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**IMPORTANT ENCLOSURES ON
FORTHCOMING ASSOCIATION EVENTS**

**SCARBOROUGH CONFERENCE—SUMMARIES
OF PAPERS AND BIOGRAPHIES OF AUTHORS**
(See page 35)

**JOURNAL OF THE
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COLOUR
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The measurement of ultraviolet radiation in accelerated weathering machines
E. Capron and J. R. Crowder

Plasticiser structure in relation to flow properties of polyvinyl chloride melts
R. Khanna

The existence and significance of the Infinitesimal Hardness Behaviour (IHV)
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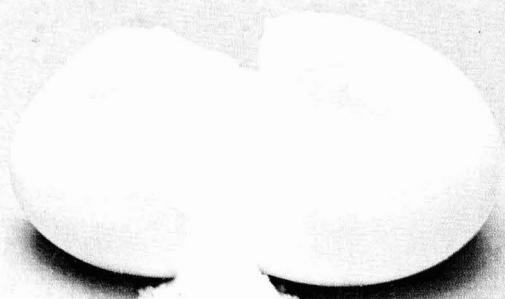
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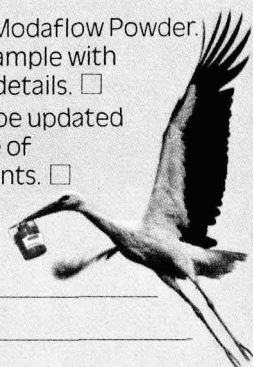
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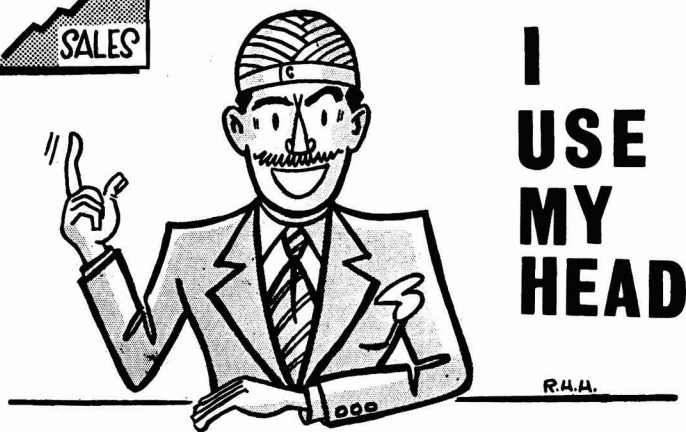
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A personal message from the President



I would like to take this opportunity of wishing all members of our Association, both in the United Kingdom and throughout the world, a Very Happy New Year.

1974 has been a very special year for my wife and myself. We have been fortunate in having had the opportunity of meeting a large number of our members, not only in the United Kingdom but also in New Zealand and South Africa. We both very much appreciated this opportunity to visit our overseas Sections, and I hope that in some small way we have helped to cement the fellowship of OCCA, itself a wonderful tradition, throughout the world.

I would also like to send greetings at this time to members of our sister Societies in America, Europe, Scandinavia, and particularly to our OCCA Australia friends, many of whom we met in Sydney in August 1974. It was a matter of great regret to us that we were unable to visit the OCCA Australia Convention, but unfortunately time did not allow us to take advantage of this opportunity.

The one thing that has come home to me during my period of OCCA presidency is that our Association is unique because of the relationship between our members throughout the world. This is based not just on a technical relationship, but in many cases on personal relationships, which can only add to the opportunities for technological advancement. Our Association is very conscious that if we are to take full advantage of the opportunities which are presented to us in the years ahead, then we must recognise and innovate change in the character of our Association. Council is now exploring ways in which the international nature of our Association can be more formally recognised. It was, indeed, a great delight for me to see the development of our overseas Sections, and the changes which are being examined are designed not only as a recognition of changes in our technical world, but as a compliment to our overseas members who have done so much to make OCCA the truly international Society it has now become.

I believe that primarily, because of circumstances, my job as President of our Association during this period has been to try and lay the basis for sound financial security so that we can move forward and play our part fully in the years to

come, without too much worry as to our immediate financial prospects. An enormous amount of work has been done by the Officers to ensure our financial stability. I believe that we can now see our way ahead in this area, and I sincerely hope that by the end of my presidency the crisis, which was created primarily by industrial problems, raw material shortages, and feed stock price escalations, is behind us, and we may have the opportunity and experience to move forward with confidence and in good heart.

I look forward to 1975, secure in the knowledge that at our Scarborough Conference, when I hand over the reins of OCCA presidency to Arthur Rudram, I am entrusting them to a man whose experience in this Association goes back many years, and who I know will lead OCCA forward in the best interests of its members throughout the world. Undoubtedly there are many problems ahead of us, as indeed there are for the industries in which we are involved, but I believe that we are capable of tackling these problems and surmounting them, and that we will continue to move forward from strength to strength.

I would like to take this opportunity of publicly thanking our Director & Secretary, Mr Robert Hamblin, and all his staff who, under great difficulties, have done a magnificent job during these last few years. To move our offices from the centre of London to the outskirts, to train new staff, and to keep the Association going economically under inflationary conditions was, in itself, a momentous task, and I am sure all members of the Association will join me in thanking Robert Hamblin for the wonderful job he did.

I believe that as an Association we have a great future to look forward to, and we will, unquestionably, play our role fully in the traditional OCCA manner, both nationally and internationally.

A handwritten signature in dark ink, appearing to read "Leske Silver". The signature is written in a cursive style with a horizontal line underneath.

January 1975

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Transactions and Communications

The measurement of ultraviolet radiation in accelerated weathering machines

By E. Capron and J. R. Crowder

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Summary

A miniature self-contained ultraviolet (UV) integrating recorder is described, which has been built to measure the UV radiation received by specimens in two types of laboratory weathering machine. Eleven interchangeable sensing heads, each fitted with a narrow band interference filter, have enabled the spectral output of the UV sources to be determined within the region 300 to 400nm.

Results are presented for laboratory weathering devices based on xenon arc lamps and on combinations of UV-emitting fluorescent

tubes in terms of: (i) the horizontal distribution of radiation; (ii) rotationally integrated spectral distribution; (iii) the effects of different combinations of fluorescent tubes; and (iv) the deterioration in output, over the UV range, of a xenon arc lamp during its service life.

It is suggested that by modifying the arrangement and selection of tubes, a compound source could be developed with a spectrum more nearly matching the UV-component in solar radiation.

Keywords

Processes and methods primarily associated with analysis, measurement or testing
accelerated weathering

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

Miscellaneous
ultraviolet light

La mesure de la radiation ultra violet dégagee dans les appareils de vieillissement accéléré

Résumé

On décrit un intégrateur incorporé pour doser la quantité de radiation ultra violet reçue par les éprouvettes exposées dans deux types laboratoires d'appareil de vieillissement accéléré. Grâce à onze têtes sensibles échangeables dont chaque une porte un filtre pour éliminer une bande d'interférence bien définie, on pouvait déterminer le rendement spectral des émetteurs de rayons ultra violet dans la région 300-400nm.

On présente les résultats dérivés des dispositifs laboratoires de vieillissement, qui comprennent les lampes à arc à vapeur de xénon, ou des combinaisons de tubes fluorescents qui émettent

les rayons ultra violet, selon les rubriques suivantes: (i) la répartition horizontale de la radiation; (ii) la répartition de la radiation spectrale intégrée de rotation; (iii) les effets de diverses combinaisons de tubes fluorescents; et (iv) la diminution du rendement, à travers la portée ultra violet, d'une lampe à arc à vapeur de xénon au cours de sa vie utile.

On suggère qu'en modifiant la disposition et la sélection des tubes on puisse perfectionner une source composée qui posséderait un spectre qui correspond plus exactement au constituant ultra violet de la radiation solaire.

Messung der Ultraviolettstrahlung in Kurzbewitterungsapparaten

Zusammenfassung

Beschreibung eines unabhängig funktionierenden, Miniatur-Ultraviolett integrierenden Registrierapparates, der so konstruiert ist, dass er die von Prüflingen in zwei Arten von Laboratoriums-Kurzbewitterungsapparaten empfangene UV Bestrahlung misst. Mit Hilfe von elf Messköpfen (sensing heads), jeder mit einem Engbandinterferenzfilter ausgestattet, konnte der spektrale Ausstoss der UV Quellen innerhalb von etwa 300 bis 400nm bestimmt werden.

Resultate werden vorgelegt für Bewitterungsvorrichtungen, die auf Xenonbogenlampen und Kombinationen von UV ausstrahlen-

den, fluoreszierenden Röhren wie folgt basieren: (I) horizontaler Verteilung der Strahlung; (II) drehungsweise integrierter spektraler Verteilung; (III) der Auswirkung verschiedener Kombinationen fluoreszierender Röhren; (IV) der Verschlechterung des Ausstosses einer Xenonlampe im UV Bereich während der Dauer ihrer Brauchbarkeit.

Es wird vorgeschlagen durch Modifizierung des Arrangements und der Röhrenausswahl eine kombinierte Quelle mit einem der UV-Komponente in der Sonnenbestrahlung beinahe gleichenden Spektrum zu entwickeln.

Introduction

Refs. 1-11

Accelerated weathering machines are extensively used to simulate the effects of weather on paints, plastics and other organic materials¹⁻⁴. Ultraviolet (UV) radiation is a major factor causing degradation of these materials⁴⁻⁶ and laboratory weathering machines are based on lamps that are intended to simulate solar radiation, in particular its UV component.

The available sources of radiation are known to give only imperfect matches to sunlight^{1, 2}, and also to change their outputs with age^{8, 9}. Until recently, however, there has been no adequate means of characterising or monitoring the radiation received at the surface of samples exposed in accelerated weathering machines, apart from spectrometric measurements of the output from single lamps^{7, 10}. The instrument described in this paper was developed for this purpose and is complementary to the Building Research Establishment (BRE) UV sensor¹¹ on which it is based and

which has been used to monitor the radiation received by samples exposed to natural weathering.

The instrument was designed specifically for use in two machines, the "Xenotest 450"* and the Frigistor "Climatest"† and is unique in being small enough to be fitted in the specimen position in either of these two machines. It rotates with the specimens around the UV source and integrates the varying radiant flux in selected narrow wavelength bands over measured periods of time. It, therefore, provides means of accurate assessment of the radiant energy received by exposed samples, which is a considerable improvement on the current but inadequate practice of describing the radiation received by samples in terms of "hours of exposure".

Matching laboratory UV sources to sunlight

Refs. 6, 12, 13

It is important that the spectrum produced by a UV source in an accelerated weathering machine should be similar to solar radiation, both in terms of intensity and spectral distribution. Maximum UK levels of solar radiation, determined in the middle of a day in mid-summer under clear sky conditions, are taken as the standard; solar radiation curves used in this paper as illustrations relate to such determinations.

There is evidence^{6, 12} in the case of many polymers that specific degradative effects, such as yellowing and fading, are related to definite wavebands within the UV region from 300nm upwards. For example, radiation shorter than 360nm tends to promote the yellowing of polyesters and polyvinyl chloride (pvc). Exposure to wavelengths longer than 360nm, extending up into the visible region above 400nm, has been observed to exert a bleaching effect on plastics and also on other organic materials¹². It has been clearly demonstrated that certain polymers are activated by quite specific narrow wavebands. If the laboratory UV sources do not closely match solar radiation, therefore, differences are to be expected between the effects of natural and laboratory weathering. The fact that none of the currently used sources provides an adequate match is reflected in the limited reliance generally placed on the results of laboratory weathering. To achieve and define any improvement a measuring device, such as the instrument described, is necessary.

Moreover, if regular periods without irradiation are used to simulate the night-day natural cycle in the accelerated weathering machine (this has been found to give improved correlation between natural and artificial weathering with some plastics, in particular, pvc), continued monitoring of UV intensity is desirable to permit comparison of the "total radiation received" in the weathering machine and on the exposure site.

With both the "Xenotest" and the "Climatest" machines, it is particularly important that any measuring device should travel round the radiation source with the specimens, as the output of the source is not uniformly distributed. This facility enables an integrated value for radiation to be determined. In the Xenotest, the radiation is filtered through a "lantern" of different radiation-absorbing glasses. In the Climatest, the radiation source is a combination of a number of fluorescent tubes of different types, the arrangement of which may be altered.

*Manufactured by Quarzlampe GmbH, Hanau, W. Germany.

†Manufactured by Mectron Ltd., Slough, England.

The UV recorder

Design

The recorder was designed as a self-contained self-powered instrument, and the available space limited the external dimensions to 245 × 115 × 55mm. Additional requirements were the ability to function unattended for long periods at relatively high temperatures, and at high humidities; in addition sensing heads for the different wavelength bands should be easily interchangeable and should be capable of movement in the vertical plane to permit examination of variations in intensity at different points on the exposed specimens.

A rear view of the recorder is shown in Fig. 1, which identifies the main components. A front view would merely show the aluminised surface containing the silica window with a sensing head fixed in it. Figs. 2 and 3 show the instrument installed in the Xenotest 450 and the Climatest, respectively. Fig. 4 is a block diagram of the circuit arrangement and Fig. 5 shows the construction of one of the interchangeable sensing heads: the only difference between the heads is the passband of the interference filter.

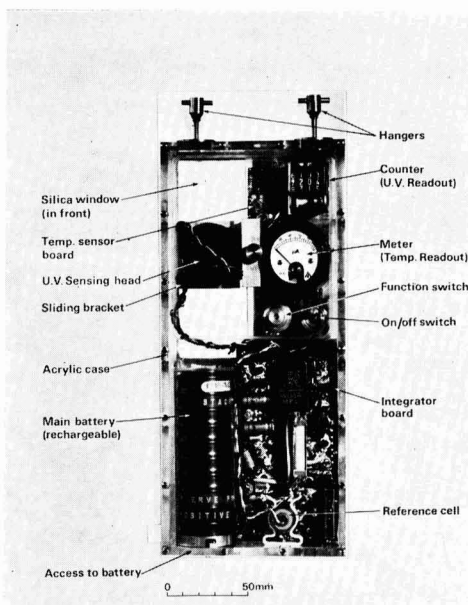


Fig. 1. The complete instrument—rear view

Eleven sensing heads were made, spanning the UV range from 300-400nm in approximately 10nm intervals; the average half-peak band width is about 2nm. The design is similar to that of the BRE UV Sensor¹¹, with diffuser, filter, phosphor and photocell, although it was not considered necessary to hold the optical components at constant temperature, as in the field version, since the environment in use would provide this facility. Instead, a built-in thermometer was incorporated in each head and readout provided in the main instrument. (The sensing head is very similar to that which is covered by UK Patent Application No. 17511/72.)

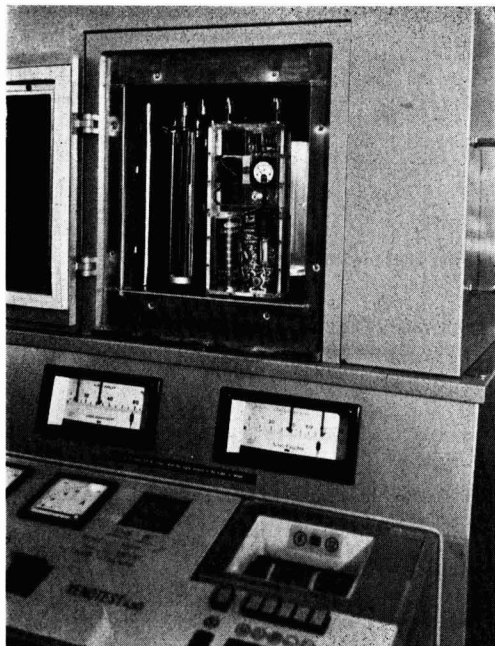


Fig. 2. The instrument installed in the Xenotest 450

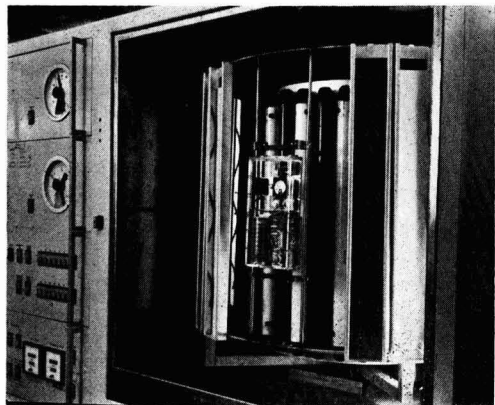


Fig. 3. The complete instrument installed in the Climatext

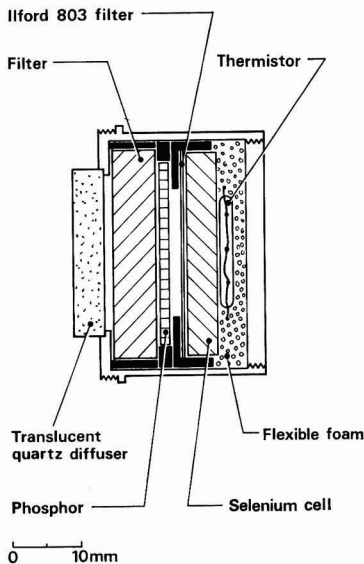


Fig. 5. Section through sensing head

The sensing head thermometer

Both the output of the selenium cell and the conversion rate of the phosphor are temperature-sensitive. The head thermometer consists of a small glass-bead type thermistor cemented to the rear face of each selenium cell and connected to an appropriate circuit. The temperature can be determined after calibration from the scale reading on a miniature microammeter (Fig. 1). The reference voltage is provided by a 1.35V mercury cell. In service, the UV recorder attains thermal equilibrium in about one hour.

The integrator

This was obtained commercially, and incorporates a high-gain amplifier which converts the millivolt DC output from the sensing head to pulses at a frequency proportional to the incident radiation. The pulse count is registered by a miniature electro-mechanical 4-digit counter. Integrator and counter are powered by a rechargeable 12V 1.8kC DEAC battery which gives about five days' running at a count rate of 100 cycles per hour.

Thermal gain

The considerable thermal gain of the recorder has been reduced to an acceptable level by aluminising the rear surface of the front acrylic panel for maximum reflection of IR, and painting the case of each sensing head matt-black to re-radiate excess heat. Thus protected, the instrument may be run at temperatures not exceeding 45°C in the Xenotest and this is well within the safety limit.

