the Exhibitions held by the Association from 1965 to 1969 and has many attractive features as the venue for this unique Exhibition. Alexandra Palace occupies a commanding position high on the North London hills and is less than two miles from the North Circular Road. Since the Association's last Exhibition at Alexandra Palace, travel arrangements have been improved by the addition of the Victoria Line to the Underground system, which now links Victoria Station to the Piccadilly Line at Finsbury Park. The building of the extension of the Piccadilly Line from Hounslow to the Airport terminal at

OCCA-28

For the benefit of intending visitors to the Exhibition, a map is reproduced below of the area around Alexandra Palace showing the mainline stations in central London, the Piccadilly, Circle and Victoria Lines of the underground system and also the road links with the North Circular Road and motorways
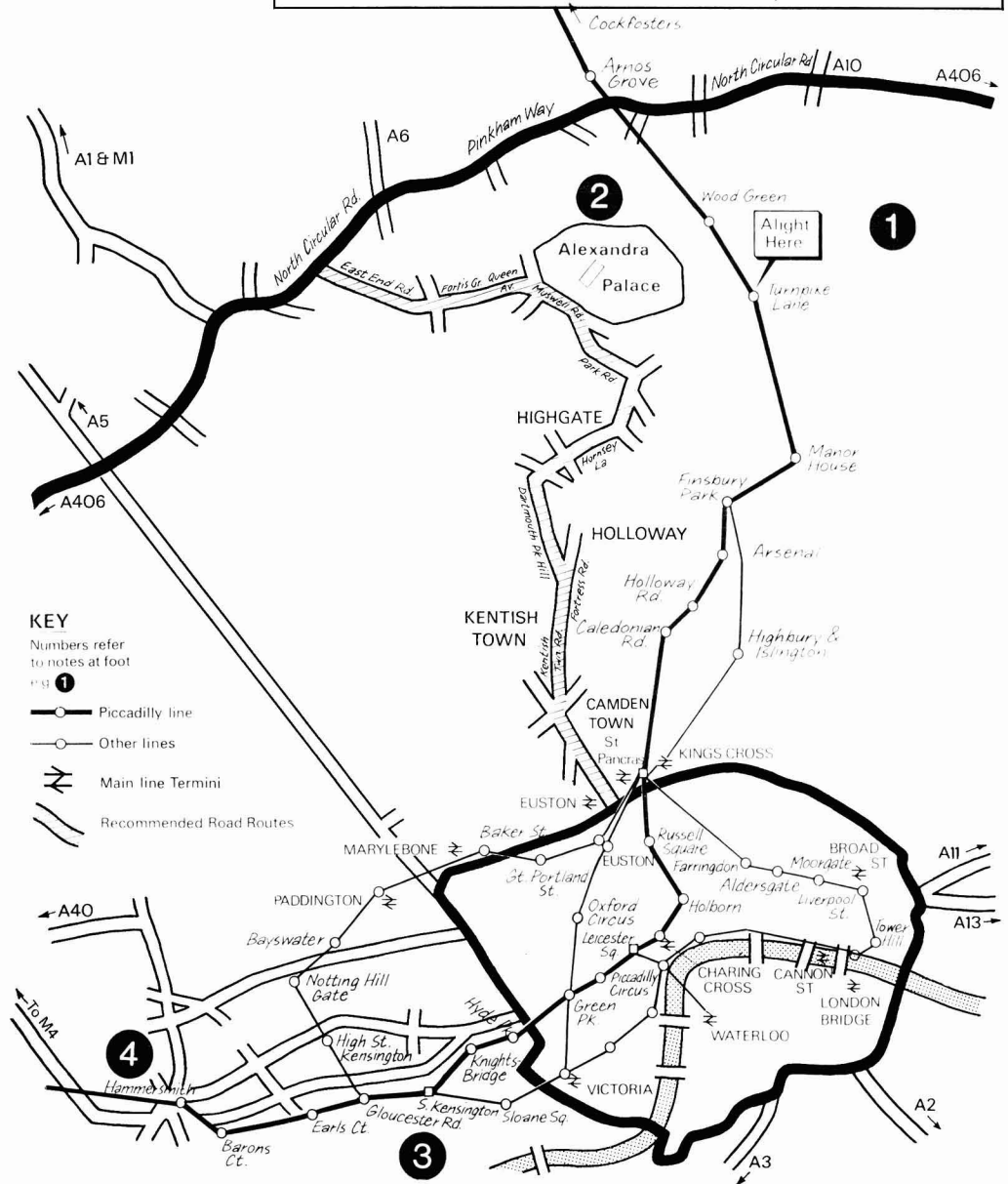
1. A free bus shuttle service will operate between Alexandra Palace and Turnpike Lane station on the Piccadilly Line (Underground). which is denoted by the thick coloured line. Destinations of trains may be marked as "Cockfosters" or "Arnos Grove" or "Wood Green".

