ISSN 0030-1337

December 1990
Vol. 73, No. 12

JOURNAL OF THE OIL AND COLOUR CHEMISTS' ASSOCIATION

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Vol. 73 No. 12 DECEMBER 1990

JOCCA

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ISSN 0030 - 1337

An Official Journal of the Society of British Printing Ink Manufacturers and the Paintmakers Association of Great Britain Ltd.

DECEMBER 1990

Contents

Cover: Accelerated weathering of paint films. (Photo by courtesy of Tioxide UK Limited)

Forthcoming Features: January – Greener Coatings; Process Operation (PO), Filling & Weighing; February – Polymers & Resins; PO, Small order manufacture; March – Corrosion; PO, Equipment for quality control.
Contributions are welcomed at least five weeks prior to publication date.

JOCCA (ISSN 0030-1337) is published monthly by the
Oil and Colour Chemists' Association,
Priory House, 967 Harrow Road, Wembley HA0 2SF, England
Tel: 081-908 1086 Fax: 081-908 1219 Telex: 922670 (OCCA G)
1990 annual subscription rate: UK £82.00, rest of the world (excluding USA) £90.00, USA $180.00. Single copies £8 ($16).
Air freight and mailing in the USA by Mercury Airfreight International Ltd Inc., 2323 Randolph Avenue, Avenel, NJ 07001. Second class postage paid at Rahway NJ. US Postmaster send address changes to JOCCA c/o Mercury Airfreight International Ltd Inc., 2323 Randolph Avenue, Avenel NJ 07001.

Editorial correspondence should be addressed to the Honorary Editor, and general correspondence addressed to the General Secretary at the Association Headquarters.

Printed by The Herald Press, 17 Rother Street, Stratford-upon-Avon, Warwickshire CV37 6NB Tel: 0789 66261

17-18 March 1992

Harrogate Exhibition Centre
Ripon Road Harrogate North Yorkshire

SURFEX 92 is now officially launched and will be offered for general sale from 17 December 1990.

The venue will again be Harrogate, but SURFEX 92 will be held in Halls A and B of the Exhibition Centre with hospitality suites in the adjoining Royal Hall and adjacent hotels. The decision to remain in Harrogate was reached after a wide ranging consultation with visitors and exhibitors. 80% of exhibitors responded to the questionnaire and over 70% of the respondents voted in favour of the Harrogate Exhibition Centre, Halls A and B.

The Halls will provide for an attractive more spacious layout, carpeted throughout, with provision for some larger stands up to a maximum of 32 sq.m. At the same time the full range of smaller stands will still be provided. It has also been possible to maintain prices per sq.m to those typical of Hall D stands at SURFEX 90.

Apart from the move to Halls A and B, all the regular features of SURFEX will remain, including exhibitors' reception, best stand awards and the popular SURFEX Dinner.

There will be a new face on the SURFEX team with the appointment of Peter Stanton as the Honorary Exhibition Officer, taking over from Fred Morpeth who has relinquished the position to take on the role of President Designate and President in June 1991. Peter, an exhibitor, the organiser of the SURFEX 90 Dinner and a long serving member of the West Riding Committee, will prove a very worthy successor to Fred.

Any company interested in exhibiting should contact Chris Pacey-Day at the SURFEX office for their copy of the SURFEX 92 information pack. The other contacts are: Advertising – Victoria Craik; Official Guide – Peter Fyne; and, Administration and Accounts queries – Hilary Pooley. All may be contacted by telephone 081 908 1086 or fascimile 081 908 1219.

Modification to ISO 9000 logo for the chemical industry

At a recent meeting of the Certification Authority for the Chemical and Allied Industries, agreement was reached on the modification of the ISO 9000 series registration logo for those companies involved in the chemical industry. In future, chemical manufacturers will use in place of ''Registered Firm"; "Registered Chemical Manufacturer" and distributors correspondingly "Registered Chemical Distributor". These symbols can be used after the 1 October 1990 and it is recommended that the change should be completed by the end of 1991 .