Calibrations

The linearity of the BRE UV sensor has been established¹¹; check calibrations (Fig. 6) with the sensing heads of the present instrument, operating at constant temperature,

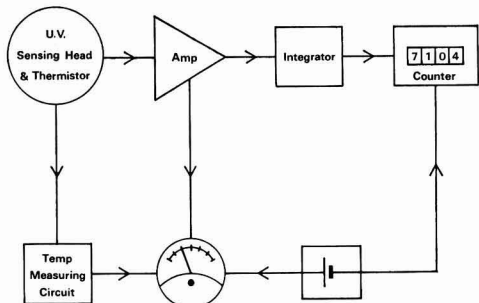


Fig. 4. Block diagram of instrument

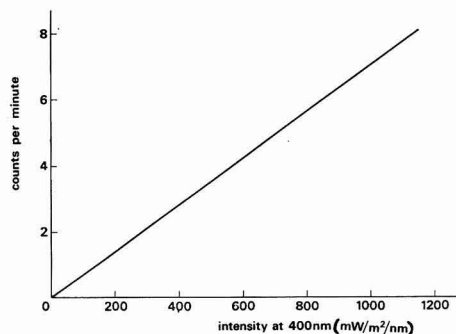


Fig. 6. Calibration curve for 400nm sensing head—showing the variation of output with ultraviolet light intensity at constant temperature (40°C)

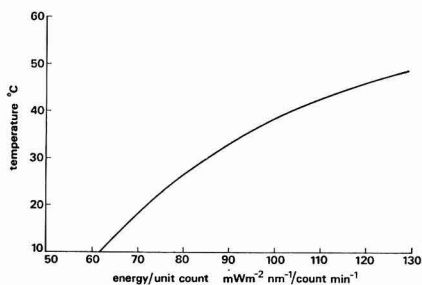


Fig. 7. Temperature calibration curve for 350nm sensor

showed good linearity. Each head was also calibrated for a range of temperatures at constant illumination from a tungsten halogen lamp, using the instrument's in-built temperature-measuring circuit. The lamp output was determined at each wavelength, using the technique developed by Harris¹¹ and based on the ferri-oxalate method of Hatchard and Parker¹³, and was used for comparison of the integrator output. A typical calibration curve is shown in Fig. 7.

Results

Horizontal distribution of radiation

Refs. 14, 12

The horizontal distribution of radiation¹⁴ from weathering machine sources is not uniform, in either the intensity or spectral content. For example, in the Xenotest 450, the xenon arc is surrounded by a lantern comprising six sheets of heat absorbing glass (KG1) and one sheet of short-wave UV transmitting glass (UG 11). Some preliminary instantaneous measurements of intensity, at three wavelengths (315, 350 and 400nm) were made, using a modified BRE UV sensor, and these are presented graphically in Fig. 8. This shows a marked reduction in 400nm radiation opposite the UG 11 filter glass, and a small corresponding increase in 315nm radiation.

Measurements of horizontal distribution were also made in the Climatest with the following fluorescent tube lamps:

- A "Black lamps" TL 20W/08: 16 tubes
- B "Sun lamps" TL 20W/12: 8 tubes
- C "Trucolor" TL 20W/37: 8 tubes

arranged in order $-A-B-A-C-A-$ etc round the drum.

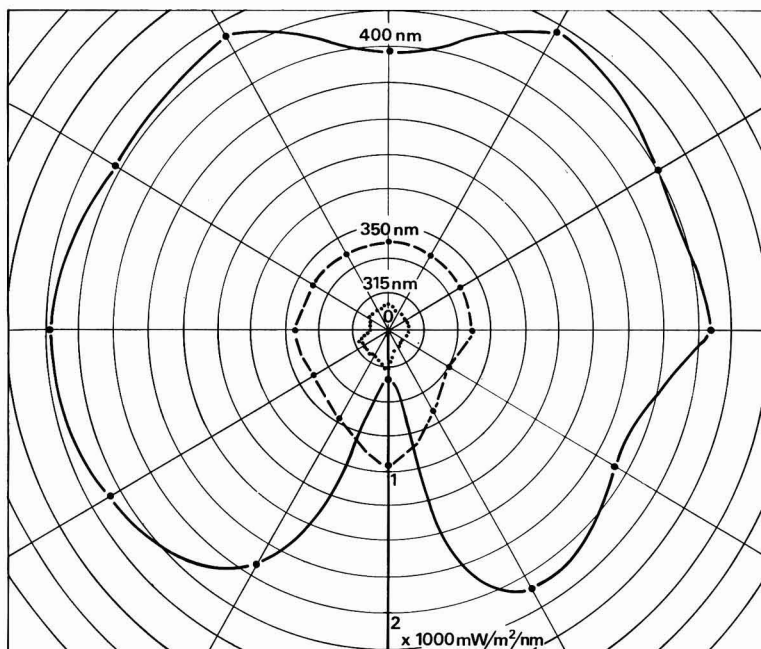


Fig. 8. Horizontal distribution of radiation in Xenotest 450

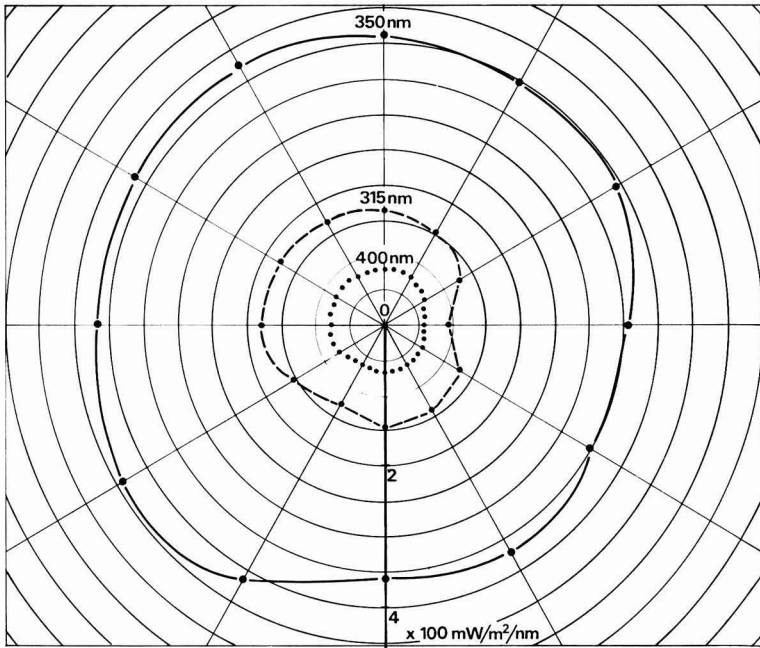


Fig. 9. Horizontal distribution of radiation in Climatest

Pairs of tubes were changed in rotation every 100 hours and, with 32 tubes in the machine, each tube remained in service for 1 600 hours. The ageing of lamps which takes place over this period accounts for the asymmetric distribution of intensity, illustrated¹² in Fig. 9. Aged and new lamps were adjacent to one another at the position corresponding to 7 o'clock in the diagram; the output at 3 o'clock is lower than would be expected, probably because of tube-to-tube variability.

Rotationally integrated spectral distribution

The effective spectral distribution, as received by rotating specimens, was determined using the UV-integrating recorder and is illustrated in Figs. 10 and 11 for the Xenotest 450 and the Climatest, respectively. Each point shown on the curves is a mean of five determinations, each of which was made over a ten-minute period of rotation of the recorder around the UV source. Comparison is shown with spot measurements of solar radiation made on a cloudless day in midsummer. It can be seen from Fig. 10 that correspondence is good between 300 and 340nm, but at longer wavelengths, that is the "fading" wavelengths, output from the Xenotest is relatively too high.

Curve 1 in Fig. 11 was obtained with the arrangement of tubes in the Climatest as originally supplied, that is 16 "Black lamps" and 16 "Sun lamps" placed alternately round the drum. Other combinations of tubes were investigated and Curve 2 shows the closer correlation of output

obtained with 8 "Sun lamps" and 24 "Actinic lamps" (TL 20W/05) arranged symmetrically. The match with solar radiation is still some way from that which is desirable, being deficient in the regions 320 to 340nm and above 380nm, but the curves do illustrate how determination of output is possible with the integrating recorder.

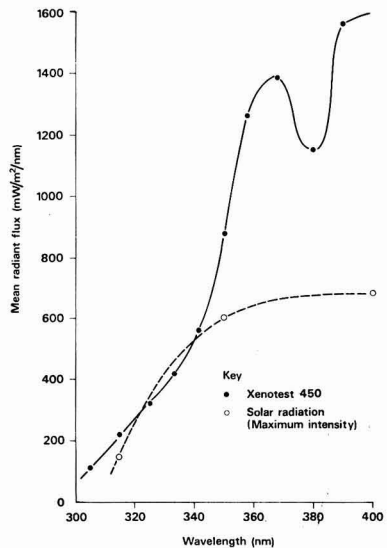


Fig. 10. Spectral distribution rotationally integrated: Xenotest 450

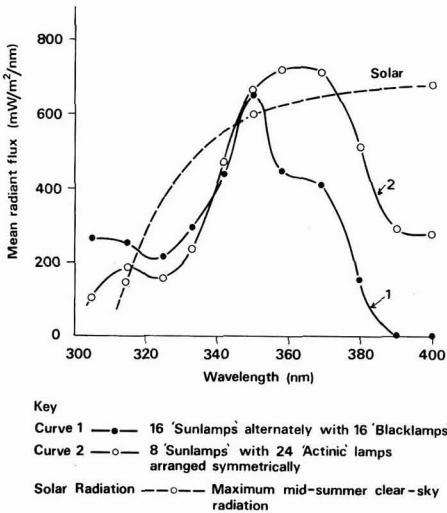


Fig. 11. Spectral distribution rotationally integrated: Climatest

Ageing of xenon arc lamp

Refs. 8, 9

Reference has been made already to the decline in output of a xenon arc throughout its operational life. The Xenotest manufacturer has stated that it is not advisable to use lamps for more than 1 500 hours, although indicating⁸ that change in spectral energy distribution up to this period will not cause falsification of the test result. However, Campbell⁹ has demonstrated that during operation the UV output of this type of xenon arc may fall by 48 per cent at 350nm and 63 per cent at 300nm.

Using the UV integrating recorder, the output of the 4.5kW xenon arc was determined in the Xenotest 450, first when almost new (100 hours) and later when, according to the manufacturer's recommendations, it should be discarded (1 500 hours). Fig. 12 compares output at the two stages and Fig. 13 relates the percentage reduction in output to UV wavelength. At 400nm the fall-off was about 25 per cent,

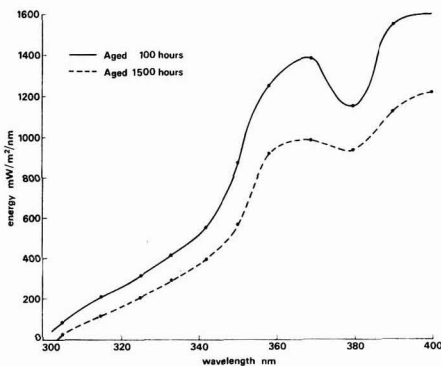


Fig. 12. Comparison of a new and aged Xenotest 450 radiator—corrected to 4.5kW

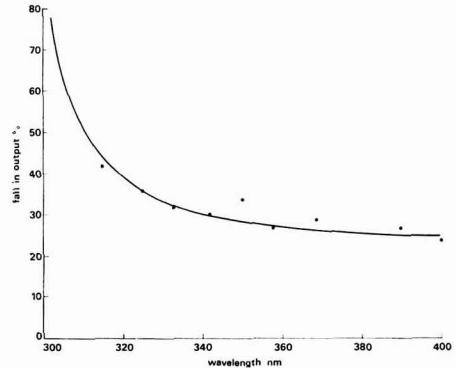


Fig. 13. Percentage reduction in output at various wavelengths: Xenotest 450 radiator after 1500 hours ageing

rising to 60 per cent at 305nm. The Pyrex glass cylinder which surrounds the xenon arc had been well aged for many thousands of hours before the measurements were made, and it is unlikely that a significant proportion of the fall-off was due to its "solarisation".

Discussion

Ref. 10

Neither in the Xenotest 450 nor in the Climatest do specimens receive radiation which closely matches the solar spectrum throughout the UV region, either in intensity or spectral distribution.

The relatively high intensity of longer wavelength UV radiation from the xenon arc is accentuated as the lamp ages and thus there is a tendency to exaggerate the fading action compared with sunlight. The Xenotest equipment is not manufactured specifically for use with plastics or paints and, for example, it appears highly suitable for use in the textile industry, where it is widely used for accelerating the fading of dyestuffs.

On the other hand, the best combination of fluorescent tubes so far found for the Climatest is deficient for "fading" wavelengths, but produces more radiation shorter than 300nm than occurs with sunlight and, therefore, tends to cause excessive yellowing and polymer degradation. Although fluorescent tubes are subject to ageing, a more nearly constant level of radiation is made possible by scheduled replacement of aged tubes. It should be emphasised that this source can be tailored to meet requirements for spectral energy distribution. The system would probably be further improved by increasing the proportion of tubes that emit strongly at the blue end of the visible spectrum. A combination of one "Sun lamp" with one each of two different "Black lights" has also been suggested recently¹⁰, as providing the best simulation of the spectral distribution of sunlight in the UV region. Because of the deficiency of this source in longer wavelength UV and visible emission compared with sunlight, it is not recommended for testing the stability of materials predominantly photosensitive to wavelengths in these regions. The measuring instrument that has been described provides a means to determine the effects of any changes in the lamp arrangement.

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Plasticiser structure in relation to flow properties of polyvinyl chloride melts*

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Summary

Plasticisers are used in polyvinyl chloride formulations to facilitate processing and to improve the low temperature flexibility of a system. Both thermodynamic and kinetic effects have been studied;

it is shown that kinetic effects predominate and that these control the flow behaviour.

Keywords

Raw materials:

binders (resins, etc)
vinyl chloride resin

paint additives; plasticisers
plasticiser

Properties, characteristics and conditions primarily associated with materials in general

consistency
dilatancy
plasticity
rheological property
viscosity

Le rapport entre la structure du plastifiant et les caractéristiques d'écoulement des mélanges fondus au chlorure de polyvinyle

Résumé

On utilise les plastifiants en mélanges au chlorure de polyvinyle afin de faciliter le confectionnement du système et en même temps d'améliorer sa souplesse à basse température. On a étudié à la

fois les effets thermodynamiques et cinétiques; on a démontré que les effets cinétiques exercent une influence prédominante et qu'ils déterminent les caractéristiques d'écoulement.

Weichmacherstruktur bezogen auf Verlaufeigenschaften von Polyvinylchloridschmelzen

Zusammenfassung

Weichmacher werden in Polyvinylchloridrezepten verwandt, um die Verarbeitung zu erleichtern und die Elastizität eines Systems bei niedrigen Temperaturen zu verbessern. Sowohl thermody-

namische, als auch kinetische Auswirkungen wurden untersucht; es wird aufgezeigt, dass die kinetischen Auswirkungen überwiegen, und dass diese das Verlaufsverhalten kontrollieren.

Introduction

The choice of type and amount of the plasticiser for a formulation is based not only on the specific processing conditions to be used, but also on the properties required in the product. The present investigation was carried out in order to seek a better understanding of the flow properties of plasticised pvc, and it covers the use of a range of plasticisers of different characteristics. It is concerned firstly with the practical value of such concepts as solubility parameter, hydrogen bonding, etc, and their relation to melt viscosity. Secondly, it attempts to relate plasticiser properties to the way in which they behave in pvc. Finally, it is hoped that a better understanding of the mechanisms of plasticiser interaction will lead to the more effective formulation of new grades of pvc.

Premixes of the polymer and appropriate ancillary ingredients were milled in a Henschel mixer at 150 to 160°C for 15 minutes. Detailed compounding conditions have been described by the author¹. The Banbury mixer stage was deliberately avoided in an attempt to eliminate degradation and permit more freedom in the investigation for interaction effects on melt viscosity.

Rheological measurements

Viscosity measurements on the plasticisers were made using an Ostwald capillary viscometer. Measurements on the compounds at very low shear rates were made in a rotational cone and plate instrument. The high shear rate studies were carried out in a capillary rheometer at 130°, 150° and 170°C over a range of shear rates from 1 to 1000sec⁻¹.

Dilatometric measurements

The effect of the plasticiser on the glass transition temperature T_g was measured by dilatometry. The volume expansion was recorded over a temperature range from 22° to 160°C.

Fracture surface studies

Pvc specimens were subjected to various heat and shear treatments in a Brabender Plastograph, then fractured by cooling in liquid nitrogen. The fracture surfaces were then studied by means of a scanning electron microscope. This

Experimental

Material and sample preparation

Refs. 1, 2

The basic composition was Corvic D60/11, 100 parts by weight (w/w); tribasic lead sulfate 4w/w. All plasticisers were added at a level of 30w/w and those studied were tricresyl phosphate (tcp), dioctyl phthalate (dop), trioctyl phosphate (top), dioctyl sebacate (dos), and dimethyl phthalate (dmp).

*Paper based on Phd thesis submitted to Brunel University.

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instrument, although inferior in resolution to the transmission electron microscope, is invaluable for the study of irregular surfaces because of its greater depth of focus.

A further study of polymer-plasticiser interaction using optical microscopy was made by an extension of the technique first described by Anagnostopolous and others.² Each pvc plasticiser system was examined by transmitted light hot-stage microscopy at a magnification of $\times 32$. A single very small piece of pvc was heated in the presence of excess plasticiser to increase the temperature at a rate of 10°C per minute to 30° below its previously determined melting temperature T_m . The heating rate was then reduced to 1°C per minute for an accurate determination of T_m (Fig. 1). T_m was found to vary with the plasticiser but to be reproducible within $\pm 3^{\circ}\text{C}$. From the values of T_m and the volume fraction of plasticiser V_1 at the temperature T_m the Huggins-Flory constant χ was calculated.

Results and discussion

Ref. 1

A great deal of the theoretical and experimental work on the rheology of other thermoplastic materials does not apply to pvc because the latter does not readily form a true melt, and memory of the original particle structure is difficult to erase. A structure for rigid pvc melts has been proposed already¹, and it is suggested that a similar mechanism obtains for plasticised materials, although it is clear that plasticiser will facilitate the breakdown of particles at lower temperatures. Both particle/particle slip and molecular deformations are involved in the flow mechanism of plasticised pvc, depending on the compounding conditions. The effect of compounding at different temperatures is summarised in Fig. 2.