2. Those travelling by road will find free car parking facilities at Alexandra Palace.

3. Visitors arriving at West London Air Terminal may board the Piccadilly Line trains at Gloucester Road station.

4. At present the Piccadilly Line reaches westward as far as Hatton Cross Station and in 1977 will be extended by the addition of a further station in Heathrow Airport Terminal, so that visitors will then be able to travel direct from the airport terminal to Turnpike Lane station. In the meantime, a bus service operates between the air terminal and Hounslow West station on the Piccadilly Line.

5. The map also shows the position of the main line stations in relation to the Piccadilly Line.

OCCA-28 Exhibition

Alexandra Palace, London. 23-26 March 1976

◎   ◎   ◎   ◎   ◎   ◎   ◎   ◎   ◎   ◎   ◎

The target for 1976!

◎   ◎   ◎   ◎   ◎   ◎   ◎   ◎   ◎   ◎   ◎

The international forum for technical display and discussion in the surface coating industries

Heathrow is now well advanced and when this is completed in due course it will give a direct link with Turnpike Lane Station on the Piccadilly Line. In the meantime, a bus service operates from Heathrow Airport to Hounslow West Station. Those travelling by the Piccadilly Line should alight at Turnpike Lane Station and the Association will be running a bus shuttle service from this station to and from Alexandra Palace. The journey from central London to Turnpike Lane takes approximately 18 minutes.

Visitors who prefer to travel from Heathrow to the West London Air Terminal in order to leave their luggage at hotels before travelling to Alexandra Palace can board Piccadilly Line trains at Gloucester Road Station. The map on page 394 will be reproduced in the *Official Guide*, and shows the connections between the mainline stations and the Piccadilly Line and also the link with the North Circular Road.

For those travelling by car from the Midlands or the North, the motorway system now links with the North Circular Road, from which Alexandra Palace is easily accessible; there will be ample free car parking space available at Alexandra Palace. The Exhibition will be open on four days commencing on the Tuesday morning at 09.30 a.m. and closing on the Friday at 16.00. There are two restaurants with full dining facilities together with a cafeteria and several bars.

Motif of the Exhibition

The motif for 1976, designed by Robert Hamblin, continues the theme of the last two Exhibitions—in which attention was drawn to the heavy participation by exhibitors and visitors from overseas—by showing the target for 1976 as London, where all the previous Association annual Exhibitions have been held. The colours of the motif this year are those associated with an archery target and these colours will be carried throughout the publicity leading to the Exhibition. Two of them will be incorporated on the facias of the stands—the blue colour from North to South and the yellow colour from East to West. As in previous years, this will give the Exhibition both an entity in design and will afford pleasing differences in colour as visitors move from corridor to corridor.

Invitation to Exhibit

Copies of the Invitation to Exhibit have been despatched to companies and organisations in the United Kingdom and overseas which have shown at previous OCCA Exhibitions or have requested information for the first time for the 1976 Exhibition.

Any organisation which has not previously exhibited and wishes to obtain an Invitation to Exhibit should contact the Association's offices immediately.

International forum

At OCCA–XXVII in April 1975 there were direct exhibits from 15 countries. Listed in the *Official Guide* were over 150 companies, many of whom were overseas companies showing through British associates. The full review of this important Exhibition appeared in the June issue of this *Journal*.

Technical education stand

In continuance of its interest in the educational field, the Association will once more provide a stand devoted to technical education.

Information in foreign languages

As in previous years, the Association will be circulating information leaflets in six languages, which will contain application forms for those wishing to purchase copies of the *Official Guide* and season admission tickets before the Exhibition.

"Official Guide"

This unique publication will contain descriptions of all exhibits and advertising space is available both to exhibitors and those organisations not able to show at the 1976 Exhibition. As in previous years, the *Official Guide* will be published several weeks in advance of the Exhibition so that intending visitors can obtain copies and plan their itineraries.

For the 1975 Exhibition, it was decided that a small charge should be made both for the *Official Guide* and for season admission tickets to the Exhibition. This policy undoubtedly prevented casual visitors who might otherwise be attracted to exhibitions for which no admission charge was made and who gathered quantities of technical literature from the stands. The innovation was welcomed by many exhibitors and in no way acted as a deterrent to visitors to this Exhibition. It is envisaged that a similar charge will be made for the *Official Guide* to OCCA-28.

Each Member of the Association, at home and abroad, will be sent a copy of the "Official Guide" and free season admission ticket.

As for the 1976 Exhibition, copies of the *Official Guide* and season admission tickets will be available several weeks in advance of the Exhibition (*pre-payment only*) from the Association's office and they will also be available for purchase at the entrance to the Exhibition Hall.

Full details of advertising in this publication, the basic rates for which are the same as for the *Journal*, can be obtained from the Association's offices.