ICI Resins products gains BS5750

ICI Resins' Hillhouse International production facility in Lancashire, UK, has been awarded BS5750/IS09002 registration. The production units registered produce: Haloflex, water borne latices for anti-corrosive paints, adhesives and paper coatings, and NeoRad UV-cured resins for optical fibre coatings.

Products

Tioxide launch new TiO_2 grades

Tioxide Group has launched world-wide four new high quality grades of titanium dioxide pigment:

TR20 is a medium crystal, highly dispersible grade for plastics applications, with good durability which makes it particularly suitable for UPVC.

TR44, is a paper laminate grade giving both excellent lightfastness and retention in the paper-making process.

TR63 is a highly durable pigment for automotive and industrial coatings, offering excellent gloss, dispersibility and stability.

TR80 has a combination of extremely high gloss and high opacity, for printing inks.

For further information Enter M101

Wolstenholme new products

Wolstenholme have launched at R&P 90 a range of dusting bronzes and preparation, complementary to surface chemistry on bronze flake and is based on new technology which is compatible with new and old Dreissig machines.

And with the successful launch of the Superoto range at DRUPA '90 in May, manufactured for the liquid ink industry, Wolstenholme expects the 'family' of five different qualities to become as famous as their Unipak, ready-mixed offset metallic inks for quality, stability and performance.

For further information Enter M102

Equipment

New computer system for paint manufacturers

Byproduct is a new computer based system, from Byline Systems Limited and Olivetti Systems and Networks, designed specifically for the paint industry to provides information on production. It monitors statistical information, such as wastage, performance and profitability of the process. It is a complete system and covers the entire cycle from raw materials, finished goods, sales orders and distribution, together with a comprehensive range of accounting facilities to profit and loss balance sheet.

For further information Enter M103

The complete gloss-measurement

BYK-Gardner GmbH, manufacturer of gloss and color measuring equipment, have introduced a new bench-type of glossmeter. The haze-gloss measures gloss at 20°, 60°, 85°, reflection haze, and mirror gloss. Measuring gloss and reflection haze simultaneously enables a differentiation of surfaces to an extent which, up to now, was not possible with conventional glossmeters. Additionally, the reflection haze value may be used as an indirect measure to evaluate the paint characteristics; such as, dispersion behavior and tendency to flocculate or as a control tool for optical characteristics of anodized coatings on metals. The haze-gloss is a reference glossmeter, due to its high precision and long-term stability. No warm-up time is required and calibration has to be done only every two months. An RS-232 interface allows data transfer to a PC and instrument control from the PC.

For further information Enter M104

People

New Group Managing Director for Jenson & Nicholson

Roger Regan has been appointed Group Managing Director of Jenson and Nicholson Limited, a UK based multi-national company with 24 overseas subsidiaries which market paint brands such as Berger, Crown, Magicote, Robbialac, Brolac and Luxol. In addition, Mr Regan will be responsible for the recently acquired UK based Cementone-Beaver Limited and Windeck Paints Limited; UB International (UK) Limited; Soyco plus future acquisitions of the group.

Mr Regan joins Jenson and Nicholson from Caradon Everest Limited, where he was Managing Director. His previous posts have included Group Managing Director of AG Stanley Holdings PLC, the paints and wallcoverings multiple retailer; Managing Director of the Paints Division of Manders PLC; as well as positions at Crown Paints and Tetrosyl Limited. ■

Radiation Curing of Polymeric Materials

Edited by Charles E. Hoyle and James F. Rinstle, ACS Symposium Series 417.
ISBN 0-8412-1730-0 Price $119.95 Published December 1989.

This book arose from a symposium sponsored by the ACS Division of Polymeric Materials: Science and Engineering which was part of the 197th National Meeting of the American Chemical Society, held Dallas, Texas in April 1989. The appearance befits this origin; the camera-ready format has provided 36 "chapters" each having a different typeface. The chapters are written by groups of researchers and are based on the oral presentations at the original symposium.