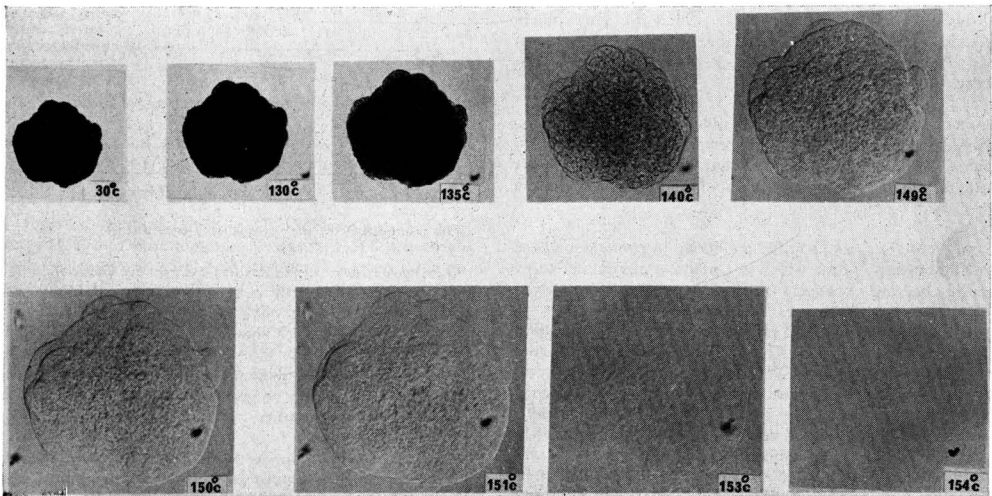


Fig. 1. Photomicrograph showing the effect of heating a single pvc particle in dioctyl sebacate

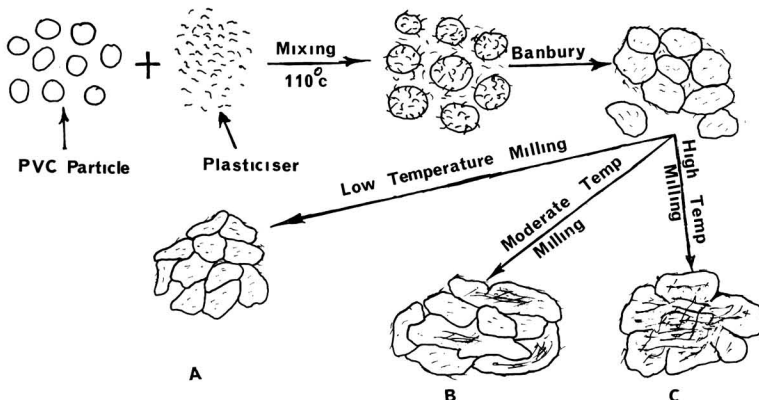


Fig. 2. Structure of a plasticised pvc compound

Structure of the plasticiser molecule and its relation to viscosity

Ref. 3

The principal function of the plasticiser is to interpose itself between the polymer chains and effectively reduce the inter-molecular forces. Opposing these are the forces of attraction between the polymer molecules, which depend on the chemical and physical structure of the pvc polymer. The forces between the plasticiser molecules themselves and between plasticiser and polymer are equally important; plasticising action occurs most effectively only when these three interactions are of the same order of magnitude.

A number of theories have been advanced on plasticiser action. The most significant of these appears to be the mechanistic theory originally due to Doolittle³, and this has formed the basis of much of the work in this investigation.

There are many types of forces acting between molecules, but the most important and those which are particularly applicable to plasticiser/polymer interaction are dispersion forces, dipole-dipole forces, hydrogen bonds and induction forces.

In pvc each chlorine atom is relatively "rich" in electrons. This makes the adjacent carbon atom and the hydrogen atom attached to it slightly positive, so that the -CHCl- group as a whole represents a dipole orientated perpendicular to the axis of the molecule.

Many materials have a softening effect on pvc when added in small amounts. Those which are effective plasticisers show a good balance between solvent power, compatibility, efficiency and permanence. They include many esters of phthalic, adipic, sebacic and phosphoric acids. The molecules of these plasticisers contain one or more polar groups and several non-polar groups. When considering the chemical composition of plasticisers, most importance is often attached to the polar section of the molecule. The non-polar part, however, is worthy of equal consideration. It is important to distinguish between two different types of non-polar component (a) polarisable, such as the benzene ring, (b) non-polarisable, such as aliphatic groups. When the benzene ring is near to the polar group it becomes polarised by induction and greatly increases the effectiveness of the polar group, as in tcp.

An aliphatic group is non-polar and non-polarisable and so is unaffected by polar forces. For example, in "top" the dipole of the plasticiser molecule is surrounded by three bulky aliphatic groups which do not participate in the polar forces of attraction. The plasticiser dipole, therefore, can exercise polar forces in one direction only; the other direction is blocked. An analysis of the structure of the commonly used plasticisers shows that they contain three components (Fig. 3) which can be combined in the eight ways set out in

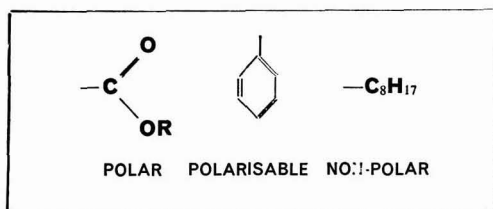


Fig. 3. Molecular components

Table 1. This shows that all the plasticisers which have proved useful in practice contain at least one polar and one polarisable group (combination 1), or one polar and non-polar group (combination 2), or one polar, one polarisable and one non-polar group (combination 3). The plasticisers with poor compatibility are referred to as extenders (combinations 4 and 5).

Table 1
Analysis of the structures of commonly used plasticisers

Combinations	Molecular component*			Example
	Polar	Polarisable	Non-polar	
1	P	P	A	tcp, dmp, diphenyl phthalate
2	P	A	P	top, dos, dioctyl adipate
3	P	P	P	dop, diop
4	A	P	A	With poor compatibility, extenders benzyl-naphthalene,
5	A	P	P	n-butyl, stearate, chlorinated paraffin, dimethyl oxalate
6	P	A	A	Not suitable as plasticisers
7	A	A	A	
8	A	A	P	

*P = present; A = absent

The mechanism of pvc-plasticiser interaction

Materials whose molecules are exclusively polar or non-polar are incompatible with pvc, since they associate with one another and "sweat out". Both the polar and non-polar groups are important. The polar groups are attracted to the pvc dipoles and the non-polar components increase the distance between the pvc chains. Plasticisers rich in aromatic groups enable pvc to be gelled by heating alone, and have a low solution temperature.

Since gelation comprises the diffusion of the plasticiser into the pvc for the purpose of obtaining a homogeneous dispersion, a plasticiser with good gelling properties must diffuse quickly. It must also be capable of penetrating between the mutually attracting pvc dipoles. The diffusion of some of the plasticisers in pvc is illustrated in Fig. 4 which refers to experiments such as those illustrated in Fig. 1. Plasticisers containing an aromatic ring, such as tcp and dop, start diffusing rapidly into pvc at an around T_g (86°C). With aromatic plasticisers saturation was reached at the relatively low temperatures of 105°C and 117°C for tcp and dop, respectively. Fig. 4 shows that with aliphatic plasticisers, such as dos and top, the diffusion starts at a lower temperature (50°C), but it is slower and more prolonged and, therefore, the saturation point is attained only at about 150°C.

The combination 2 in Table 1 covers plasticisers whose molecules are composed of polar and non-polarisable groups, that is top. The dipoles of such plasticisers become attached to pvc dipoles whilst the aliphatic parts of the molecules act as a shield and mechanically prevent other pvc dipoles from approaching. The longer the aliphatic chain the greater will be its shielding effect. The plasticisers in this group are more effective in lowering T_g , although gelation requires a higher temperature and the solution temperature is high (53-55°C).

The third combination in Table 1 contains plasticisers such as dop and dialphanol phthalate with structures intermediate

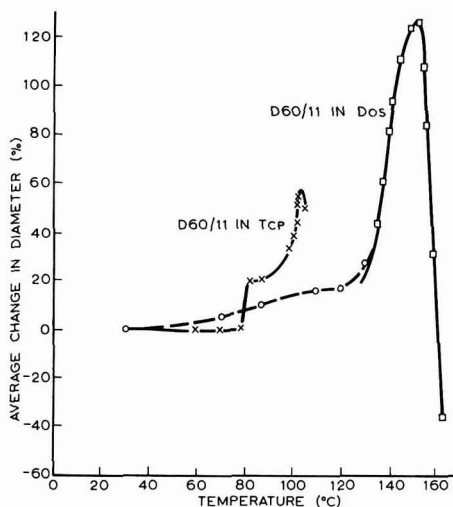


Fig. 4. Change in pvc particle diameter with temperature

between aromatic and aliphatic plasticisers. Materials containing these compounds show correspondingly intermediate properties.

The effect of plasticiser type on the swelling ratio of pvc extrudate at 170°C over a range of shear rates from 10 to 1000 sec⁻¹ is shown in Fig. 5. A high swelling ratio occurs with tcp. Aliphatic plasticisers, such as dos and top, do not gel pvc so effectively, and give low swelling ratios. Dop, which is composed of an aromatic ring and a short aliphatic chain, gives intermediate swelling. This suggests a correlation between swelling ratio and ease of gelation.

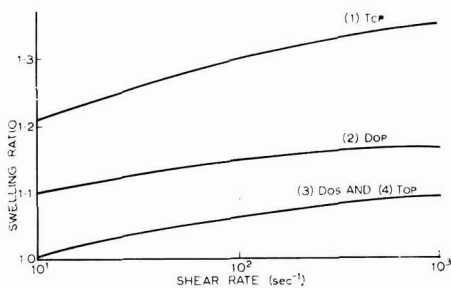


Fig. 5. Swelling ratio of plasticised pvc compounds

Solubility parameter: Huggins-Flory interaction constant

Refs. 4, 5

The solubility parameter, a quantity defined by Hildebrand⁴, is useful as a rough system of classifying materials and it gives a first approximation to solvent power.

The Huggins-Flory treatment⁵ is a somewhat more refined thermodynamic analysis of solvent power than is given by the solubility parameter. The treatment is applicable to pvc-plasticiser systems and leads to some interesting observations, although it does not succeed in explaining the viscosity or extrudate quality of plasticised pvc compounds.

According to Flory's theory⁵, a plot of the reciprocal of the depressed melting temperature $1/T_m$ against the quantity $(\theta_1 - \chi\theta_1^2)/V_1$ (where V_1 is the molar volume of the diluent and θ_1 is the volume fraction of the diluent at the depressed melting temperature T_m) should be linear for all pvc-plasticiser systems, provided the heat of fusion δH_u has a constant value independent of the type of diluent used. The results shown in Fig. 6 are consistent with the theory. The theory also suggests that the parameter χ , the Huggins-Flory interaction constant, has a definite physical significance. It has been shown, both theoretically and experimentally, that a value of χ of about 0.55 is the dividing line between poor solvents and non-solvents. The region of poor solvents power extends from 0.55 down to 0.31 and values of χ less than about 0.30 indicate good solvents. In general, therefore, the smaller the value of χ the stronger the polymer-diluent interaction and hence the better the solvent.

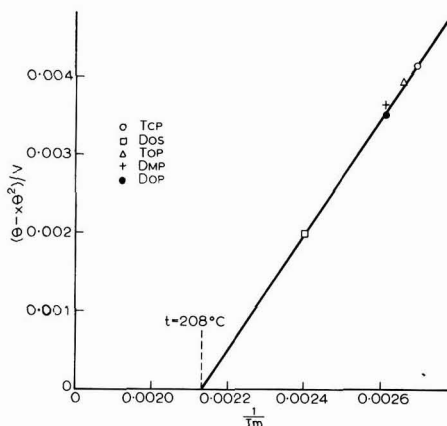


Fig. 6. Plot of $(1/T_m)$ against $(\theta_1 - \chi\theta_1^2)/V_1$ for various plasticised pvc compounds

Kinetic and thermodynamic effects and activation energy

Ref. 6

The thermodynamic character of an interaction between pvc and a plasticiser can be expressed by the Huggins-Flory interaction constant. The kinetic character, however, depends largely on the shape and size of the molecules.

According to Bueche's theory of polymer flow,⁶ the effect of the plasticiser on melt viscosity is given by the product of two terms, one of which depends on the kinetic properties of the diluent, and in particular on its own viscosity, and secondly by one which could depend upon the thermodynamic interaction between the polymer and the diluent, the latter being measured by the Huggins-Flory interaction constant χ . The kinetic effects can be measured by determination of the activation energy. Fig. 1 shows that plasticiser molecules diffuse into the pvc particle and disrupt the polymer structure by separating the points of contact. Plasticisation and dissolution of a pvc particle are slow processes; energy is needed to break up the particle and separate the dipoles. The process may be characterised, therefore, by an activation energy.

The activation energy for viscous flow was measured for plasticisers and for compounds by viscosity measurements at a range of temperatures. A plot of log viscosity (at a fixed shear stress) against the reciprocal of absolute temperature

gives a line from the slope of which the activation energy can be calculated (Fig. 7). Values are listed in Table 2. It is clear that for the limited range of materials studied, the activation energy of a plasticised pvc melt correlates with that of the plasticiser. The latter also correlates with the viscosity of the plasticised pvc compound. Dimethyl phthalate is an exception, probably due to its high volatility or to its low molecular weight. The temperature dependence of viscosity of plasticised pvc and the corresponding plasticisers are in the same order (Table 2). Swelling ratio, elastic shear modulus and the number of links per unit volume of pvc melts also correlate with the activation energy of plasticisers or the plasticised compounds. Good extrusion quality is favoured by a low activation energy for the plasticiser.

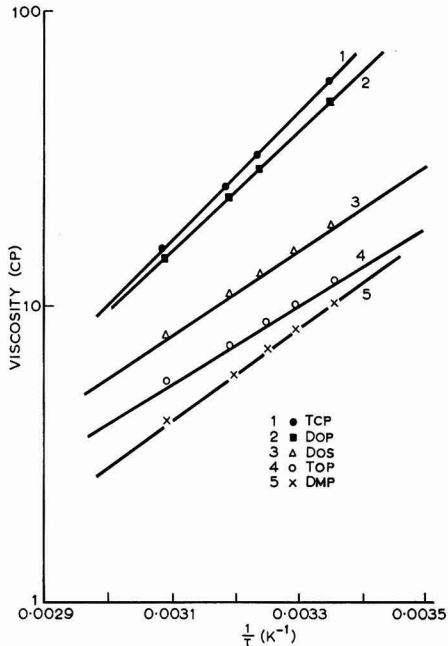


Fig. 7. Viscosity versus $(1/\text{temperature})$ rheograms for various plasticisers

Conclusions

Both the thermodynamic and kinetic effects are important in pvc-plasticiser systems; the former, which can be expressed by the Huggins-Flory interaction constant χ is only partially successful in predicting the viscosity behaviour of plasticised pvc melts, but it has been found that kinetic effects predominate and control the flow behaviour.

A larger number of plasticisers should be investigated to obtain a comprehensive picture, but the results within a relatively limited range of materials has shown that the viscosity of a plasticised compound correlates with the activation energy of the plasticiser, with the exception of dimethyl phthalate. In addition, the temperature dependence of the viscosity of a plasticised compound correlates with that of the plasticiser.

There appears to be a good correlation between swelling ratio and ease of gelation, which itself depends on the structure of plasticisers.

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Table 2

Viscosity/temperature data used for plotting graph shown in Fig. 7

Plasticiser	Activation energy		Viscosity of compound at 130°C , 10sec^{-1}	Rheological parameters at low shear rates 150°C . Constant shear stress = $7.36 \times 10^3 \text{ Nm}^{-2}$			
	of plasticiser	of compound at shear rate 10sec^{-1}		Shear rate $\dot{\gamma}$	Elastic modulus G	Viscosity	No of mol links per m^3
Top	0.496kJ kg^{-1}	3.76kJ kg^{-1}	$1.1 \times 10^6 \text{ Ns/m}^2$	$3.44 \times 10^{-5} \text{ sec}^{-1}$	$1.47 \times 10^5 \text{ N/m}^2$	$2.14 \times 10^8 \text{ Ns/m}^2$	1.27×10^{25}
Dop	0.418	2.68	6.2×10^5	3.92×10^{-5}	1.31×10^6	1.98×10^8	1.12×10^{25}
Dos	0.276	2.23	5.8×10^5	5.73×10^{-5}	1.28×10^6	1.28×10^8	1.09×10^{25}
Top	0.254	2.16	4.4×10^5	5.73×10^{-5}	1.12×10^6	1.28×10^8	0.96×10^{25}
Dmp	0.648	2.37	1.6×10^6	1.15×10^{-5}	1.89×10^6	6.42×10^8	1.60×10^{25}

The existence and significance of the infinitesimal hardness behaviour (IHV)*

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Summary

Interest in the determination of the elastic modulus of the surface of a firmly adherent coating by the assessment of the Infinitesimal Hardness Behaviour (IHV) has greatly increased in recent years. It has, however, met with some objections.

Keywords

Equipment primarily associated with analysis, measurement or testing

indentation hardness tester

This paper shows, both experimentally and mathematically, that the IHV value represents a true characteristic of materials. Some further examples are given to illustrate the significance of the IHV value.

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

elastic modulus
infinitesimal hardness behaviour

La réalité et la signification des caractéristiques de la dureté à l'infini, (IHV)

Résumé

Au cours des années récentes on a mis en évidence un intérêt forte augmenté à la détermination du module d'élasticité de la surface d'un revêtement solidement adhérent au support en effectuant l'appréciation des caractéristiques de la dureté à l'infini, (IHV). Toutefois, on a formulé certaines objections.

Cet exposé montre à l'aide des résultats expérimentaux et en même temps au point de vue des mathématiques que les valeurs IHV sont en effet une caractéristique réelle des matériaux. On donne quelques exemples supplémentaires afin de démontrer la signification des valeurs IHV.

Das Bestehen und Die Bedeutung des Infinitesimalen Härteverhaltens (IHV)

Zusammenfassung

Die Ermittlung des Elastizitätsmoduls der Oberfläche einer festhaftenden Beschichtung durch Bestimmung des Infinitesimalen Härteverhaltens (IHV) hat in letzter Zeit grösseres Interesse gefunden. Es sind aber auch Einwände erhoben worden.

Vorliegende Arbeit soll nun experimentell und mathematisch zeigen, dass der IHV-Wert eine reelle Charakteristik der Werkstoffe darstellt. Einige weitere Beispiele von Aussagen des IHV-Wertes werden angeführt.

Introduction

Refs. 1-5

At the meeting of the Study Groups II and III of the Swiss Association for Varnish and Paint Chemists (SVLFC) on 21 August 1973, Eprecht presented a paper on "Hardness measurements on paints by the indentation method" and the present author on "Existence and significance of the Infinitesimal Hardness Behaviour (IHV)". The lecturers had opposing objectives, the denial and the proof of the existence of the IHV value.

The curves obtained for the deeper penetration of the indenter into a film are less relevant, because the layers in the deformed area become transformed by the enormous specific surface pressure to such an extent that they no longer represent the original resin under test. Conclusions drawn from the curves for such indentations must be regarded, therefore, with great caution.

The present paper refers to the second of these lectures and deals with the concept and physical meaning of the IHV value, in order to prove subsequently on the basis of epitaxy measurements (see page 24) that the IHV has a positive numerical value, which is characteristic of the surface layers of coatings. The characteristic significance and the many possible practical uses of the IHV value are known from earlier investigations.

During the years 1964 to 1968, Study Group II of SVLFC undertook a study of the "penetration hardness" measurements of paint films on metal substrates. Brugger and Landolt¹ performed penetration measurements on films applied to metal substrates using the classical method. (Test samples supplied by CIBA, Basle.) The curves for the penetration depth y of a variably loaded Vickers pyramid indenter are shown in Fig. 1. The penetration depth for such an indenter into a film, for the case where the measuring period is kept constant, is related to the load applied F by the expression:

$$y = f(F)_t$$

Single values of the deeper penetrations of an indenter (sphere, cone, pyramid) into a resinous layer are highly dependent on the operating parameters and are, therefore, of far less value for the assessment of the characteristics of a surface layer. Mathematical formulae derived for the deeper penetration of an indenter cannot be used for the purely elastic, reversible deformation in the region of the IHV values.

and y was found to depend upon such parameters as the form of the indenter, the duration of the loading, the film thickness and so on. This dependency on experimental parameters means it is difficult to obtain any real insight into the structural behaviour of the materials investigated and Oesterle² sought for some other, less parameter-dependent method of expressing the characteristics of the surface of the film,

*This paper first appeared in German in *Farbe und Lacke*, 1974, 80, 9, by whose permission it is reproduced here.

perhaps of the film structure itself, or a kind of hardness behaviour. This led to the concept of the Infinitesimal Hardness Behaviour (IHV), first described by Oesterle² in 1966 and reported in greater detail by Oesterle, Brugger and Landolt³ at the FATIPEC Congress in 1968. In later work³⁻⁵ it was shown how many varied aspects of film properties, such as structure, resistance and behaviour, could be deduced from suitably performed IHV measurements (for example, elastic modulus, interactions between pigment and binder, internal stress in the film, phenomena of water or contamination occlusion in films, diffusion behaviour, early detection of the breakdown during weathering under normal conditions and so forth).