Alexandra Palace map

Reproductions are available of the two-colour map shown opposite of the area around Alexandra Palace. Members may obtain copies by sending a stamped, self-addressed envelope, marked "Alexandra Palace map" in the top left-hand corner, to the Association's offices.

South African Section

Non-conventional coatings

The South African Section, in collaboration with the Council for Scientific and Industrial Research, announce that their sixth Technical Symposium will be held in Port Elizabeth, South Africa, from 7 to 9 October 1976.

The subject of the Symposium is "Non-conventional coatings", which will cover all aspects of surface coatings technology other than conventional paints. Powder coatings, water-based materials (other than conventional types), non-toxic coatings, cementitious materials, seamless flooring, mastics and sealants may be included. New techniques and developments in surface preparation, application and curing and specialised applications in the auto-

motive, marine and pipe coating industries are suitable subjects.

Authors who wish to present papers are invited to forward a synopsis of approximately 300 words for consideration by the Committee. Such synopses must be received not later than 31 December 1975 and the selected papers will be required by 31 July 1976.

All communications should be addressed to:

The Council for Scientific
and Industrial Research
Symposium Secretariat—S.125
PO Box 395
Pretoria 0001
South Africa.

South African Section—*Continued*

The Hon. Officers and Committees of the South African Section and its Branches are reproduced below to include postal codes and other information not available when the list was first published in the August issue of the *Journal*.

Chairman: D. J. Pienaar, MSc, c/o SABS, Private Box X191 Pretoria 0001

Vice-Chairman: E. Wright, c/o Shell Chemicals SA (Pty) Ltd, PO Box 494, Johannesburg 2000

Hon. Secretary: E. A. Duligal, BSc, ARIC, ARTCS, FICorrT, c/o SABS, Private Bag X191 Pretoria 0001

Hon. Treasurer: G. H. Munro, BSc, 16 Stander Street, Brackenhurst, Alberton 1450

Hon. Publications Secretary: P. A. J. Gate, BSc, ARIC, FTSC, PO Box 11270, Johannesburg 2000

Committee:

I. Lancaster, 18 Cedarberg Road, Noordheuwell, Krugersdorp 1740 Tvl

T. G. Edwards, BSc, Pinecrest Farm, PO Box 334, Halfway House 1685 Tvl

C. E. Simons, c/o Plascon-Evans (Pty) Ltd, PO Box 10, Luipardsvlei 1743 Tvl

Natal Branch .

Chairman: D. J. House, BSc, PO Box 1386, Durban 4000

Hon. Secretary: T. J. de Waal, MSc, ATSC, Private Bag 4, Umbogintwini 4120

Hon. Treasurer: K. M. Englebert, PO Box 3783, Durban 4000

Hon. Publications Secretary: L. F. Saunders, BSc, FTSC, PO Box 10, New Germany 3620

Immediate Past Chairman: R. A. Eglington, BSc, c/o Crown-Cebestos (Natal) Pty Ltd, PO Box 46, Jacobs 4026

Committee:

A. B. Spargo, c/o PO Box 78, New Germany 3620, Natal

K. E. Piggott, BSc, FTSC, 450A, Essenwood Road, Durban 4001

D. E. A. Williams-Wynne, MSc, PhD, FRIC, 15 Melrose Circle, Westville 3630

Cape Branch

Chairman: R. P. Johannsen, PO Box 42, Epping CP 7470

Hon. Secretary: A. G. Shepherd, Box 6449, Roggebaai 8012

Hon. Treasurer: H. J. Schone, PO Box 65, Strand CP 7140

Hon. Publications Secretary: A. Byrns, Box 2441 CT 8000

Committee:

E. G. Warman, Box 3847, CT 8000

D. F. Smith, Box 15 Observatory CT 7935

H. D. Saunders, 5 Private Road, Somerset West VP 7130

Transvaal Branch

This is administered by the Section Committee acting as the Branch Committee.

Report of Council Meeting

A meeting of Council was held at the Great Northern Hotel, London N1, on 10 July 1975 when 27 members of the Council were present. Mr A. T. S. Rudram took the Chair for the first time since his election as President at the Annual General Meeting on 20 June.

He extended a welcome to all new members of Council both at home and overseas.

The dates of the Council meetings for the forthcoming session were agreed, together with the composition of the committees of the Council. Consideration was given to the Association's representation on other organisations and these were agreed.

Reports were received on the Scarborough Conference which had been attended by delegates from 11 overseas countries; the five Technical Sessions had all been well attended and the discussions following the papers were lively and protracted. Papers will be appearing in forthcoming issues of the Journal. The arrangements for Symposia which would be taking place in 1976 were discussed and it was agreed to rearrange the dates for the 1977 Association Conference so that this would be held at Eastbourne. This would commence on a Thursday evening and disperse on the Sunday morning. The final details of the Reunion Dinner for all Council members, past and present, which will take place at the Rugby Club on 15 October, were agreed.