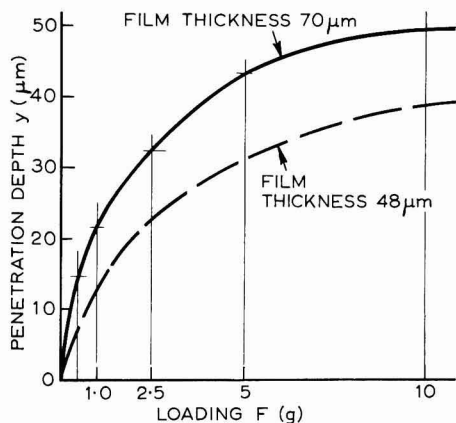


Fig. 1. Penetration measurement with the Wallace micro-indentation tester H7 (W. Landolt, 1966)

Vickers pyramid $w = 136^\circ$; primary weight 0.5 and 0.1g; temperature of measurements 22 to 23°C; loading time 20s; paint Plusol/Cibamin, pigmented white; hardening 30 minutes at 80°C

Nevertheless, until quite recently opinions were expressed that the IHV value did not exist in reality, but that it resulted from a false extrapolation of the measured data.

In this paper, the principles of the IHV method will be repeated briefly and followed by the mathematical and physical justification of the existence of the IHV value.

Final approach to the IHV value

Ref. 6

The IHV method arises from a consideration of the disturbing and decreasing effects initiated in the material when a plastic or paint film is increasingly penetrated by an indenter (Fig. 2).

The disturbing effects in the bulk of the film become smaller as the load on the indenter is decreased and it penetrates less deeply into the film. By gradually decreasing the load (and performing each repeat test on a previously unused area of the surface), a condition is finally reached where the indenter no longer penetrates into the material, but causes only a reversible elastic deformation. The situation is somewhat analogous to that of a centrally loaded beam, so weighted that only a reversible elastic deformation occurs and there is no irreversible deformation (Fig. 3).

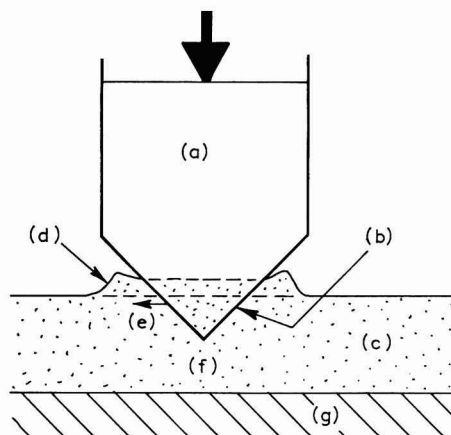


Fig. 2. Penetration of indenter into resin

a indenter; b friction, indenter/resin; c resin film; d displaced material (roll); e flow; f compression; g substrate

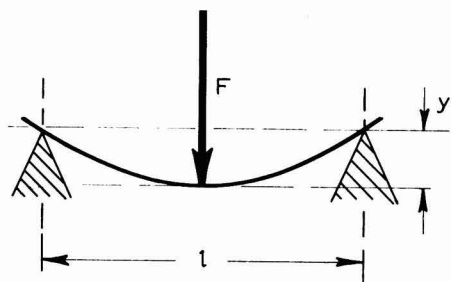


Fig. 3. Reversible deformation of an elastic centrally loaded beam

When pigmented paints or plastics are subjected to mechanical indentation, a plastic flow of the pigment particles within the resinous mass occurs with extreme ease. If a sufficiently small load is applied, a stronger or weaker cohesion will persist between the pigment and the binder, depending on the conditions of wetting⁶. It is possible to apply a load to the pigment/resin system so small that the pigment particles are not torn from their binding and, therefore, the system is not brought to a state where plastic flow occurs. Pigment particles, elements of resin, micelles, entanglements, etc. all remain elastically bonded to each other. The IHV value corresponds to this final state of the purely reversible, elastic deformation attained by a continuous decrease of deformation and can be defined as the border value at the point of transition from viscoelastic deformation to purely elastic reversible deformation, when the load on the indenter becomes infinitely small. Pigmented plastics and paint usually have structures which permit continuous transition from one state to the other.

Differential equation leading to the IHV value

Refs. 7-9, 11, 12

A paint or plastic surface, when loaded with an infinitely small force (such as an indenter in the form of a sphere, cone or Vickers pyramid) does not correspond to the centrally loaded beam shown in Fig. 3, but to that of the elastic

deflection of a centrally loaded disc supported on a non-yielding substrate (Fig. 4).

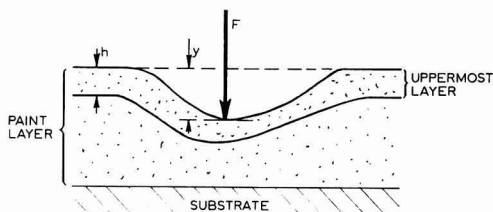


Fig. 4. Elastic deformation of a centrally loaded circular elastic disc on a yielding substrate

Hertz⁷ and later Föppl⁸ determined a differential equation of higher order for such a case. Föppl starts from the general equation for the deflection of an elastic disc⁹:

$$\frac{m^2 \cdot E \cdot h^3}{(m^2 - 1) \cdot 12} \left(\frac{\delta^4 \xi_0}{\delta x^4} + 2 \frac{\delta^4 \xi_0}{\delta x^2 \delta y^2} + \frac{\delta^4 \xi_0}{\delta y^4} \right) = p \dots (1)$$

in which m = Poisson constant in the Hertz-Föppl terminology (Table 1), E is the elastic modulus, h is the thickness of the disc (in this case the thickness of the uppermost layer of the paint film), ξ_0 the elastic deformation, x and y , are perpendicular co-ordinates to the ξ_0 axis and p is the loading per unit surface area.

Table 1

Comparison of the Poisson constant m (usually between 3 and 4) according to Hertz/Föppl and ν (usually 0.3 to 0.4) according to Young¹¹

Hertz/Föppl	Young
$G = mE/(2(m + 1))$	$G = E/(2(\nu + 1))$
	$\nu = 0.305$ for polystyrene, and $0.45g$ for cellulose acetobutyrate
For the mixture: $m = 3.01$ $G = (3.01/(2 \times 4.01))E$ $= 0.374E$	$\nu = 0.34$ $G = (1/(2 \times 1.34))E$ $= 0.374E$

According to equation (1), Föppl gave the determining differential equation for the elastic deformation of a disc on a yielding support when loaded by F at the centre of the disc⁸ as:

$$-K \cdot l^2 \cdot \left[\frac{d}{dx} \left(\frac{d^2 \xi_0}{dx^2} + \frac{1}{x} \frac{d \xi_0}{dx} \right) \right]_{x=a/l} \cdot 2a + F = 0 \dots (2)$$

$$l = \sqrt[4]{\frac{m^2}{m^2 - 1} \cdot \frac{E}{12K} \cdot h^3} \dots (3)$$

where K is equal to q/ξ_0 (force per unit area per unit length), which is equivalent to the "bedding value" of the yielding support, with q being the force per unit surface area exerted by F on the elastic disc (corresponding to the skin of a paint layer (Fig. 4).

These equations enabled Hertz, by the use of Bessel functions, and Föppl, by using another method, to integrate the expression and so obtain a solution for the bending of a disc of infinitely large diameter supported by a yielding support under the load F .

$$y = \xi_0 = \frac{F}{8K \cdot l^2} = \frac{F}{8K \sqrt{\frac{m^2}{m^2 - 1} \cdot \frac{E}{12K} \cdot h^3}} = y \cdot 8K \sqrt{\frac{m^2}{m^2 - 1} \cdot \frac{h^3}{12K}} \cdot \sqrt{E} \dots (4)$$

$$F = c \cdot y \sqrt{E} \dots (5)$$

According to the last section, the IHV value is simply the value of the differential quotient dF/dy for the purely elastic deformation of the uppermost layer of the paint or resin under the limiting conditions where the load becomes infinitely small.

$$\left(\frac{dF}{dy} \right)_0 = IHV = c \sqrt{E}$$

This proves that, in practice, the IHV value cannot approach zero, but has a clear physical meaning. In other words, the IHV value is none other than the reciprocal value of the slope of the curve at the origin of the penetration curve, that is, where the deformation is purely elastic and reversible.

This gives rise to two extreme cases, neither of which is applicable to paints in practice:

- (a) $IHV = 0$, the slope of the curve is infinitely great; (this corresponds to the case of an infinitely mobile system of points, for example an ideal gas).
- (b) $IHV = \infty$, the slope is zero (the case of an infinitely hard non-yielding body).

Both these cases are untenable for resin and paint films. For these the IHV value must lie between the extremes; therefore, it is finite and thus the IHV value must exist.

It has been shown that, in practice, the value of c for resins and paint films is almost constant, so that by estimation of the IHV value the E modulus of the outermost layer of a resin or paint film on a substrate can be determined in a simple manner; otherwise it would be necessary to use a detached film, but the removal of a film from its substrate may cause such changes in the structure of the film that the modulus of the free film may no longer be the same as when it was attached to a substrate. In many cases, the characteristics obtained from the IHV value for the uppermost layer of the film may be applicable to the whole mass, but it would not be true for a paint system of a heterogeneous multi-layered character.

The E modulus was determined for various materials by classical methods (bending, oscillation) and compared with values calculated from the IHV values. Table 2 shows that good agreement was obtained.

Since the quantities m and h used in equations (3) and (4) are only approximately equal for the types of material concerned, the results will vary somewhat on this account and the constant c may also vary within narrow limits so that the E modulus values may vary on this account. Since the variation of the quantities k , m and h will have only a small effect upon the calculations, however, the differences remain within the limits of the accuracy of the measurements and the inhomogeneities in the material.

Table 2
Comparison of the E modulus

Material	IHV value	$E = (IHV/c)^2$ ($c = 2.12$)	E by classical method
Polystyrene 475K	330	24 300	24 000
Vinoflex KR3303*	360	28 800	28 000
Polystyrene 143E	380	32 000	31 000
Vinoflex 516*	410	37 400	35 000
Teluran 886K*	300	20 000	20 000†
Soft rubber	9-10	18-20	20-25
Glass	1 500-1 600	500 000-580 000	500 000

*Teluran and Vinoflex are resins from BASF Limited¹²

†Standard value taken for the evaluation of constant c

Experimental determination of the IHV value

Refs. 3-5

As described above, first the penetration depth y will diminish proportionally with the progressive reduction of the load F applied to the indenter and will change continuously with the bending depth (y as $F \rightarrow 0$), giving the IHV value ((dF/dy) as $F \rightarrow 0$).

Calculation of the slope of $y = f(F)_t$ leads to the curve $F/y = F_t$ (see Figs. 5 and 6) and extrapolation of the $F/y = F_t$ curve to the limit $F \rightarrow 0$ gives the IHV value.

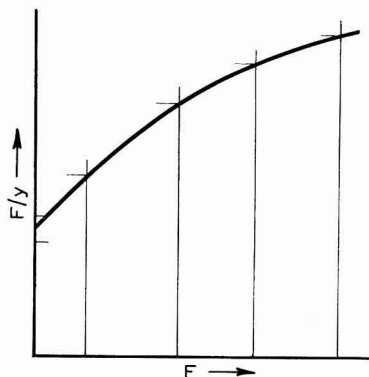


Fig. 5. Graphical determination of the IHV value
(See JOCCA, 1974, 57, 353)

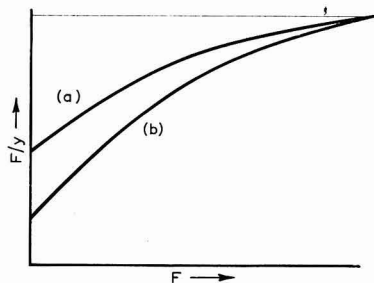


Fig. 6. Epitactic detection of the continuous change of the F/y value to the IHV value
Polyester 10:10 (FPL); a on glass; b on Teflon; $y_{ep} = 39\mu\text{m}$

Special developments can be employed to reduce the drawing and mathematical work involved. Even using the previous drawing methods, it has been possible to discover significant effects, for example, indication of structural changes and internal stresses caused by loading, pigment/binder reciprocal effects and so forth³⁻⁵. Several initial loading values should always be employed and the results at each loading should not be averaged; the $F/y = f(F)$ curve should be drawn as a regression curve of many F/y values (Fig. 5).

By this means the discrepant results are given their appropriate weight. The IHV value is then given as the point of intersection of the regression curve with the F/y ordinate.

If this extrapolation process appears to be unsafe, an interpolation method can be used, as the most certain point on the indentation curve is where $y = 0$, when $F = 0$. The $y = f(F)$ curve may be extended to this point and then interpolated to obtain a number of y , F and F/y values in the region of the IHV value. (See JOCCA, 1974, 57, 353.)

By these procedures, results accurate to within 2 to 10 per cent can be obtained, according to the homogeneity of the sample, for example the fineness of grinding.

Detection of epitaxial effects* in the outermost layer of films by the IHV value

Detection of the continuous transition of the F/y value from the IHV value to the F/y values for deeper penetration

Ref. 13

Sell¹³, by means of wetting contact angle measurements and the calculation of surface tension σ , was able to detect epitaxial effects when the same saturated polyester resin was hardened on glass and "Teflon". The IHV values were determined on the surfaces which had been facing the two different substrates (that is, on the film after detachment) and the results, shown in Table 3 and Fig. 6, indicate a marked difference.

Table 3
Surface tension and IHV value for the saturated polyester resin 10.10*

	Hardened on glass	Hardened on Teflon
Surface tension (N m^{-1})	0.035	0.026
IHV value	46	18-22

*Research Institute for Pigments and Paints eV, Stuttgart (Professor K. Hamann) and Institute for Physical Chemistry of Surfaces (Dr P. J. Sell).

These tests prove that the IHV value characterises the conditions existing in the outermost layer of plastics, paint films and so forth.

If it is further considered that in the above case the same resin was applied to two different substrates, it appears obvious that increasing the distance from the boundary

**Epitaxy* This effect was first recognised as an orientated overgrowth during crystal formation. For instance, NH_4I normally crystallises in regular form, but when allowed to crystallise on mica it does so on a triangular form. This is an effect due to field forces arising from the substrate. The same type of effect arises in paints, and especially in clear lacquers, where the arrangement of macromolecules is influenced by field forces arising from the substrate.

layer, that is increasing the penetration into the resin layer by increasing the loading on the indenter, must lead finally to a constant F/y value (Fig. 6). This is what happens and thus provides further confirmation that the IHV value applies only to the outermost layer of the film.

For finely pigmented systems the IHV value and, therefore, the E modulus of the outer layer, will depend upon the E modulus of the binder and of the pigment and on interaction between the pigment and binder. It can also be calculated from Fig. 6 that the epitaxial effect apparently extends to a depth of:

$$y = 3\,500/90 = 39\mu\text{m}$$

Assuming that the actual epitaxial forces propagate their influence from the interface only to a distance of the order of 10^{-9}m into the bulk of the substance, and in this case the effect is apparent up to a depth of $4 \times 10^{-8}\text{m}$, the sole logical explanation which comes to mind is that a gradually diminishing transfer of effects from layer to layer occurs. In the present case, the effect has tailed off at $40\mu\text{m}$. In the case of pigmented films it would have occurred much earlier.

Assessment of the elastic component of a viscoelastic system by means of the IHV value

Refs. 4, 5, 14-16

The IHV value is determined from the normal penetration curve $y = f(F)$. Since the IHV value refers only to the elastic reversible deformation component, it seems useful to keep the plastic factors contributing to the deformation value as small as possible. Measurements should be started, therefore, with the smallest possible loads, and with a suitable, not too short constant loading time. The time should be kept as short as possible, depending on the skill with which the penetration depth can be observed and measured with certainty. Fig. 7 shows the variation of the depth of penetration with time for constant loads of different magnitudes.

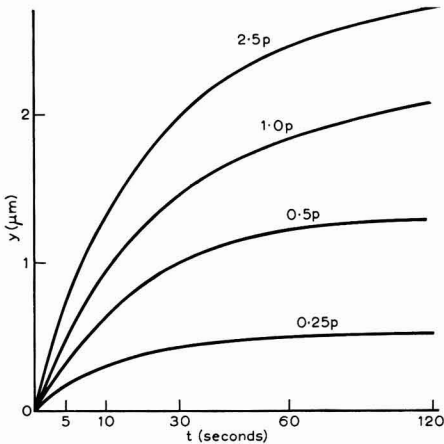


Fig. 7. Elastic and plastic deformation

Paint film EB20 μm . Approx. 10.4.73, measured with Wallace apparatus with Vickers pyramid $\alpha = 136^\circ$ on 30.7.73 (23°C, 50% RH)

It is apparent that in the case of this resin, when using a load of 2.5p on a Vickers pyramid indenter, the penetration depth continues to increase with time, even after a measuring period of 120s; that is, plastic flow of the material still modifies the elastic behaviour.

With smaller loading the plastic flow decreases substantially, so that it can be assumed that the plastic component will

disappear when the load tends towards zero. This is a further argument that the IHV value concerns only the purely elastic state and corresponds to a state of equilibrium between the loading and the opposing elastic forces.

In addition, Fig. 7 illustrates that when selecting the constant loading time to be employed when plotting a curve $y = f(F)$, loading times which are too short should be avoided. Five seconds should be the absolute minimum for manual measurements.

A doubtful demonstration of the non-existence of the IHV value

The non-existence of the IHV value is expressed occasionally, and two experimental considerations may be quoted:

(a) The first assumes that the penetration curve follows a parabolic course for the curve $F/y = f(F)$, and it is concluded, therefore, that all these curves must be parabolic and hence vertical at the zero point. Consider the measured F/y curve shown as curve I in Fig. 9; this appears to be curved like a parabola, but it can be calculated that if point 1 should be a part of a parabola then the points 2 and 3 should lie on curve II instead of on the measured curve I. This is not the case, however, and so the hypothesis of a parabolic course, and from it the argument that the IHV does not exist, is weak.

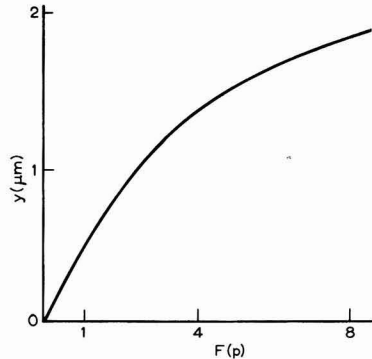


Fig. 8. Penetration curve obtained with paint Sch EB (μm) on 27.4.73 using ICI microindentation tester (apparatus for which the scale of penetration was only provisionally standardised, character of the curve not decisive)

Sapphire sphere 0.397mm dia. Penetration corresponds to a loading time of 20secs.