A full report on the 1975 Technical Exhibition and the preliminary arrangements for the 1976 Exhibition were considered.

Details were submitted showing the number of members whose subscriptions

for the current year had not been received and Council were pleased to note that this compared favourably with the position at a similar time in previous years.

Council were notified that, owing to an industrial dispute in the printing industry, the despatch of the June issue had been delayed.

The Council also considered information supplied by the Professional Grade Committee on admissions which had been made up to that date. It was pleased to learn that at least four colleges were now helping students in the preparation of their dissertations for the Licentiate grade.

Council considered the capitation fee for 1976 for OCCA Australia and reaffirmed that this would be at £6.00 each to include the whole membership of OCCA Australia.

Reports on the activities of Sections were carefully considered; in particular the forthcoming Manchester Section celebration of their 50th Anniversary and the arrangements which were being made to implement the setting up of South African and New Zealand Divisions.

It was agreed to confer a Commendation Award upon Mr T. R. Smith, Hon. Treasurer of the West Riding Section, in recognition of the outstanding part which he had played in furthering the welfare of that Section.

A Report was received on the Liaison Committee Meeting which had taken place at the Scarborough Conference and members were asked to inform the Director and Secretary of any members of the Association who were known to be present-

ing papers at any of the forthcoming conferences of the other societies in the International Alliance.

There being no other business the President thanked the Members for attending and declared the meeting closed at 3.40 p.m.

London Section

One-day symposium

The Section will hold a one-day symposium with the Kent sub-section of the Plastics and Rubber Institute at and in conjunction with the Thames Polytechnic, Woolwich, commencing at 9.30 a.m. on Wednesday 19 November 1975. The titles of papers to be presented and the lecturers are given below:

1. "Printing on flexible packaging," by Mr D. A. Fagg of Bowater Flexible Packaging Ltd.

2. "Printing on rigid plastics," by Mr K. Kohlhepp of Hoechst (UK) Ltd.

3. "Vacuum metallising—as a technique," by Mr B. J. Williams of Edwards High Vacuum Ltd.

4. "Decoration of floor coverings," by Mr P. J. Lyon of The Marley Tile Co. Ltd.

5. "Problems in the pigmentation of plastics," by Mr D. M. Varley, of CIBA-Geigy Ltd.

6. "Decoration of plastics with surface coatings," by Dr S. Kut of E. Wood Ltd.

London Section—*Continued*

Works visit

Acknowledgment is given to M. F. Worsdall, a registered student attached to the London Section, for his comprehensive report, from which this summary has been compiled.

A party visited the Harwich Refinery of Carless Solvents Ltd on 23 July 1975.

I●was stressed that the refinery received no crude oil, but that it was basically for the treatment of hydrocarbon liquids arising from the processing of natural gas from the North Sea. The products could be divided into those used for deposition, those used for chemical processes and those used as fuels. In the first category, spirits of specific boiling point were widely used for the extraction of animal and vegetable oils and fats and this covered the fields of soap manufacture, extraction of oil from oil-bearing seeds and leather degreasing. In the second category, the spirits were used for the manufacture of adhesives and adhesive tape, tyre manufacture and fabric proofing. The spirits were also used for the preparation of cellulose lacquers as solvents for resins in the paint industry and in the preparation of photo-gravure inks in the printing ink industry. In the third group, gasoline and naphtha were used for reforming in town gas plants, the chemical feedstocks being used for such products as ethylene and its derivatives; and in the fourth group, large quantities of kerosene were manufactured for use as fuel.

An enjoyable afternoon was had by all and a vote of thanks to Carless Solvents Ltd for arranging the visit and for providing a lavish tea, was made by the Section Chairman, Mr J. Tooke-Kirby.

News of Members

Dr R. H. Leach, an Ordinary Member attached to the London Section, and director of the Fishburn Printing Ink Company, has retired. Dr Leach was one of the best-known personalities in the printing ink industry and his unique contribution to the formulation of varnishes and inks for packaging and publications earned him a high reputation. He joined the Fishburn Printing Ink Company in 1937, having gained his doctorate in chemistry at University College, London, and became Fishburn's first research chemist. Over many years he played a major part in the technical developments of the company, and today there are nine separate Fishburn laboratories in Britain.

Dr Robert Leach

Mr C. H. Morris, an Ordinary Member attached to the Newcastle Section, has been appointed general manager of the Resinous Chemicals Division of Berger Chemicals, Newcastle. Mr Morris joined Berger in 1969 as marketing manager of the Resinous Chemicals Division and, as a member of the Association, he has served as Chairman of the Midlands Section and as a Vice-President.

OCCA paper to be presented at the FATIPEC Congress 1976 at Cannes

It is felt that members will be pleased to learn that the Association's paper to be presented at the FATIPEC Congress, which will take place at Cannes from 2 to 7 May 1976, will be given by Mr J. C. Bax of Scott Bader Co. Ltd under the provisional title "The formulation of gloss emulsion paints".

Register of Members

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

Ordinary Members

ABBOTT, KENNETH, Canada Colors & Chemicals Ltd, 160 Bloor St. East, Toronto, Ontario M4W 1C6.
(*London Section—Ontario Branch*)

AVIS, JEREMY PHILIP, BSc, 30 Castle Crescent, St. Briavels, Nr. Lydney, Glos. (*Bristol*)

BRETTELL, STANLEY VICTOR, Llewellyn Ryland Ltd, Haden Street, Birmingham B12 9DB. (*Midlands*)

FERGUSSON, GRAHAM KENNETH, BSc, 25 Vine Street, Mandere East, New Zealand. (*Auckland*)

FIBIGER, WALTER, Canadian Hoechst Ltd, 100 Tempo Avenue, Willowdale, Ontario. (*London Section—Ontario Branch*)

FRASER-THOMSON, GORDON, Delamere, Towers Road, Poynton, Nr. Stockport, Cheshire SK12 1DF. (*Manchester*)

LEBENON, BADDIE, Cedar-Radex Paint & Lacquer Industries (Pty) Ltd, PO Box 39752, Bramley 2018, Transvaal.
(*South African*)

LOCKIE, ANDREW ROBERT, Lime & Marble Ltd, Private Bag, Nelson, New Zealand. (*Wellington*)

MADDY, ALAN JOHN, 10 Marlborough Close, Bishop's Stortford, Herts CM23 3NT. (*London*)

MALIK, JAVED HAIDER, 18-C-2 Gulberg III, Lahore, Pakistan.
(*General Overseas*)

MCNAB, JOHN CROMBIE, BSc, Sandeman Bros. Ltd, 50 Bilsland Drive, Maryhill, Glasgow G20 9SE. (*Scottish*)

NORRIS, BRIAN, 65 Stewart Street, Goodwood 7460.
(*South African*)

POWELL, HAROLD CRAVEN, BSc, Buckley & Young Ltd, PO Box 5641, Auckland. (*Auckland*)

REDMOND, ALLISTER, 858 York Mills Road, Don Mills, Ontario.
(*London Section—Ontario Branch*)

ROSSI, GIANCARLO, PO Box 39595, Bramley 2018, Transvaal.
(*South African*)

WALLER, ANDREW GEORGE, BSc, Hoechst (UK) Ltd, 48 Seymour Grove, Old Trafford, Manchester M16 0LN. (*Manchester*)

WHALLEY, BARRY THOMAS, AG Healing & Co. Ltd, PO Box 19127, Avondale, New Zealand. (*Auckland*)

WILSON, DAVID SMITH, 2170 Sherobee Road, Apt 1512, Mississauga, Ontario. (*London Section—Ontario Branch*)

YICK, PAUL, BSc, 82 Peter Street, Toronto, Ontario.
(*London Section—Ontario Branch*)

Associate Members

BLEWMAN, MICHAEL JOHN, Buckley & Young Ltd, PO Box 30-148, Lower Hutt, New Zealand. (*Wellington*)

COX, WILLIAM HUTCHINSON, 122 Park Hill Road, Birkenhead, New Zealand. (*Auckland*)

EASTON, BRUCE WAYNE, Buckley & Young Ltd, PO Box 5641, Auckland. (*Auckland*)

HUNTER, MICHAEL NEIL, Henry H. York & Co. Ltd, Marine Parade, Petone, New Zealand. (*Wellington*)

KUNZ, ERNST, 858 York Mills Road, Don Mills, Ontario.
(*London Section—Ontario Branch*)

MORKEL, DAVID WILLIAM, Gypkor (Pty) Ltd, PO Box 1, Philippi 7781. (*South African*)

RACK, SHAUN BRIAN, Plascon-Evans Paints (Cape) Ltd, Box 42, Eppindust, Cape Town. (*South African*)

RIVERS, PAUL DAMIAN, Box 3747, Wellington. (*Wellington*)

Forthcoming Events

Details are given of meetings in the United Kingdom up to the end of the month following publication.

October

Thursday 2 October

Thames Valley Section: "Safety: recent Acts" by Mr R. E. L. Everett, Safety Officer, ICI Paints, to be held at the Beaconsfield Crest Motel (White Hart), Aylesbury End, Beaconsfield, Bucks at 6.30 pm for 7.00 p.m.

Newcastle Section: "Recent developments in organic pigments" by Dr E. R. Inman, Ciba-Geigy (UK) Ltd, to be held at the Royal Turks Head Hotel, Newcastle upon Tyne at 6.30 p.m.

Friday 3 October

Hull Section: Annual Dinner/Dance to be held at the Willerby Manor Hotel, Willerby, Hull.