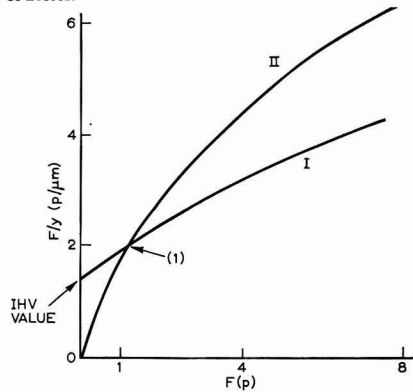


Fig. 9. F/y curve (I) calculated from measurements shown in Fig. 8 and the comparison at point (1) and the zero point lying on parabola (II)

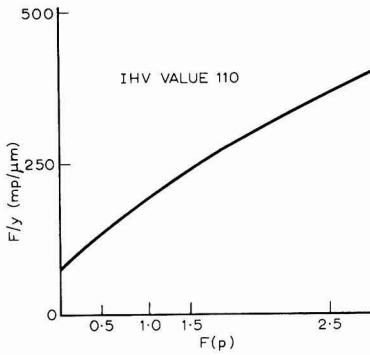


Fig. 10. Plate B from FATIPEC 1972 (Oesterle, Signer and Luscher) TiO₂ pigmented alkyd/melamine formaldehyde stoving lacquer

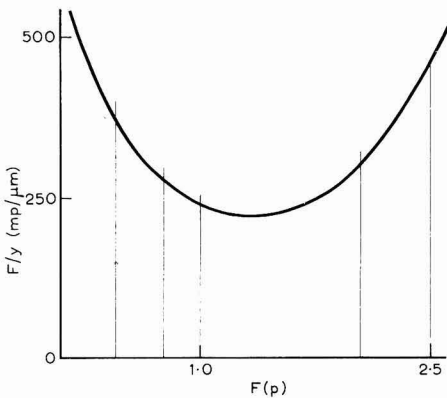


Fig. 11. WM 829.3 mechanically stretched ZnO pigmented additive containing polyester-Versamide combination

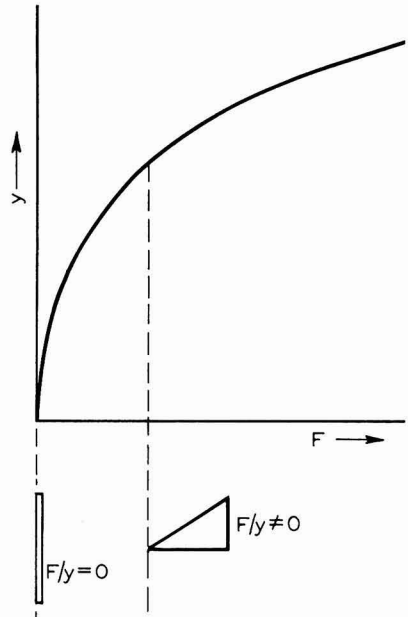
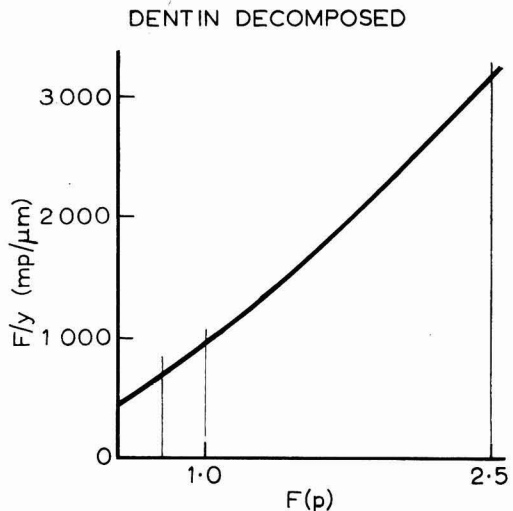
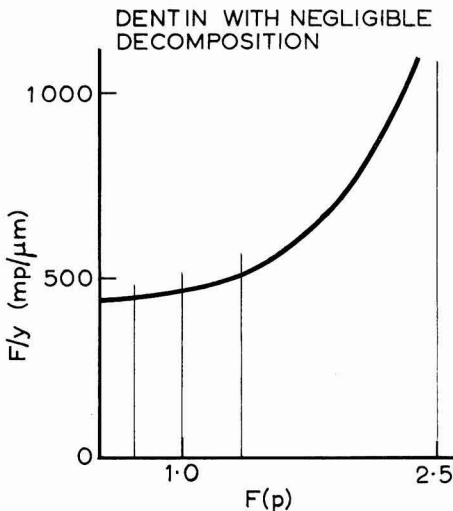


Fig. 14. Proof of the impossibility of the IHV value of a resin film becoming zero. $F/y = 0 =$ hypothetical course of curve

This situation is still more clearly shown in Figs. 10-13. In the case of Fig. 10, the course of the F/y curve is a straight line and in the special case of Fig. 11, this has changed to a concave upwards curve. Both these cases exist in practice and arose during research on the determination of the IHV value.

This shows that the F/y curve clearly reflects the highly complex conditions caused in the intricate structure of the



Figs. 12 and 13. Dental caries preserved in formaldehyde (Dr med. dent. M. Oesterle)

film by the deep penetration of the indenter. However, the F/y curve will never pass through the origin.

There is one further reason why the F/y curves are not likely to pass through the origin of the co-ordinates. Suppose, for example, the loading-penetration curve $y = f(F)$ takes the form shown in Fig. 14, so that the tangent to the curve at the origin is perpendicular to the F axis. Then in this case $(F/y)_{F=0}$ is actually equal to zero. This would mean that the application of an infinitely small force would cause an infinitely great displacement of the parts (deformation = penetration depth = y). This corresponds to the behaviour of an ideal gas and not to the behaviour of a solid film—nobody would assert that a paint film is an ideal gas!

(b) Another (apparent) reasoning for proving the “non-existence of the IHV value” is based on the Hertz equation for the deformation of a sphere and a plate. In his work published in 1882 Hertz¹⁴ assumes that both the deforming sphere and the plate deformed by this sphere have identical elastic moduli. This could hardly be the case when a steel, diamond or sapphire sphere penetrates a paint film. Hertz later specifies that both the sphere and the plate should undergo only purely elastic deformation. In the case of deep indentation this is not true in practice. However, in the attempt to justify the above reasoning, the expression given by Hertz for the approach α of the sphere centre towards the plane of the plate surface is quoted:

$$\alpha = 1.23 \sqrt[3]{\frac{P^2}{E^2 \cdot r}} \dots \dots \dots (6)$$

In this equation α = the movement of the sphere centre towards the plate due to elastic deformation of the sphere and plate; P = pressure on the plate and sphere; r = radius of the sphere, and 1.23 is a factor for the geometrical relations including the Poisson constant $(10/3)^{1/5}$.

In the nomenclature used here ($y = \alpha$; $F = P$) then

$$y = 1.23 \frac{F^{2/3}}{E^{2/3} \cdot r^{1/3}} \dots \dots \dots (7)$$

$$= \text{constant} \times \left(\frac{F}{E}\right)^{2/3} = c \times \left(\frac{F}{E}\right)^{2/3}$$

$$y = c \cdot E^{-2/3} \cdot F^{2/3} \dots \dots \dots (8)$$

Differentiation gives:

$$(dy/dF = (2/3) c E^{-2/3} F^{-1/3} \dots \dots \dots (9)$$

so that for $F = 0$, $(dy/dF) = \infty$ and $(dF/dy) = 0!$?

whereby it is apparently proved that the IHV value does not exist.

According to Lee and Radok¹⁶ the formula:

$$\frac{E}{1 - \nu^2} = \frac{3}{4} \frac{mg}{r^{1/2} \cdot h^{3/2}} \dots \dots \dots (10)$$

(in which E is Young’s modulus, ν Poisson’s constant, h the (total) depth of penetration of the sphere, r radius of the penetrating sphere, and mg the weight acting on the indenter) can be applied to viscoelastic bodies, if the E modulus is replaced by the corresponding viscoelastic magnitude.

Equation (10) can be written, replacing mg by the symbol F and the symbol h by y to give:

$$y = \text{constant} \times E^{-2/3} \cdot F^{2/3} = c \cdot E^{-2/3} \cdot F^{2/3} \dots \dots (11)$$

and this equation corresponds to the Hertz equation (10) exactly, and on differentiating and setting F to zero, gives:

$$dF/dy = 0$$

Although the equation (10) might be used for the calculation of Young’s modulus from a single value of deep penetration of an indenter into the resin layer, it must never be applied for the borderline case where F tends towards zero, after which the viscoelastic conditions no longer prevail. It is here that the striking remark by Föppl should be remembered “. . . only he who has clearly understood the basic principle upon which the formulae are based, may use them in confidence; for he will recognise the limitations for their application and be able to make some fair guesses even beyond these limits”.

The significance of the penetration and recovery curves

Refs. 4, 5

In Fig. 15 the penetration curve $A-B$ with a Vickers pyramid indenter loaded with $2.5p$, and the recovery curve $B-C$ without any loading are shown. The tests were carried out with a Wallace micro-indentation tester.

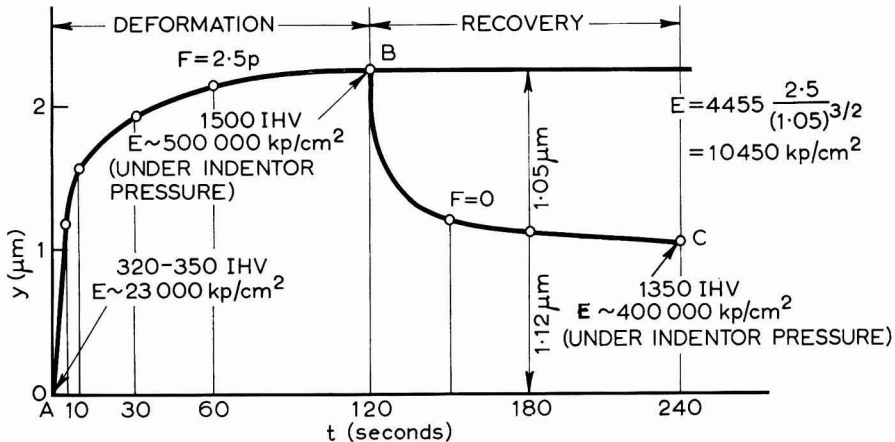


Fig. 15. Paint EB20 μ m. Approx. 10.4.73 measured in Wallace apparatus with Vickers pyramid $w = 136^\circ$ on 24.7.73

At points *B* and *C* the IHV values were determined by a new method, measuring in the penetration spot of the Vickers pyramid indenter. Whilst the paint film in its original state at *A* gave a value for *E* of 23 000, under the pressure of the indenter tip to point *B*, the film has been transformed to one of different structure with *E* equal to 500 000, which up to the point *C* had recovered only slightly. This is a sign that the elastic limit has been far exceeded. Considering the great specific surface pressures which are exerted by an indenter, whether sphere or pyramid, this is by no means surprising (Table 4).

Table 4
Specific surface pressure of an indenter

Indenter	Penetration depth <i>y</i> (m)	Loading <i>F</i> (p)	Specific surface pressure σ_a (p/cm ²)
Vickers pyramid	2×10^{-6}	2.5	5.0
Sapphire sphere (0.397mm dia.)	2×10^{-6}	2.5	0.3*

*It may seem that a sphere ought to be a more suitable indenter tip than the tip of a pyramid or a cone. This is true in the case of a perfectly plane paint film surface. However, the occurrence of an even minute unevenness (micropore, grain, etc) upon which the sphere may rest would give rise to changes in the specific surface pressure to a great but unknown extent.

This value for *E* of 500 000 seems impossible especially if, misunderstanding the situation, it is remembered that with increasing penetration of the indenter into the material, the specific load per unit surface area diminishes. It must be stressed that the specific pressures were not measured in *A* and in *B*, but the IHV value was measured for the non-compressed material in *A* and for the heavily compressed material in *B*.

It becomes clear from these results that the deformation and recovery curves cannot be used for the characterisation of the original paint or plastic.

This fact is all the more important because, as earlier works have shown^{4,5}, once deformed films will never return spontaneously to their original state. Thus measurements taken at points *B* and *C* (Fig. 15) are unsatisfactory, since at these points the film no longer corresponds to its original state.

Both the deformation and recovery curves may be used for the comparison of coatings, but due to the fact that these curves are highly dependent on the operating parameters, such comparisons must be treated with extreme caution and regarded only as relative measurements between materials of a very similar type.

Conclusions

It has been shown by a review of earlier work and also on the basis of new epitaxial measurements that determination of the Infinitesimal Hardness Behaviour (IHV) characterises, in a quantitative and practically parameter-free manner, the outermost layer of plastic, glass, ceramic materials, paint films and so forth. As the outermost layer, which is characterised by the IHV value, responds very rapidly to external influences, the IHV measurements can be used for the quick, informative characterisation of these materials. Consideration, however, must be given regarding the extent, if any, to which the outer surface behaviour is characteristic of the whole bulk.

From the Hertz equation for reversible elastic deformation, which is the only one applicable to the IHV value, it can be shown mathematically that the IHV value cannot be zero. Calculated from the *E* modulus, it is a constant of the material

and has a definite finite value, obtained from the measurement of the purely elastic, reversible bending of the resin film.

The *F/y* curves, because they lead to the IHV value, cannot be extended beyond this final value.

The equations for the irreversible viscoelastic deformation are not to be applied to the entirely different region of purely elastic, reversible film deformation. Consequently, they cannot be used to prove that the IHV value does not exist.

Some measure of the hardness of a plastic or paint film may be obtained from the *E* modulus, but it does not give a complete picture. For the estimation of the "hardness" of a film in the usual sense, both the *E* modulus and the flow behaviour of the film have to be considered simultaneously. A film will be called "hard" if it has a high *E* modulus and exhibits only slight plastic flow. In this sense the time curves of deformation and recovery provide a certain amount of information, but it has to be realised that whilst performing the experiment, the film has already undergone certain changes due to the high surface pressure of the indenter and is no longer in its original state; therefore, the calculation of the elastic modulus from any single value of the deformation curve presents difficulties which do not arise in the determination of the elastic modulus by the IHV method.

[Received 29 January 1974]

Acknowledgment

The author is indebted to the Hon. Editor, Mr S. R. Finn, for translation of this paper into English. The Hon. Editor, in turn, expresses his gratitude to Dr M. Ellinger for her valuable advice during this work.

Notes on proof

It should be noted that due to the limited time available for production, it has not been possible to reproduce some of the Figs in this paper exactly to scale. The author will be pleased to supply more precise details on request.

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Reviews

Antifouling

By I. Lunn

B.C.A. Publications, Thame, England, 1974.
Pp. xvi + 86. Price £2.85

The title of this book might suggest that it will appeal mainly to a specialist readership concerned with marine compositions. It should appeal more widely because it is a very readable account of the development of an industry, simple in its presentation and written by one who obviously cares about his subject matter. Anyone interested in ships and shipping—and there should be many in this maritime nation—can read, enjoy and learn from this simple treatise, mainly historical, which is so pleasantly written. Practitioners in the technology of protecting ships are unlikely to find much that is new to them of the technology, but they are likely to discover a great deal about the origins and development of their industry and the companies engaged in it. Those less directly concerned with the subject will find it an absorbing historical account and it is well annotated and illustrated.

A. RUDRAM

Section Proceedings

Hull

The protection of structural steel

A joint meeting with the Institute of Chemical Engineers was held on Monday 7 October 1974 at the Haven Inn, Barrow Haven, South Humberside, with Mr E. Armstrong in the chair. Some 28 members and guests attended to hear Mr J. R. Bourne, Mebon Limited, give a lecture entitled "The protection of structural steel".

For the benefit of those present with little or no knowledge of paint technology, Mr Bourne began by giving a brief synopsis of the three main constituents of a paint. An anti-corrosive paint acted as a barrier between the substrate and the environment and the pigments used were selected almost entirely for their ability to inhibit metallic corrosion. The speaker discussed the relative merits of zinc dust, zinc chromate, red oxide, zinc phosphate and micaceous iron oxide as pigments for use in anti-corrosive paints.

The paint binders used for protecting steel structures included oleoresinous, alkyd, bituminous, chlorinated rubber, epoxy ester, two-pack epoxy/polyamide, two-pack polyurethane, two-pack epoxy/pitch and vinyl systems. The advantages and disadvantages of each were detailed in terms of dry film properties, durability, application properties and cost effectiveness.

More failures were due to poor substrate preparation than to any other cause. Abrasive blasting was by far the best method of preparing a surface for painting; the blasting time was important and the grit size had to be of the correct order. Factors which must be considered when selecting a paint included application environment (rural, industrial or marine) and economics.

Anti-corrosive paints could be applied either inside a factory (for example, on steel girders for bridges) or on site. A large number of coloured slides illustrated the uses of anti-corrosives on motorway bridges and lamp standards, jetties, gas holders, fertiliser plants, petroleum tanks and

Assignments for vibrational spectra of 700 benzene derivatives

By G. Varsanyi (Edited by L. Lang)

Adam Hilger, London, 1974.
Two volumes. Pp. 668. Price £25

Although there are collections of infra-red spectra of resins, oils, solvents and pigments in paint laboratories possessing an infra-red spectrometer, it is sometimes not so easy to find a reference spectrum of a simple organic compound, for example, isolated from a paint system as an additive or a derivative. The volumes under review, therefore, form a useful collection. Many of the compounds included may be isolated from paint systems as accelerators, curing agents, ultraviolet screening agents, biocides, antioxidants, etc., and require identification. There are of course adequate collections of the spectra of organic compounds in the main science reference libraries, but they are fairly formidable and one visit would pay for the cost of the book under review. The theory of the vibrational spectra is considered in detail.

L. A. O'NEILL

dockside cranes, and the different types of paint system for these applications were explained.

In conclusion, Mr Bourne emphasised that maximum benefit from any system could be achieved only through close consultation between manufacturer and user.

The lecture was followed by a short discussion during which Mr Bourne answered questions put to him by his audience. A vote of thanks was proposed by Mr P. W. Munn who complimented the speaker on his attractive presentation.

D.M.W.

Microscopy for paint and pigment chemists

A student evening was held on Monday 21 October 1974 in the Leonard Balmforth Lecture Theatre at the Hull College of Technology, Queens Gardens, Hull. Mr K. Green of the Hull College of Technology delivered a lecture entitled "Microscopy for paint and pigment chemists".

Mr Green began his lecture by giving a brief history of microscopy from the primitive microscope to the electron microscope. Microscopy had now been adopted by industry for its quickness in solving certain analytical problems associated with the physical characteristics of pigments and emulsions.

Resolution was defined and the speaker discussed the relationship between numerical aperture, resolving power and magnification. The mechanics of the microscope and its illumination were equally important as the lens system; with the aid of a series of slides, the use of illuminators and special lighting techniques was illustrated.