Saturday 4 October

Scottish Section—Eastern Branch and Student Group: Subject to be announced. The meeting will start at 10.30 a.m. in the Lady Nairn Hotel, Willowbrae Road, Edinburgh, followed by lunch and the annual skittles match.

Monday 6 October

Hull Section: "Polyesters for boat building, tank storage and chemical engineering" by a speaker from Scott Bader Ltd. Joint meeting with the Institution of Chemical Engineers at the Haven Inn, Harrow Haven, Lincs.

Thursday 9 October

Midlands Section—Trent Valley Branch: "Lead chromes: their present uses and future trends, with particular emphasis on recent regulations" by Mr R. M. W. Wilson of SCC Colours, London, to be held at the Crest Hotel, Pastures Hill, Littleover, Derby at 7.00 p.m.

Friday 10 October

Manchester Section: "Handling of powders and pigments in bulk" by Dr N. Harnby, School of Chemical Engineering, University of Bradford, to be held at Manchester Literary and Philosophical Society, George Street, Manchester at 6.30 p.m.

Tuesday 14 October

West Riding Section: "Gloss emulsion paints" by Dr Faulkner of Rohm & Haas (UK) Ltd, to be held at The Griffin Hotel, Boar Lane, Leeds at 7.30 p.m.

Wednesday 15 October

Reunion Dinner for past and present members of Council to be held at the Rugby Club, 49 Hallam Street, Portland Place, London W1N 5LJ at 6.30 for 7.00 p.m. Informal dress.

Thursday 16 October

London Section: "The coatings industry in a changing world" by Dr H. Wittcoff, Prof. of Chemistry, University of Minnesota and Special Adviser to General Mills Chemical Inc. *Afternoon meeting:* lecture commencing at 2.30 p.m. at the Imperial College of Science & Technology, Prince Consort Road, Kensington, SW7.

Scottish Section: "The Health and Safety at Work Act" by Mr F. Hyland, HM Factory Inspectorate, to be held at the Beacons Hotel, 7 Park Terrace, Glasgow at 6.00 p.m.

Friday 17 October

Irish Section: "Fuel economy" by Mr S. W. Carroll, Institute for Industrial Research & Standards, to be held at the Clarence Hotel, Dublin at 8.00 p.m.

Midlands Section: "The coatings industry in a changing world" by Dr H. Wittcoff, of General Mills, to be held at the Apollo Motel, Hagley Road, Edgbaston, B15 at 6.30 p.m.

Friday 24 October

Manchester Section: 50th Anniversary Dinner/Dance, Piccadilly Hotel, Manchester.

London Section: Ladies' Evening at the Piccadilly Hotel, London W1.

Friday 31 October

Midlands Section—Trent Valley Branch: Halloween Dance at Cross Keys Inn, Turnditch, at 7.30 p.m.

Bristol Section: "Gloss paints: water thinned or solvent thinned?" by Mr D. G. Dowling of Berger Paints, to be held at the Royal Hotel, Bristol at 7.15 p.m.

November

Monday 3 November

Hull Section: "The factory inspector and industry: partnership is preferable to imposition" by Mr R. V. Souter of HM Factory Inspectorate.

Thursday 6 November

Manchester Section: 50th Anniversary Foundation Dinner, Midland Hotel, Manchester.

Newcastle Section: "Industrial applications of organic titanates" by Dr P. D. Kay, Titanium Intermediates Ltd.

Thames Valley Section: "Treatment and disposal of hazardous industrial wastes" by a speaker from Re-Chem International Ltd.

Tuesday 11 November

Scottish Section—Student Group: "Physical measurements of pigments" by Mr G. Murdoch of CIBA-GEIGY (UK) Ltd.

West Riding Section: "Prospects for surface coating resins in the European market" by Mr C. Martin of Amoco Chemicals Europe.

Thursday 13 November

Manchester Section: Ladies' evening with "Beauty" lecture, at Last Drop, Bolton.

Midlands Section—Trent Valley Branch: Joint meeting with the Society of Dyers and Colourists. Subject and venue to be arranged.

Wednesday 19 November

London Section: "Decoration of plastics". One-day joint symposium with the Kent Sub-Section of the Plastics and Rubber Institute, to be held at and in association with Thames Polytechnic, Woolwich, commencing at 9.30 a.m.

Thursday 20 November

Scottish Section—Eastern Branch: "Dispersion machinery" by Mr R. Webster of J. C. Forbes Ltd.

Friday 21 November

Bristol Section: "The Health and Safety at Work Act" by Mr J. F. Alcock, HM District Inspector of Factories.

Irish Section: Annual Dinner Dance, Clarence Hotel, Dublin, at 8.30 p.m.

Midlands Section: "The outworker" by Mr D. E. Hopper of Ludford Enamelling. Student lecture.

Friday 28 November

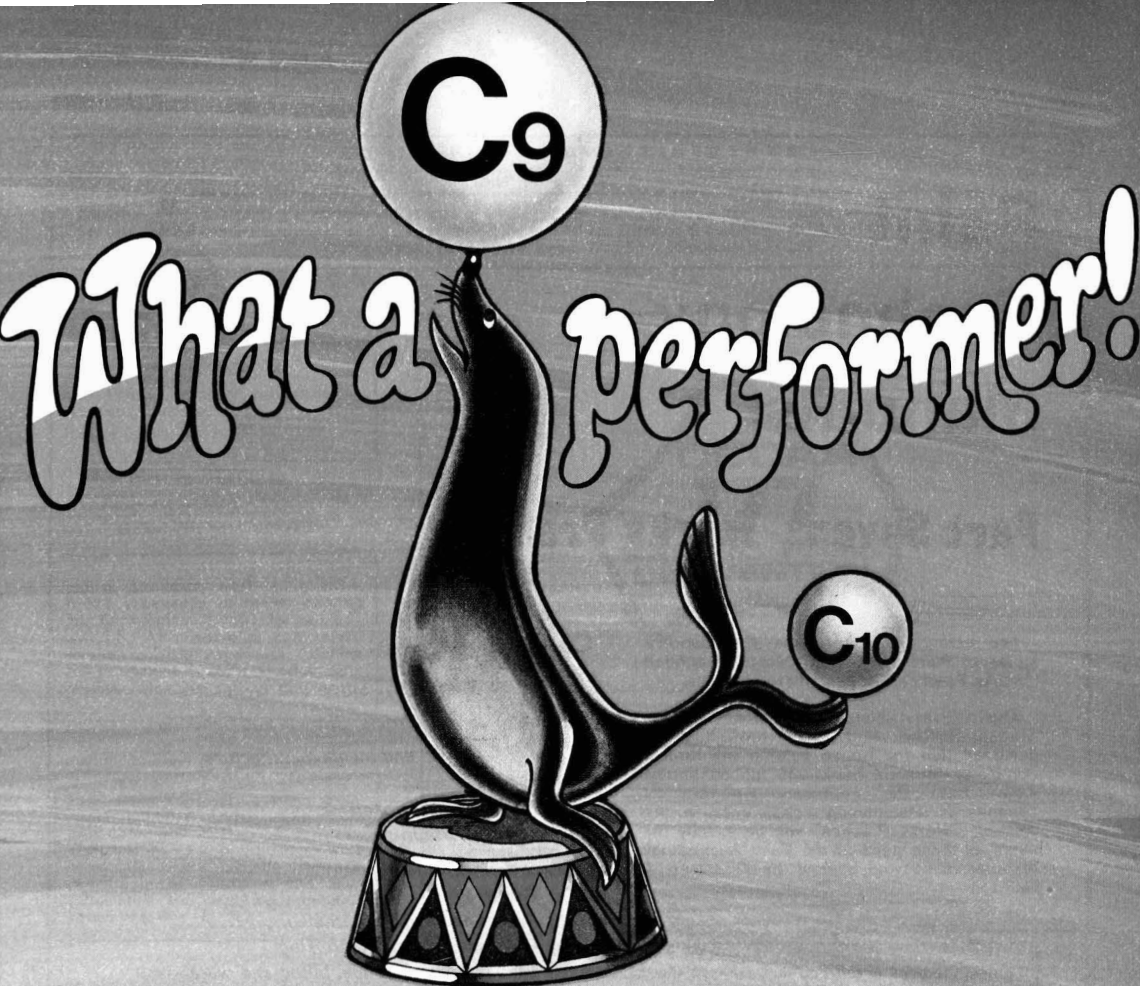*West Riding Section:* Dinner and Dance at the Crown Hotel, Harrogate.

Reunion Dinner for past and present members of Council

Following the successful innovation in 1973 of a Reunion Dinner for those members who have served on Council at any time, Council has decided to hold a similar event this year. The Dinner will take place on Wednesday 15 October 1975 at the Rugby Club, 49 Hallam Street, Portland Place, London W1N 5LJ, at 6.30 for 7.00 p.m., and informal dress will be worn.

The price of the ticket, for the dinner and wine, will be £7.00 (inclusive of VAT), and a cash bar will be provided at the reception and after the dinner. Past Presidents, Past Honorary Officers, Honorary Members and Founder Member have been invited as guests of the Association. All other past and present Members of Council must send the necessary remittance with their completed application form. Any member with service on Council who wishes to attend should write to the Director & Secretary immediately.

Paint Technology Manuals

Part Seven: Works Practice

The seventh volume of the Association's series of Paint Technology Manuals, entitled "Works Practice," is now offered for sale.

The articles which have appeared under Student Reviews each month have been so designed that the bound volume will form a complete, coherent book with full continuity. Apart from the necessary typographical changes in maintaining consistency in the *Journal,* "Works Practice" will be exactly the same as those parts of the Paint Technology Manuals already published by Chapman **&** Hall ltd.