The use of the "stage micrometer" was demonstrated and the method for calibrating the eye-piece of a microscope explained. Again with the aid of a series of slides, the lecturer illustrated the "globe method" for counting particles, the "phase contrast method" for the determination of the size of emulsion particles and the "shear method" for assessing particle size distribution.

The lecturer defined polarised light and discussed the use of polarising filters. An opportunity was given at the conclusion of the talk to handle photo-micrographic equipment, including a simple polaroid micrographic camera used for particle size determinations.

The meeting, which was attended by five members and seven visitors, was closed with a vote of thanks to the speaker from Mr A. R. Vanspall.

D.M.W.

Industrial application of organic titanates

The second meeting of the 1974-75 session was held at the George Hotel, Land of Green Ginger, Hull, on Monday 4 November 1974. Mr E. Armstrong introduced Dr P. Kay of Titanium Intermediates Limited, who delivered a lecture entitled "The industrial application of organic titanates— with particular emphasis on surface coating applications".

Dr Kay began his lecture by describing the preparation and physical properties of organic titanates. The most important reactions were those relating to exchange, hydrolysis and chelation. The areas of application of organic titanates could be categorised in terms of the action of the organic titanate, the nature of which could be catalytic, titanium dioxide forming or cross-linking.

Organic titanates could be used as catalysts for esterification reactions, for example in the preparation of di-octyl phthalate, and for alcoholysis reactions, for example in the preparation of butyl acrylate for the manufacture of acrylic lattices. They might also be used in polyesterification reactions, for example in the production of Terylene and olefin polymerisation reactions where a higher and narrower molecular weight distribution could be achieved.

Iso-propyl titanate had been used to produce very thin films of titanium dioxide (50nm thick) on the surface of glass.

Midlands

Petrochemical-based raw materials for the paint industry

Mr M. E. Robinson of ICI Limited presented a paper to a meeting at the Apollo Hotel, Hagley Road, Birmingham, on Friday 27 September 1974, with the title "An appreciation of the availability and prices of petrochemical-based raw materials for the paint industry".

The speaker commenced with a brief resumé of his company's interests in the petrochemicals field and then traced the developments of the current situation from mid-1972. At that time, the increasing demand for petrochemical-based raw materials was being adequately met, and spare capacity was high. Profit margins were poor and consequently investment was low. Over the following few months, various shortages became increasingly apparent, with world markets buoyant and low prices still prevailing. The superimposition of the energy crisis on this situation led to massive price increases. British industry, in particular, was embarrassed at an international level by the imposition of price controls, which have only recently been relaxed.

Forecasts for future trends were difficult because of the unpredictable effects of inflation. However, short term prospects were likely to show reduced demand, leading to improved availability and a measure of price stability. After perhaps 12 months, shortages were again probable, and these might persist for two to three years. After this period, capacity would tend to catch up with demand, and competition would once more become a factor in marketing, but at considerably higher price levels. Buyers would, in future, have to choose between price and security of supply.

The lecturer's invitation to discuss the various points raised was enthusiastically taken up by the audience. A vote of

The films deposited in this way gave very much smoother surfaces to milk bottles than before, and consequently the cost of breakages had been greatly reduced. Polybutyl titanate had been used as an aid to improve the adhesion of printing inks on polypropylene substrates and as an additive to upgrade the heat and corrosion resistance of zinc and lead aluminium paints. Dr Kay exhibited weathered car exhaust pipes to demonstrate the use of polybutyl titanate for corrosion resistance.

Titanates when used in combination with terephthalate polyester-imide resins in the formulation of wire enamels, had been found to promote better flow, increase the rate of cure, lower the cure temperature and improve adhesion and shock resistance. In addition, butyl titanate could improve the water repellency of coatings for textiles, leather, paper and masonry.

Tri-ethanolamine chelates could produce reversible gels in some latex paint systems, producing films free from brush-marks and sagging. Their action was not fully understood as it had been found that not all emulsion paints could be gelled using chelates. The nature of the emulsion stabiliser was important and an excess of surfactant could prevent gelation. With vinyl acetate emulsions, yellowing due to the liberation of vinyl acetate monomer could occur, but this could be overcome by careful selection of the chelating agent. The pH value of the paint was also important and in this respect it had been found best to select a pH value and then choose a chelating agent to suit the system.

Dr Kay's lecture was well received by the audience and the speaker was subjected to a large number of varied questions, all of which were well answered. The meeting closed with a vote of thanks from Mr R. Tyngate and the audience expressed their appreciation in the usual manner. The meeting was attended by 20 members and visitors.

D. M. W.

thanks was given by Mr P. Goodale, whose approval of the content and delivery of Mr Robinson's lecture was obviously shared by the rest of the audience.

F.W.C.

Water or what?

The meeting on 18 October was opened by the Chairman, who introduced the speaker, Mr K. O'Hara of Cray Valley Products Limited. Mr O'Hara presented a lecture entitled "Water or what?" on behalf of Mr R. H. E. Munn.

In order to predict future trends, some of the paint industry's statistics from the recent past were examined, emphasis being laid on the water-based coatings, which would find their greatest utility in non-polluting paints. Next followed a resumé of the requirements of Rule 66, with broader references to the economics of environmental controls and energy conservation. Improvements were being brought about in three ways; namely, by the elimination of solvents, the reduction of solvent and/or replacement by less polluting solvents, and the use of water. Water-based coatings could be classified as either aqueous dispersions, hydrosols, or water-reducible coatings.

The advantages and disadvantages of the various systems under discussion were pointed out, and the speaker was convinced that conventional paint systems would continue to be used until legislation inevitably forced the use of less polluting alternatives.

A lengthy discussion followed; one interesting point which emerged being the looseness of terminology which is still very apparent within the paint industry, leading to vagueness in the interpretation and definition of a number of commonly used terms.

A vote of thanks was proposed by Mr A. S. Gay, the audience registering their approval in the usual manner.

F. W. C.

Information Received

Foamaster

Diamond Shamrock (UK) Limited has incorporated the name Foamaster in all its defoamer products as part of a policy to unify its present diverse range of product names and prefixes.

Lithene plant on stream

Revertex Limited announced start up of its "Lithene" liquid polybutadiene plant towards the end of last year. The plant, claimed to be the biggest of its kind in the world, has an annual capacity of several thousand tonnes.

Magnetic level indicators

Auriema Limited has been appointed UK representatives for Weka AG of Switzerland, manufacturer of a range of magnetic level indicators.

Stabilising wood with sugar

The Paint Research Association, working on behalf of the International Sugar Research Foundation Incorporated, has developed a new bulking treatment for wood, based on sugar, which considerably reduces dimensional changes. The sugar is chemically modified to produce an aqueous resin solution which with an added curing catalyst is used to impregnate the wood. After curing, the resin forms a substantial amount of water-insoluble intractable material within the wood.

Simon-Rosedowns contract in Brazil

Simon-Rosedowns Limited of Hull, which specialises in the design and manufacture of equipment for the vegetable oil and protein industries, has been awarded a contract by Olvebra S.A. Brazil worth £400 000 to supply the continuous "Rotocel" solvent extraction plant to process soya beans.

New products

Highly active non-mercurial preservative

A non-mercurial bactericide, with performance claimed equivalent to phenyl mercury acetate in interior PVAc matt emulsions, has recently been announced by the Troy Chemical Corporation, USA and is being distributed in the United Kingdom by Cole Chemicals Limited.

Troysan 192-2-(hydroxymethyl amino)-2-methylpropanol is a water-soluble liquid recommended for use as an "in can" preservative for emulsion paints and for polymer and resin emulsions.

Low volume pipette

F. T. Scientific Instruments Limited has introduced a low cost pipette with a capacity continuously variable up to 1.0ml. The instrument is claimed to be ideal for organic solvents and poisonous/

obnoxious samples as all parts except the tip (which is disposable) are made from metal or glass and are, therefore, sterilisable.

Meetings

OCCAA Convention

The Oil and Colour Chemists' Association Australia will hold its first International Paint Convention in September 1977. Pacific Paint Convention '77 will have as its theme "Modern coatings—efficiency in formulation, production and application".

Those interested in presenting a paper at the Convention should contact the Chairman, Technical Programme Subcommittee, OCCAA Pacific Paint Convention '77, PO Box 93, Punchbowl, NSW 2196, Australia.

The Colour Group—Newton Lecture

The Newton lecture of the Colour Group of Great Britain will be given on 5 February 1975 at the Royal Institution London, when a lecture "Sky-blue pink" by R. W. G. Hunt will be followed by the Colour Group Dinner. Those wishing to attend should contact Mr J. Hutchings, The Pyghtle, Milton Ernest, Bedford (Tel. Bedford (02 34) 55251).

Notes and News

Forthcoming Section Symposia

Newcastle

Ultraviolet polymerisation and the surface coatings industry

The Section is organising a one-and-a-half-day symposium to be held on 10 and 11 April 1975 with the title "Ultraviolet polymerisation and the surface coatings industry—theoretical and practical considerations". Papers will be presented by speakers from the industry and universities, covering practical and theoretical aspects of the rapidly developing technology of ultraviolet curing. Topics will include photo-initiated polymerisation, photo-condensation polymerisation, photo-degradation and photo-stabilisation, ultraviolet curing sources, photo-sensitive oligomers and polymers, and photo-initiators.

All enquiries should be directed to the Section Hon. Social Secretary, Mr H. Fuller, Tioxide International Limited, Carlton Weathering Station, Yarm Back Lane, Stockton-on-Tees, Cleveland TS21 1AX. Further details will appear from time to time in this *Journal*.

Thames Valley

Gloss and its assessment

The Section is to hold a one-day symposium at Brunel University on Tuesday 25 March 1975 with the theme "Gloss and its

assessment". The subjects covered will include gloss emulsion paints, printing inks, pigments and recent physical methods for gloss assessment. Further details will be announced in the *Journal*.

London

Aqueous coatings

The Section is organising a one-day Symposium on aqueous coatings to be held at the Polytechnic of the South Bank, London, commencing at 9.30 a.m. on 21 January 1975.

The papers to be presented are given below, and during the day there will be a discussion in two parts on the topics arising from these:

"A rationalisation of economic and technical factors in the development of emulsion polymers for paints" by J. E. R. Reynolds, B. W. Bulezuick, and K. A. Safe (Vinyl Products Ltd.).

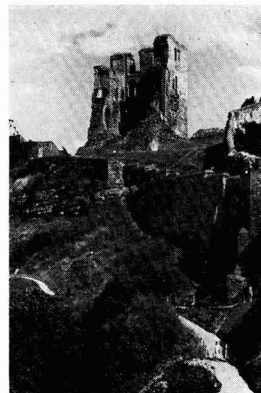
"Water-based epoxy coatings" by F. B. Richardson (Thomas Swan & Co.).

"The use of titanium dioxide in aqueous decorative gloss paints" by J. Clarke (Tioxide International).

"Water-based pigment systems" by A. A. Abel (Hoechst UK Ltd.).

"Industrial aqueous coatings" by S. Erratt (Valentine Varnish & Lacquer Co.).

OCCA CONFERENCE 1975— See page 36



The photograph shows Scarborough Castle and some of the pleasant walks around its perimeter. It was from this view that Robert Hamblin made a sketch which forms the basis for the Conference motif. It is a most effective symbol, with the staunch bare walls of the castle providing as great a challenge to the surface coating specialists as will the wealth of information to be gleaned from technical and workshop sessions and from informal discussion at the forthcoming Conference.



OCCA—XXVII Exhibition

Olympia, London. 22-25 April 1975

The International Forum for Technical Display and Discussion in the Surface Coatings Industries

★ Belgium ★ Finland ★ France ★ Germany ★
 ★ Holland ★ Hungary ★ Italy ★ Norway ★
 ★ Poland ★ Rumania ★ Sweden ★ Switzerland ★
 ★ UK ★ USA ★

General information

As announced in the December issue of the *Journal* there has been one of the heaviest demands for stand space by exhibitors for OCCA-XXVII, the Association's annual international forum for technical display and discussion in the surface coatings industries, which will take place at Olympia, London, from 22 to 25 April 1975.

Since the list of exhibitors was published on page 11 of the December issue, there have been additional allocations and these are shown in bold type in the list given below.

Directly represented now among exhibitors are organisations from the UK and the following thirteen overseas countries: Belgium, Finland, France, Germany, Holland, Hungary, Italy, Norway, Poland, Rumania, Sweden, Switzerland and the USA.

The "Official Guide" and season admission tickets

The "Official Guide" to the Exhibition is now being prepared for publication; advertisements can be accepted from non-exhibitors and those wishing to do so should contact the Assistant Editor to the Association at its headquarters in Priory House, Wembley.

Members of the Association will automatically be sent an individual copy of the Official Guide, together with a season admission ticket, when the booklet is published (normally several weeks before the opening day). It has been decided on this occasion, however, to make a small charge to non-members of £1.00 to cover both the Official Guide and the season admission ticket. Non-members wishing to receive tickets and copies of the "Official Guide" in advance should send to the Association's offices the relevant form enclosed in this issue of the *Journal*, together with the necessary remittance, as soon as possible.

Copies of the Official Guide and season admission tickets will also be available at the entrance to the Exhibition.

Venue

As in 1974, the exhibition will be of four days' duration and will open on the Tuesday morning at 09.30 and will close on the Friday at 16.00. The exhibition of raw materials, plant and equipment used in the

paint, printing ink, colour, and allied industries will take place at the Empire Hall, Olympia, London.

Tuesday 22 April	..	09.30-17.30 hrs
Wednesday 23 April	..	09.30-17.30 hrs
Thursday 24 April	..	09.30-17.30 hrs
Friday 25 April	..	09.30-16.00 hrs

Motif of Exhibition 1975

The motif for 1975, designed by Robert Hamblin, continues the theme of OCCA-26, when attention was drawn to the European Economic Community, by extending it to show the world-wide interest aroused by the association's annual exhibitions in London, which in recent years have attracted visitors from more than 50 overseas countries. The motif is printed in colour elsewhere in this issue, and these colours (two shades of blue and one of green) will be used throughout the publicity material for the exhibition. Two (cascade blue and leaf green) will be incorporated in the fascias of the stands, thus giving both an entity to the design and pleasing changes of colour to visitors as they move from corridor to corridor. A feature of OCCA exhibitions has always been the provision of special seating areas, where visitors can meet friends and discuss problems—and these areas continue the colour theme chosen for each particular exhibition.

Aim of the Exhibition

The aim of the Exhibition is the presentation of technical advances in those industries supplying the paint, printing ink, colour, linoleum and allied industries and the Exhibits may relate not only to new products but also to new knowledge on existing products and their uses and existing knowledge which is not generally available in the consuming industries.

The Committee stipulates that exhibitors present a technical theme—that is display in a technical manner the technical developments in raw materials, plant or apparatus illustrated by experimental evidence. It is essential that a technically or scientifically trained person, who has full knowledge of the products displayed, be available on the stand throughout the official hours of opening.

Special visits by overseas trade delegations

Following the great success of the delegation from Osaka, Japan, on the occasion of

OCCA-26 when special arrangements were made for works visits etc, requests have already been received for similar facilities for a delegation from Czechoslovakia, Switzerland and for a further Japanese party.

Any company wishing to be placed on the list of those willing to accept such delegations should write to the Director & Secretary as soon as possible, setting out the countries in which they are interested and the scope of the activities which would be displayed.

The OCCA Exhibition and the raw material shortages

At the 1974 exhibition, when the acute shortage of raw materials was causing so many problems, the value of this annual technical display and the opportunity which it gives for the free interchange of ideas between suppliers and manufacturers in a relaxed atmosphere was especially evident, and new exhibitors at that exhibition were delighted with the response achieved and the goodwill built in a period of the most adverse of conditions in the industry. Indeed, the 1974 exhibition demonstrated the great strength of this unique and remarkable exhibition in weathering the frustrating problems and difficulties, such as the three-day working week in the United Kingdom at the beginning of the year, which severely handicapped so many other functions and caused the cancellation of others. The Council of the Association is convinced that the enthusiastic support which it received from exhibitors was reflected in the interest aroused by visitors and augurs well for the 1975 and future exhibitions.

Travel Agents

The Wayfarers Travel Agency Ltd., Cranfield House, 97/107 Southampton Row, London WC1B 4BQ, will be allocated a stand adjacent to the OCCA Information Centre at the Exhibition and will be prepared to advise on, and arrange, hotel accommodation and travel facilities to the Exhibition. They will also be able to make theatre ticket reservations for the evenings of the Exhibition.

Please address all enquiries to The Wayfarers Travel Agency AT THE ADDRESS SHOWN ABOVE, and not to the Association.

The Exhibition Committee has allocated space to the following organisations:

- | | | |
|--|---------------------------------------|----------------------------------|
| Abbey Chemicals Ltd. | *Croda | *Meijer's, Rudolph, Inc. |
| *Akzo Chemie (UK) Ltd. | *Degussa | Microscal Ltd. |
| *Amoco International | Diffusion Systems Ltd. | Montedison |
| Anchor Chemical Co. Ltd. | *Durham Raw Materials Ltd. | *Norwegian Talc, A/S |
| *Arco | *Dynamit Nobel AG | †Phillips Petroleum Company |
| Arcode (Imeco) | *Dyso Industrier AS | †Polychrome Ltd. |
| *Baker Perkins Chemical Machinery Ltd. | Eastman Chemical International AG | Polymers, Paint & Colour Journal |
| †BASF Farben & Fasern AG, Bereich | GAF (Great Britain) Ltd. | *Polyvinyl Chemie Holland BV |
| Siegle, Stuttgart | Glen Creston (Willy Bachofen) | *Porter Lancastrian Ltd. |
| *BASF United Kingdom Ltd. | *Henkel & Cie. GmbH | Research Equipment (London) Ltd. |
| *Bayer AG | *Hercules Powder Co. Ltd. | *Rhône-Poulenc |
| Boulton, William, Ltd. | *Hilton-Davies | *R.K. Chemical Co. Ltd. |
| *British Industrial Plastics Ltd. | *Hoechst UK Ltd. | *Roehm GmbH |
| *British Steel Corp. (Chemicals) Ltd. | Imperial Chemical Industries Ltd. | Rohm & Haas (UK) Ltd. |
| *Byk Mallinckrodt | Jacobson van den Berg & Co. (UK) Ltd. | *Sachtleben Chemie GmbH |
| *Cabot Carbon Ltd. | Jenag Equipment Ltd. | *Sandoz Ltd. |
| *CdF Chemie | *Joyce-Loebel Ltd. | Sangamo Weston Controls Ltd. |
| *Chemische Werke Huls AG | *Kemira Oy Vuorikemia | S.C.C. Colours Ltd. |
| Chemolimpex | *Kinhurst Engineering Ltd. | *Schering AG |
| *CIBA-GEIGY (UK) Ltd. | Laporte Industries Ltd. | *Sheen Instruments Ltd. |
| Ciech | *Manchem Ltd. | *Shell Chemicals UK Ltd. |
| Crosfield, Joseph, & Sons Ltd. | Marchant Bros. Ltd. | Sun Chemical Corporation |
| *Cornelius Chemical Co. Ltd. | | *Synres International BV |
| | | *Tenneco Chemicals Europe Ltd. |
| | | Toioxide International Ltd. |
| | | Torrance & Sons Ltd. |
| | | *Union Carbide Europe SA |
| | | *Vebe-Chemie AG |
| | | *Wacker-Chemie |
| | | *Wheatland Journals Ltd. |
| | | Winter Osakeyhtiö |
| | | Worsdall Chemical Co. Ltd. |

Presentation of Scroll of Honorary Membership



The photograph shows the presentation of a scroll of Honorary Membership of the Association conferred on Robert W. Matlack (right) at the 1974 Annual General Meeting of the Association being presented to him by the President of the Federation of Societies for Paint Technology, Michael W. Malaga, on the occasion of the Convention of the Federation at Atlanta City. The scroll reads:

It was unanimously agreed this day by the Council of the Association to confer Honorary Membership upon Robert Matlack Esq., formerly Executive Vice-President of the Federation of Societies for Paint Technology, in recognition of the outstanding service which he rendered for many years in promoting co-operation between this Association, the Federation and with kindred organisations internationally.