To hold the price of this book at its present low level, it is essential to reduce book keeping to a minimum. Copies of this book will only be supplied if remittance is sent with the order. Accredited Bookseller's discount: 25 %.

Contents include:

To: Oil and Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middx. England HAO 2SF, England.

Please send **...** copies of Paint Technology Manual Part Seven, "Works Practice," @ £3.00 per copy, post free in U.K. I enclose a cheque/P,O. for £.....

Name ..

Address

CLASSIFIED ADVERTISEMENTS

Classified Advertisements are charged at the rate of £2.50 per cm. Advertisements for Situations Wanted are charged at 60p per line. A box number is charged at 50p. They should be sent to the Director & Secretary, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF

SITUATIONS VACANT

WANTED
Shoe Polish Consultant, manufacturing experience essential.

Replies to Box No. 414.

SITUATIONS WANTED

Indian, 33 years, working in USA. Broad experience in paint, printing ink formulation. Knowledge of G.C. and I.R. Also, emulsion and acrylic solution polymer experience. Looking for an opening in India or other parts of Asia. Box No. 410.

Paint technologist HNC, FTC, group leader (additive development), seeks responsible post in England or Wales. Technical sales and/or laboratory work acceptable. Fluent German (sales experience in W. Germany) plus paint formulation and corrosion prevention knowledge. Box No. 412.

MISCELLANEOUS

PAINTMAKERS' ASSOCIATION SYMPOSIUM
The Paintmakers' Association has announced the holding of a Symposium "Powder for Profit" which will take place from Tuesday 28 to Thursday 30 October 1975 at Penns Hall Hotel, Sutton Coldfield, near Birmingham. The Symposium is being organised for the Powder Coatings Group of the Paintmakers' Association of Great Britain Limited by the Conference Division of Wheatland Journals Limited, 157 Hagden Lane, Watford WD1 8LW. The registration fee for three days will be £50.00 plus VAT, and for two days £35.00 plus VAT.

You can send a telex for an advertisement in JOCCA (or in reply to one), at any time of day or night, by dialling:

922670 (OCCA WEMBLEY)

OCCAA '77 PACIFIC PAINT CONVENTION

The Oil and Colour Chemists' Association Australia will be organising its first international Paint Convention at Canberra 25-28 September 1977.

The lecture programme, under the heading "Technical & Management Efficiency in the coatings business" will have two concurrent themes—Paint Technology, and Management as it relates to paint manufacture.

The initial circular has been printed and copies are available from N.M. MacLeod, OCCA Australia, Pacific Paint Convention '77, P.O. Box 93 Punchbowl, 2196, Australia.
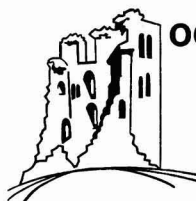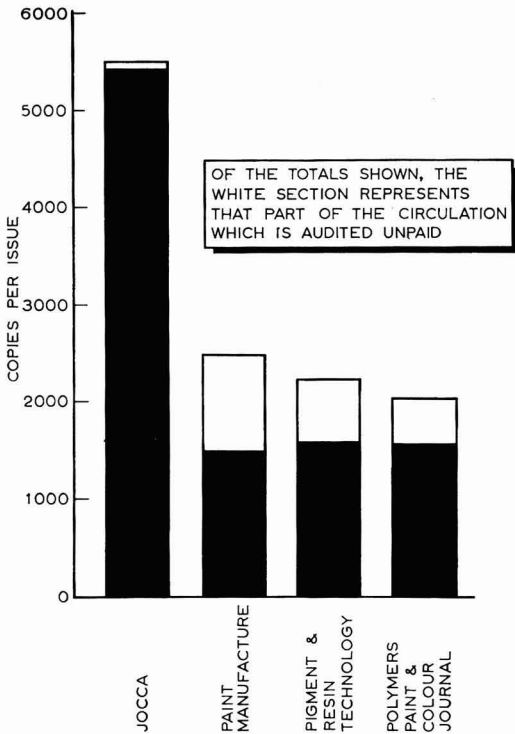
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PRE-PRINTS

Papers given at the Association's Conference 17-21 June under the title "The performance of surface coatings—does the reality match the theory?" are being published with discussions in the *Journal*, and the first appeared in the September issue.

There are, however, a few spare copies of the bound pre-prints available, and these may be purchased at £10.00 per copy (*Prepayment only* to Oil and Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF, England).

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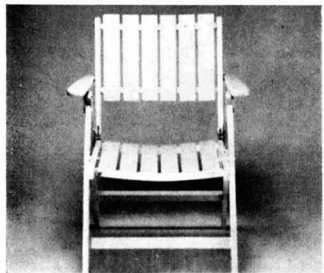**For full details of advertising in this, and other Association publications, contact C. A. Tayler, Assistant Editor**

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