National Westminster Bank Ltd.
OCCA Information Centre
Wayfarers Travel Agency
Technical Education
Interpreters

*Did not exhibit at OCCA-26
†Has not previously exhibited

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News of Members

J. R. E. F. Coates, an Associate Member attached to the London Section, has joined Synthetic Resins Limited as its southern area representative. Mr Coates was previously with Manchem Limited.

Mr A. S. Lewis, an Ordinary Member attached to the Manchester section, retired at the end of September last year after 25 years service with SCC Colours Limited.

Mr H. Smith, an Ordinary Member attached to the General Overseas Section and a past chairman of the Manchester Section, has been appointed a Director of Robbialac Paints (Tanzania) Ltd.

The following Members have been appointed to the Council of the British Colour Makers' Association for the session 1974-75: Mr G. K. Burrell, Mr F. B. Mortimer-Ford, Dr F. Dalton, Mr W. B. Cork, Mr P. Perkin and Mr H. Slack.

Forthcoming Events

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

January 1975

Monday 6 January

Hull Section: "Influence of paint additives on film properties" by Mr W. H. Lakin, Hardman and Holden Ltd., to be held at 6.30 p.m. at the George Hotel, Land of Green Ginger, Hull.

Thursday 9 January

Midlands Section—Trent Valley Branch: "Surface pretreatment and application" by Mr P. B. Wharton of Loyne (Site Contracts) Ltd., to be held at 7.00 p.m. at the British Rail School of Transport, London Road, Derby.

Newcastle Section: "Effects of employee participation on management" by Mr J. W. E. Morgan and Mr J. Burrell, British Titan Products Ltd., to be held at 6.30 p.m. at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne.

Friday 10 January

Scottish Section: Annual Dinner Dance to be held at the Albany Hotel, Glasgow.

Manchester Section: Joint Meeting with the Institute of Printing "Audio visual systems—are they a threat to the printing industry?" by Mr J. V. Ashworth, Manager, Telecommunications, IPC Services Ltd. *To be confirmed.*

Tuesday 14 January

West Riding Section: "Developments in painting techniques in British Rail" by a speaker to be announced to be held at 7.30 p.m. at the Griffin Hotel, Leeds.

Wednesday 15 January

Manchester Section—Student Group: "Instrumental colour control" by Mr G. H. Eastwood of Crown Paints Ltd., to be held at 4.30 p.m. at the Manchester Literary and Philosophical Society, George Street, Manchester.

Scottish Section—Eastern Branch: "Chloride process titanium dioxide pigments, properties and applications" by Mr G. R. Siddle of Laporte Industries Limited, to be held at 7.30 p.m. at the Carlton Hotel, North Bridge, Edinburgh.

Friday 17 January

Midlands Section: Annual Dinner lecture. "Extenders—Their present and future positions" by Mr R. J. Simpson and Mr D. Huxtable of English China Clays Ltd.

Tuesday 21 January

London Section: "Aqueous coatings". One-day symposium. See below.

Wednesday 22 January

Irish Section: "Current trends in synthetic resins" by Mr A. R. H. Tawn of Coates Bros & Co., to be held at 7.45 p.m. at the Clarence Hotel, Dublin.

Thursday 23 January

Thames Valley Section: "Acrylic emulsions" by Dr Washbourne, Rohm and Haas (UK) Ltd. to be held at 7.00 p.m. at the Beech Tree Hotel, Maxwell Road, Beaconsfield, Bucks.

Saturday 25 January

Scottish Section—Student Group: Works visit to Hunterston Power Station, West Kilbride.

Friday 31 January

Bristol Section: "Liquid inks and their development" by Mr H. C. C. Whitehead of Coates Bros & Co. Ltd.; 7.15 p.m. at the Royal Hotel, Bristol.

February

Monday 3 February

Hull Section: "Science and crime" by Mr G. Devonport, North Eastern Forensic Science Laboratories, Harrogate, to be held at The George Hotel, Land of Green Ginger, Hull, at 6.30 pm.

Thursday 6 February

Thames Valley Section—Student Group: "Additives" by Mr Lakin, Hardman & Holden Ltd., to be held at Slough College in the main lecture theatre at 4.00 pm.

Newcastle Section: "Adhesives and sealants" by Mr N. Macdonald, Evode Ltd., to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne, at 6.30 pm.

Friday 7 February

Scottish Section—Eastern Branch: Burns Supper in the Lady Nairn Hotel, Edinburgh.

Thames Valley Section: Buffet Dance, Great Fosters Hotel.

Monday 10 February

London Section: "The painting of metal bridges—historical and current trends" by Mr P. Ferguson, Materials Quality Assurance Directorate, at East Ham College of Technology, High Street South, London E6, at 7.00 pm.

Tuesday 11 February

West Riding Section: "Paint packaging" by C. I. Mellor, Metal Box Co. Ltd., to be held at the Griffin Hotel, Leeds, at 7.30 pm.

Thursday 13 February

Scottish Section: "Paint pollution and possibilities" by Mr R. H. E. Munn of Cary Valley Products Ltd., to be held at Beacons Hotel, 7 Park Terrace, Glasgow G3, at 6.00 pm.

Midlands Section—Trent Valley Branch:

"World economic events affecting petroleum and its derivatives" by a lecturer from Esso Chemicals Ltd., to be held at the British Rail School of Transport, London Road, Derby, at 7.00 pm.

Friday 14 February

Manchester Section: "Some forward views on the energy situation and raw material supplies" by Dr J. K. Hambling of BP Chemicals Ltd., to be held at the Manchester Literary and Philosophical Society, 36 George Street, Manchester, at 6.30 pm.

Saturday 15 February

Scottish Section—Student Group: "Chrome pigments and their usage" by Mr A. C. D. Cowley of ICI Ltd., to be held at Three Pigeons, 573 Sauchiehall Street, Glasgow, at 10.15 am.

Wednesday 19 February

Scottish Section—Eastern Branch: "Photography" by Mr D. Rosie of Craig & Rose Ltd., to be held at the Carlton Hotel, North Bridge, Edinburgh, at 7.30 pm.

Irish Section: "Ireland—agricultural or industrial?" by Mr L. Sheedy, to be held at the Clarence Hotel, Dublin, at 7.45 pm.

Friday 21 February

Midlands Section: "Chemicals from coal—the impact of the energy crisis" by Mr P. Joy, British Steel Corporation, Chemical Division, to be held at the Birmingham Chamber of Commerce and Industry, 75 Harborne Road, Birmingham B15 3DH, at 6.30 pm.

Newcastle Section: Ladies' Night at Five Bridges Hotel, Gateshead.

Wednesday 26 February

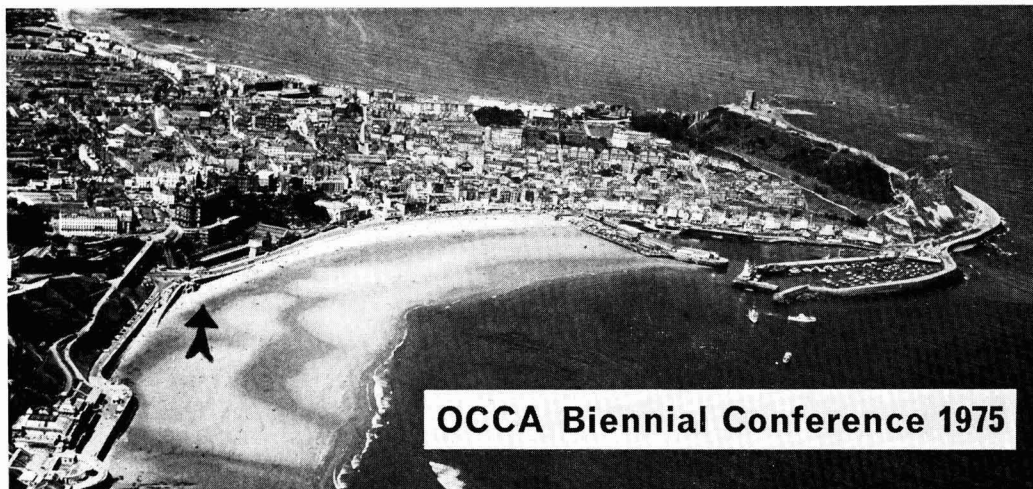
Manchester Section—Student Group: "The prevention of bacterial corrosion of mild steel with paint films" by Mr A. V. Robinson of Camrex (Holdings) Ltd., to be held at the Manchester Literary and Philosophical Society, Manchester, at 4.30 pm.

Thursday 27 February

Thames Valley Section: "Protection of off-shore oil rigs" by Mr F. G. Dunkley, to be held at the Beech Tree Hotel, Maxwell Road, Beaconsfield, Bucks, at 7.00 pm.

Friday 27 February

Bristol Section: Lecture to be arranged.



The arrow points to the Grand Hotel

Performance of surface coatings—does reality match the theory?

As already announced in the *Journal*, the next biennial Conference of the Association will take place at Scarborough from 17 to 21 June 1975. The headquarters will be the Grand Hotel, with overflow accommodated at the St. Nicholas Hotel. These two hotels are situated opposite each other on St. Nicholas Cliff. The title of the Conference will be "The performance of surface coatings—does the reality match the theory?" and it is intended that as on previous occasions, full preprints will be published for despatch to delegates in advance of the Conference.

Forms for registration are being despatched to all members of the Association attached to the Sections in the United Kingdom, Ireland and the General Overseas Section and are also included in this issue of the *Journal* for the benefit of

non-members. Council has fixed the registration fees for the Conference at £30 (plus VAT) for members, £50 (plus VAT) for non-members and £10 (plus VAT) for wives.

Non-members wishing to avail themselves of the preferential Conference fee for members should request membership application forms from the Association's offices and these should accompany the Conference registration forms.

The closing date for registration will be 1 April 1975.

There will be seventeen papers presented and a full list is given below. The Chairmen of the five sessions will be Dr G. de W. Anderson (Director, Paint Research Association); Mr A. G. Holt (Vice President); Mr G. Hutchinson (formerly Chairman of

the Eastern Branch of the Scottish Section); Mr A. T. S. Rudram (President Designate); and Mr A. R. H. Tawn (Hon. Research & Development Officer). Appearing in the February issue of the *Journal* will be the division of the lectures into the five sessions, and these will take place as follows: Sessions 1 and 2 on Wednesday 18 June; Sessions 3 and 4 on Thursday 19 June; and Session 5 on the morning of Friday 20 June. Following the Association's AGM on the Friday afternoon, there will be three Workshop Sessions (limited in numbers) on the following topics:

1. "How can we control over-specification?"
2. "What is the reality of toxic hazards for the surface coatings manufacturer?"
3. "The impact of non-impression processes on the printing ink industry."

The papers to be presented, together with summaries and biographies of the authors are given below:

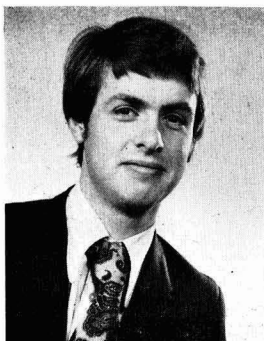
Flocculation—its measurement and effect on opacity in systems containing titanium pigments

By J. G. Balfour and M. J. Hird

Some simple tests for the detection of flocculation of titanium pigments in alkyd gloss paints are discussed. A more sophisticated technique for the measurement of flocculation involving the use of scattering of infrared radiation is described and a variety of paints examined using this technique.

The opacity of flocculent paints is then related to the pigment particle distribution in the dried paint film. A model is proposed in which the paint film is considered to consist of regions of different pigment concentrations. This model is used to interpret experimental results from air-drying alkyd and stoved alkyd/melamine systems. Finally, the parameter which defines the extent of the flocculation is compared with results from the infrared technique.

Mr J. G. Balfour graduated from the University of Leeds in 1971 with an Honours degree in Colour Chemistry. He then joined the Group Research

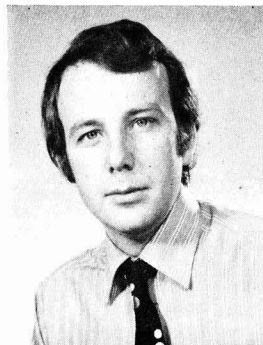


J. G. Balfour

Laboratory of Berger, Jenson and Nicholson Limited and was concerned with pigment evaluation.

In 1973 he joined the Technical Service Department of Tioxide International as a Technical Officer and is now primarily

engaged in work concerned with the physical testing of pigmented materials.



M. J. Hird

Dr M. J. Hird graduated from Sheffield University in 1968 with an Honours degree in Physics, and in 1970 with a degree of MSc. He continued his research

studies during 1970-71 at Manchester University and was awarded his doctorate in 1973.

He joined Tioxide International in 1971 as a Technical Officer and has been principally concerned with examining the optical properties of pigmented systems.

Testing of surface coatings by the customer

By D. A. Bayliss

The Protective Coatings Testing Laboratory for the Central Electricity Generating Board was started by the author in 1960.

The paper describes the thinking behind its paint testing and evaluation methods, in particular, its special emphasis on realism as opposed to reproducibility. It discusses the value or otherwise of painting specifications, codes of practice, independent inspection and guarantees, in solving problems of paint application.

The paper gives examples of successful and unsuccessful results within the CEGB and concludes with a consideration of the responsibility of user testing laboratories.



D. A. Bayliss

Mr Bayliss has worked for the Central Electricity Generating Board since 1947 when he started as a Power Station Chemist. Since 1960 as a Senior Research Officer in the Scientific Services Department he has been responsible for the setting up and control of the Protective Coatings Laboratory of the CEGB. The work deals with approval of all types of coating materials, including conventional paints, metal coatings and plastic coatings, also the investigation of paint and corrosion problems and the development of special materials.

He was on the London Section Committee of OCCA from 1968-1972 and was the Hon. Programmes Officer from 1970 to 1972. He is currently Chairman of the Institution of Corrosion Technology and of the National Council of Corrosion Societies. He is Chairman of the Panel dealing with "Inspection" for the revision of British Standard Code of Practice 2008 and also serves on BS committees dealing with paints and is on the main and Technical Committee of the Public Sector Standardisation Team.

Practice makes perfect

By A. E. Claxton

Methods for the protection of structural steelwork by paint are compared and contrasted with those used for painting in automotive production. Good painting practice can produce results which match expectations provided the theory is soundly based. Reasons for some shortcomings are discussed.



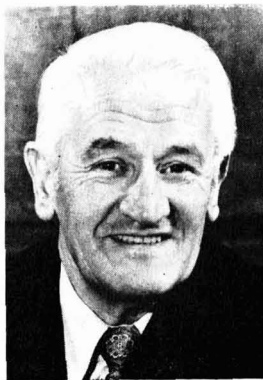
A. E. Claxton

Mr Claxton graduated in Chemistry from Imperial College London in 1949. In 1956 he joined Griffiths Bros. (now Inmont Ltd.) as Chief Chemist, and has specialised in the formulations of protective paint systems for steel. He has been a member of the Technical Advisory Committee of the Paint Research Association and a member of OCCA Council.

Quality control of painting in the construction industry—making reality match the theory

By F. G. Dunkley

If paints were correctly applied, their performance could match the theoretical expectancy. Claims are sometimes made, however, not on secure theoretical grounds but involving imaginary ideal conditions for application. Quality control can ensure that work is carried out within the theoretical tolerances of the product or at least provide an explanation of the unexpected failure and the precise conditions which led to it.



F. G. Dunkley

Mr Dunkley was previously Superintendent of the Protective Coating Laboratory, Chemical Services Division of British Railways Research Department. This laboratory was one of three specialised laboratories within Chemical Services. He later joined BIE Anti-corrosion Limited. He is past Chairman of the Trent Valley Branch of the Midlands Section and a Fellow of the Institute of Corrosion Technology, being a past Vice-President and one-time Chairman of its Yorkshire Branch.

Opportunities and constraints for the chemical industry

By G. S. Galer

The paper will deal with some of the problems of planning the chemical business in the economic and political environment of the 1970s.



G. S. Galer

Mr Galer read mathematics and economics at Cambridge and is a member of the Operational Research Society and the Society for Long Range Planning. He has been with Shell since 1957 and has gained wide experience of the Group's chemical business through assignments in the UK, Italy and the USA. His present position is Planning Manager for Shell Chemicals UK Limited.

Protection: From hypothesis to principle

By P. J. Gay

The way in which protective systems are built up will be discussed, bearing in mind the materials available, the parameters which can be measured and unmeasured factors relating to the conditions to be withstood. This general theme will be supported by specific examples.



P. J. Gay

Mr Gay graduated in London in 1930 whilst with the Paint Research Association and spent a number of years at the Paint Research Station. In 1950 he joined Hangers Paints Limited as Technical Director and in 1969 became Technical Director of Storry Smithson & Co. Ltd. until his retirement in 1974.

Since becoming a member of OCCA in 1939 he has served as London Section Programmes Officer (1941-50), a member of Council 1946-49 and 1950-69, Honorary Research and Development Officer 1951-59 and was President of the Association from 1959-61.

Prediction of corrosion protective properties of paint films by permeability data

By H. Haagen

It is generally agreed that in the mechanism of corrosion protection by paint films, the diffusion of corrosive agents to the film/metal interface and of corrosion products to the surrounding media are of great if not crucial importance. Contrary to this statement, considerable confusion has existed whether diffusion data like water permeability may be useful to predict paint performance. During the last few years it has been increasingly realised that besides water diffusion, other diffusion processes, especially oxygen diffusion, must be considered when correlating permeability data with practical performance. Results from literature will be compared with the author's data and additional evidence will be provided to show that the rate of water permeation determines the rate with which adhesion of the paint films to the metal substrate is diminished or lost, but that oxygen permeability may be the rate determining factor for the corrosion reaction occurring beneath the paint film. Apart from low film thickness of films above the critical pigment concentration, ion permeability of paint films is usually too low to influence the corrosion rate. This is in accordance with the blistering of paint films which requires semi-permeability or at least strongly permeation retarding properties of the films. Salt spray tests must, therefore, be considered as criteria for film defects like pores, scratches or holes and not for the effectiveness of corrosion protection afforded by a paint system. Methods of measuring diffusion rates will be critically reviewed and the problems involved in measuring and relating results obtained with free and supported films will be discussed.



H. Haagen

Dr Haagen received his PhD, from the University of Stuttgart. After ten year's work with the paint industry he joined in

1968 the Forschungsinstitut für Pigmente und Lacke eV as head of the application department.

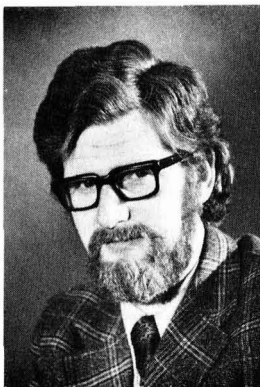
Problems encountered in testing paint films

By M. B. Kilcullen

Six of the factors which affect paint performance have been studied to determine their relative importance. The factors studied were (i) paint film thickness, (ii) type of paint, (iii) surface preparation, (iv) weather during painting, (v) weather during drying and (vi) site of exposure.

The condition of the specimens after 6.5 years' exposure has been assessed and statistical analysis of the results has been carried out.

The investigation is discussed in terms of the problems encountered with the experimental design, difficulties in assessing quantitatively the performance of paint films and some of the unexpected results obtained.



M. B. Kilcullen

The author graduated in 1962 from University College, Dublin and joined the Corrosion Control Section of the British Iron and Steel Research Association in that year. He is currently employed as a Project Officer in the Corrosion Control Section of what has now become the Corporate Engineering Laboratory of the British Steel Corporation. In addition to the work under discussion, his main research is on "weathering" steels. He is also particularly interested in laboratory simulation of corrosion and statistical design of experiments.

Theories—laboratory investigations—practical performance

By B. Lindberg

To illustrate the theme embodied in this title, examples will be given from the author's own research work and experience. The problem will be highlighted by analysis and discussion of results from the following research fields:

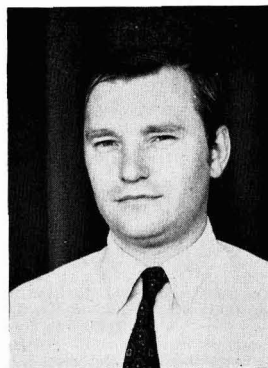
Painting plastics. Some theoretical principles concerning the relationship between adhesion, surface tension (of the dry paint film and of the substrate), wetting tension, critical surface tension (Zisman) will be reviewed and compared with adhesion values obtained with the "pull off" technique. It will be pointed out that in most

cases it has not been possible to use the more sophisticated theories.

Painting concrete. In this part, the difficulties will be stressed for obtaining correspondence between natural weather deterioration (e.g. freezing and thawing) of painted concrete compared with laboratory methods. In the laboratory tests it was not possible to imitate the deterioration claimed to occur in practice. Concrete wall blocks were painted with very different water permeable paints on the cold and on the warm side of the concrete wall. A moisture and temperature gradient affected the painted concrete blocks for a period of more than six months including freezing cycles on the cold side. No large difference could be noted between types of paint which were permeable and impermeable to water.

Correlation between natural and accelerated weathering. Six very different paint types have been investigated for changes during weathering at three outdoor exposure stations (outside Copenhagen, the west coast of Sweden and in Florida) compared to changes when using different accelerated weathering cycles in weatherometers (carbon arc, xenon lamp, with and without filters, different periods of light and dark). For evaluation of changes in the paint film the conventional properties, such as gloss, chalking, checking etc were measured, but also more advanced techniques as scanning electron microscopy, weight loss, mechanical properties (micro-indentation, tensile strength, elongation) and infrared spectroscopy have been used.

It is concluded that good or bad correlation between outdoor and laboratory testing depends on many factors, such as the type of paint, the measured properties, type of outdoor climate and weatherometer cycle, position of the panel etc. The outdoor climate at the same place may change very much during testing especially in northern Europe. Even for a specific outdoor climate, the position of the painted surface (e.g. south-north, vertical-horizontal, high up or low down) shows very different deterioration.



B. Lindberg

Mr Lindberg holds a Master's degree in chemical engineering (1966) from the Royal Institute of Technology in Stockholm and specialises in polymer science. He was a research engineer at the Grafic Arts Research Laboratory in Stockholm 1966-1968, and Head of the section of

forgery and counterfeits at the National Forensic Science Laboratory 1968-1970.

Since 1970 he has worked at the Scandinavian Paint and Printing Ink Institute (NIF) and is responsible for research projects.

Ecological coatings: the theory and the reality

By A. G. North, R. J. Little* and J. L. Orpwood*

The paper will examine how the claimed advantages of new coatings methods compare with reality in reducing pollution and energy requirements.

In the basic study, a series of coatings are examined to establish the required film thicknesses for a combination of appearance and performance in the case of a white stoving enamel. The various systems are then compared in terms of their polluting effect from the viewpoint of both quantity and relative toxicity; factors will include, for example, solvent vapour, neutralising bases and products of reaction, and waste such solid or liquid as non-re-usable overspray, the rinse from electrodeposition baths etc.

It is proposed that eight systems be compared:

- (1) conventional solvent
- (2) high solids solvent
- (3) non-aqueous dispersions
- (4) powder
- (5) ultraviolet cured
- (6) electron beam cured
- (7) water based
- (8) water based electrodeposition



A. G. North

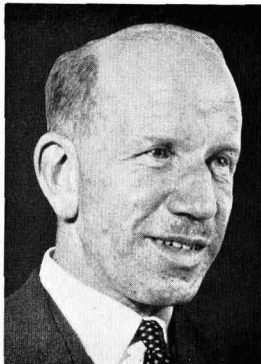
Mr North is a Londoner by birth, and was educated at Bromley Grammar School and London University, from which he graduated with an Honours degree in chemistry. After graduation, he went to the Harvard Business School, where he completed a business management course. Since 1948, Mr North has served with Cray Valley Products Limited in a variety of positions from laboratory assistant to his current position as Technical Director and Joint-Managing Director.

Why did it fail?

By A. N. McKelvie and A. F. Sherwood

Modern paints can be formulated for high durability, low permeability to water or destructive agents and good colour fastness,

but in practice too often fail to achieve long term film integrity or protection of substrates. The reasons for failure will be analysed with attention to specific cases that have been investigated in building, heavy construction and marine applications. Recommendations will be made for avoidance of premature failure by improvements in design, formulation for higher tolerance, integrated programming of construction and protection and better control of application.



A. N. McKelvie

Mr McKelvie joined ICI Explosive Division in 1928 and in 1933 transferred to Nobel Chemical Finishes later to become ICI Paints Division. He was in turn Assistant Research and Development Manager and then Laboratories Administrator responsible to the Technical Director for their general administration of Research Development and Technical Service. During his long service with ICI he took a leading part in the development of protective coatings for a wide range of markets. He is now attached to the Paint Research Association and is the Paint Technologist on the Admiralty Paint and Preservation Study Team. He is also an active member of British and International Standards Committees, and a Fellow in the Technology of Surface Coatings.



A. F. Sherwood

Mr Sherwood joined the Paint Research Association in 1943. In the intervening years he has been involved, amongst other topics, in studies on oil polymerisation, varnish chemistry, molecular weight determinations, the protection of aluminium alloys and the use of tracer techniques.

The latter were used extensively in his studies on the interactions between pigments and media. Now as Section Leader, Building Materials, he is extensively involved in the protection problems of the building industry.

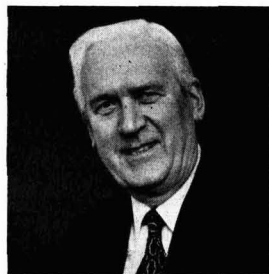
The theory and practice of film formation by co-ordination reactions involving aluminium compounds

By J. H. W. Turner, W. H. K. Lakin and P. Womersley

Investigations carried out by the author over the last few years have confirmed in general terms the theoretical possibilities of preparing media comprising compounds of alkyd and other resins with aluminium compounds which are stable on storage but which gel rapidly when exposed as thin films to the hydrolytic action of water in the atmosphere or in the substrate to which the film is applied.

Such compounds are seen to have interesting and potentially useful properties as coating media but their use may be restricted unless the compositional criteria which, theoretically, determine stability can be controlled within acceptable limits to give a satisfactory and reliable performance in practice.

The author relates the practical performance of a number of media and pigmented systems with the theoretical prediction based on compositional data and presents his conclusions.



J. H. W. Turner

Mr Turner's industrial career began in 1933 with his engagement as a paint chemist by Griffiths Bros. and Co. London Ltd. After wartime service on the manufacture of propellants for the Navy, he spent the next twelve years with British Resin Products Limited on resins and polymers, largely for surface coating applications. In 1956 he joined Hardman and Holden Ltd. and as Director of Research and Development was responsible for pioneering the uses of aluminium alkoxides and boric esters in the formulation of some interesting new metal/organic compounds for uses in driers, fuel catalysts, PVC stabilisers and other applications.

He has been closely associated with the affairs of the Paint Research Association as a member of Council and of its Executive Committee. He was also its Hon. Treasurer until his resignation this year. He continues to serve as a PRA member of the Paint Industry Technical Committee.

For most of his career, he has been a member of OCCA. During the early 1950's he was a member of the Bristol Committee and represented the Section on Council. He has also presented a number of papers on resins, polymer emulsions, driers and other metal/organic compounds

*See page 40

to OCCA conferences, Section meetings, and other learned societies and technical associations, at home and overseas. In 1972, in a paper to the FATIPEC Congress in Florence he first anticipated the development of unique compositions to which he gave the name Alumedica in which derivatives of aluminium alkoxides featured as major components. More recently, as the representative of OCCA at the 1974 meeting of the Federation of Societies for Paint Technology he presented a related paper which disclosed how, by using aluminium compounds, lead driers could be eliminated from surface coatings with advantage in safety and improvements in other aspects of performance.

As Technical Consultant to Manchem Ltd., a company in the RTZ Chemicals Group, John Turner is continuing the work on aluminium organics and other metal/organic compounds with which his name has become most closely associated.

Paints for buildings—the potential and the performance

By P. Whiteley and G. W. Rothwell

Paint has been commonly regarded as a simple, cheap and effective form of protection for perishable building materials, as well as providing economic decorative effects for almost all surfaces. Yet in spite of continuous technological progress in coatings, dissatisfaction with their exterior performance and with the economics of frequent maintenance has been evident, e.g. on wood windows and cladding, on external concrete and masonry and on steel. One manifestation of this is in the trend towards describing coatings as "plastics" not paint, (plastics now having achieved a highly acceptable role in place of their former "substitute" image), and another is the substitution of traditional painted materials by plastics. Some architects and builders have attempted to avoid paints by using "natural" finishes or none at all. Large areas of concrete become drab and dirty because the decoration which paints could offer is thought to be too short-lived. Technologically, the application of coatings by factory processes is a move towards better performance and the longer life evidently needed to retain acceptability, but it has not always achieved these aims.

This paper describes some of those difficulties, and attempts an explanation of poor performance which has been observed. Whilst it is accepted that wrong choice of paint type and unsatisfactory application have an adverse effect on the realisation of paint potential, it is suggested that coatings tend to be formulated with too great a bias towards the other necessary properties besides durability, and that test methods are not always realistic enough to show the likely results in long-term use. Thus, claims for 5-6 years' life for alkyd gloss paints conflict with the 2-year period often needed for first maintenance of new buildings and the frequent failures in the next 4-5 years. Some longer life coatings fail to reach the life claimed because the initial flexibility is lost too quickly; many coatings are evaluated by physical tests at too early a stage, before their full hardness is reached, which may require many weeks.

Coatings will continue to provide the cheapest method of changing and brightening the appearance of older buildings, but a reduction in maintenance cost must be achieved; if it is not by better performance of coatings it will be by their elimination on new buildings.



P. Whiteley

Mr Whiteley has spent almost all his working life connected with paints, the first half in industry during which he took the external Honours degree in chemistry at London University. His last industrial post was with Cellon Ltd., after which he held for several years a research fellowship in tropical paint problems, working at the Building Research Station and in many tropical countries. He is now head of the Surface Coatings Section at the BRS (now part of the Department of the Environment) and studies the performance and requirements of coatings for building materials and structures. He is an Associate of the Royal Institute of Chemistry and Fellow in the Technology of Surface Coatings.

Mr Rothwell had his early training in the metallurgical industry and the Chemical Service Division of the British Railways Board Research Department. After graduating he worked in the Corrosion and Protective Coatings Laboratories at the Railway



G. W. Rothwell

Technical Centre, Derby, and the Chemical Research Division Laboratory of British Rail at Munsell Hill. Nine years ago he joined the Surface Coatings Section of the Building Research Station (now part of the Department of the Environment) where he is a Senior Scientific Officer specialising in the performance of surface finishing materials used in the building industry. He is also an Associate of the Royal Institute of Chemistry and is an Associate in the Technology of Surface Coatings.

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

By T. W. Wilkinson and J. H. Colling

Using alkyd paints pigmented with a range of TiO_2 pigments, results from various test methods are compared. Panels were exposed to a modified BSS 3900 accelerated weathering cycle. Weight loss measurements, together with gloss reduction, TNO chalking and red reflectance measurements on blue tinted panels are used to provide a consistent picture of changes during weathering, based on the Contraction Model, the evidence for which was given in the FATIPEC XII Congress Book on page 69. Methods of interpreting detail in the results are illustrated with consideration of the chalking region.

Similar comparisons are made with natural weathering results (Nettleton UK). Differences between natural and accelerated weathering are discussed especially in terms of UV penetration. The advantages of the control possible with artificial weathering are emphasised.



T. W. Wilkinson

In 1947, after service in the RAF, Mr Wilkinson joined Leyland Paints Limited in the Research and Development Department. He graduated in Applied Chemistry at the University of Manchester Institute of Science and Technology, and joined Mander Brothers Limited in 1952 as Senior Chemist in charge of Paints Division. Whilst working at Manders, he lectured in applied chemistry and paint technology at Wolverhampton Polytechnic for eleven years.

In 1965 Mr Wilkinson joined Laporte Industries Limited Research Department where he is now Research Manager, Applications Research. In this role his responsibilities include the Technical Service and Applications Research activities of Laporte's Organics and Pigment Division.

Mr Wilkinson was a co-author of the second edition of Manfred Hess's book "Paint Film Defects".

Mr J. H. Colling studied chemistry at Manchester University where he graduated in 1966. He joined Laporte Industries Limited where he first worked in the Basic Research Department of the Organics and Pigments Division investigating the effect of titanium dioxide on optical and rheological properties and photochemical degradation processes in surface coatings. Mr Colling joined the Applications Research Department in 1974 and is currently engaged in

the investigation of gloss phenomena and in studies of durability performance and measurement.



J. H. Colling

Prediction of the performance of surface coatings by accelerated testing under normal conditions

By K. M. Oesterle

Comparisons are made between the behaviour of paints during long term outdoor exposure and the changes in IHV (infinitesimal hardness behaviour) values of paints after their exposure in a 23°C humidity cell; the IHV value seems to be a useful aid for the development of paint formulations.



K. M. Oesterle

The basis of the IHV method will be explained in a shortened form to show that the method not only helps in the determination of hardness (or elastic modulus) but also can give new insight into the variation in the behaviour of polymers with time.

Dr Oesterle was born in 1901 in Berne, Switzerland, and was educated there and in Strassburg, Germany. He then went to the Federal Institute of Technology, Zurich, studying engineering and electrochemistry until 1924, and this was followed by several years as assistant in organic chemistry and X-ray spectroscopy at the Universities of Berne and Freiburg. From 1928 to 1948 he held several positions as development engineer and director in the electro-chemical, organic food and pigment industries, and since 1948 has held leading positions in production and research of pigments, paints, plant for paint application and metal pretreatments. The fields which hold special interest for him include: pigment/solvent/vehicle interaction in solid and fluid states; electrodeposition processes; new aspects in "differential" behaviour of polymers; and rheology of polymers, paints etc. in particular with regard to aspects of Bingham and thixotropic flow. He is a member of various scientific societies and was formerly with Mäderlack AG, Switzerland.

The flow of epoxy powder coating films in relation to reactivity, rheology and wetting

By S. Gabriel

Reactivity, viscosity and heating rate are considered as interrelated parameters affecting flow during the cure of powder coatings. Moreover, the theoretical dependence of flow on viscosity and surface tension as indicated by the Rhodes/Orchard equation will be reviewed and compared with reality.

Ecological coatings—see page 38



R. J. Little

Mr Little left school at 15 and studied for City and Guilds Paint Technology, Metal Finishing and Corrosion, and HNC chemistry by part-time study at the Borough Polytechnic and Thames Polytechnic. He took GRIC by full-time study at Medway and Maidstone College of Technology, and is at present finalising an MSc in inorganic macromolecules at the Polytechnic of North London. He has worked as a development chemist on resins and paints for Domolac Duresco and Carson Hadfields and joined the technical service laboratories of Cray Valley Products Limited in 1972.



J. L. Orpwood

Mr Orpwood left school at 17 and joined Cray Valley Products as a laboratory assistant in its technical services laboratories in 1956. After three years' evening class studies, he attended the Polytechnic of the South Bank as an industry-based sandwich course student from where he obtained a degree in chemical technology in 1963. He rejoined CVP in its central research laboratories and worked for three years on water soluble coating resins. Towards the end of 1966 he took responsibility for amalgamating library/information services for the parent group, Coates Brothers & Co. Ltd. Since 1969 he has been Senior Information Officer for the Group. He is currently studying for a degree in mathematics with the Open University.

There will also be a paper by Mr S. L. Davidson, of NL Industries Incorporated, USA, entitled "A new versatile lead free pigment" and it is expected that details of this paper and the author's biography will appear in the February issue. The February issue should also include the division of papers into sessions.

* * * * *

OCCA XXVII—Additional Exhibitors

(See also pages 32 and 33)

The additional exhibitors to whom space has now been allocated are **Berol Kemie AB (Sweden)**, **BOC Special Gases Division (UK)**, **G. J. Erlich Ltd. (UK)**, and **Produits Chimiques Ugine Kuhlmann (France)**

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GENERAL

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Japanese visitors to OCCA XXVII wish to meet manufacturers of radioactivity-proof paints at the Exhibition to discuss the possibility of production under licence in Japan.

Manufacturers wishing to meet the Japanese visitors concerned should write at once to the Director & Secretary of the Oil and Colour Chemists' Association enclosing a sealed envelope marked "To be forwarded to the Japanese manufacturer" and listing on the outside of the envelope the names of any companies to which the envelope should not be forwarded. All such envelopes will be forwarded to the agent arranging the visit who will make the necessary arrangements for a meeting.

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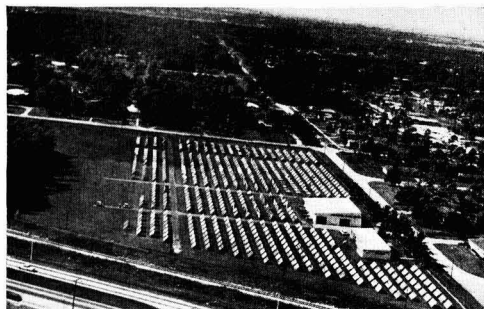
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