As the editors observe in their preface, radiation curing has emerged in the past two decades as a major force in the coatings industry. Different sources of radiation are now widely used in the processing of polymeric coatings formulated from a variety of polymers. The initial impetus for the development of these coatings some 15-20 years ago came from concern for the environment. Pressure to improve the environmental acceptability of coatings has not, of course, diminished over the years. In response to this sustained pressure, the use of radiation cured coatings continues to progress at a rate far ahead of the general coatings field in terms of annual growth.

The book opens with a general chapter by Charles E. Hoyle on Photocurable Coatings. This is an excellent contribution, giving both an overview of the whole subject of radiation curing and an introduction to the chemistry of photocuring of polymers. Not only are the chemistry and technology described in a satisfyingly concise way, but the basic principles are clearly enunciated. This chapter is most useful in setting the scene for the more specialist chapters which follow and I really enjoyed reading it.

The second introductory chapter covers Electron-Beam curing of polymers but appears to have been an afterthought. The evidence for this is that the following 1-page Introduction to the section on Photoinitiators talks about chapters 2 to 8. In fact the chapters in this section are actually numbered 3 to 9. This mistake appears throughout the book. Each section is prefaced by an introductory page and every reference to a chapter is as (n - 1) where n is the true number of the chapter. This is, though, a very minor blemish in an otherwise admirable book.

So what are the various sections of this volume concerned with? As mentioned a moment ago, the first main one is about photoinitiators and consists of 7 chapters on a variety of aspects of the subject. These include a number of novel initiators as well as methods of studying their effectiveness.

Next comes a section of 8 chapters concerned with Novel Radiation Photocurable systems. In practice, of course, this section covers aspects of the other half of the photocure system, the polymer. These eight chapters are followed by six on Properties of Radiation-Cured Materials.

One of the features of radiation curable coatings is that they are photosensitive. Despite this their photodegradation has not been widely studied. This omission is rectified to some extent by the two contributions that form the next section, one on the photolysis of bisphenol-A-based model compounds, the other on the stability and yellowing of amine-terminated diacrylate coatings cured by electron-beam and UV.

Radiation curing of cationic polymerisation is discussed in chapters 26 to 29. This is an important technology because the cationic initiation process is not inhibited by the presence of oxygen in the pre-polymer or monomer. There is thus nothing to inhibit surface or through cure of coatings cured by this means. The presence of filler, too, causes fewer difficulties with cationic systems than with free-radical systems.

Next comes a little section of just two chapters on laser initiated polymerisation. Both chapters discuss the cure of acrylic polymer systems and cover the use of both pulsed and continuous lasers. A variety of monomers, oligomers and photoinitiators are mentioned showing the versatility of lasers as a means of curing coatings.

The book concludes with a section covering High-Energy Radiation Curing. The first chapter covers the use of gamma rays to initiate cationic polymerisation of divinyl ethers. The remaining four chapters in this section cover different aspects of electron-beam curing of coatings. These include caprolactone-allyl glycidyl ether copolymers, perfluorinated acrylates and graft copolymers of polyolefins.

My overall impression of this book is of an excellent compilation of papers covering up-to-date findings on initiators, polymers and test methods appropriate to the field of radiation curing of surface coatings. My reviewer's copy has already been the source of new ideas for me and my research group, which in itself is a considerable endorsement. There is no doubt in my mind that if you are at all involved with any aspect of radiation curing of coatings you will find this book a great help to your work. Accordingly I recommend it strongly.

Dr John Nicholson ■

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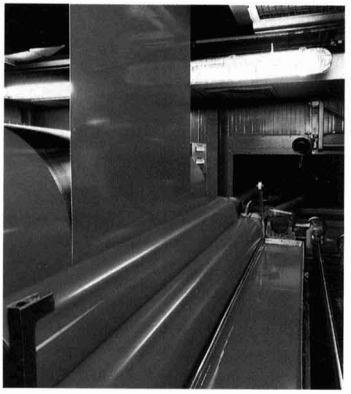

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Back to basics:

Durability and titanium dioxide pigments

by J. G. Balfour, Tioxide UK Ltd, Central Laboratories, Portrack Lane, Stockton-on-Tees, Cleveland, TS18 2NQ, UK

Summary

Durability is a topic which is of considerable importance to the surface coatings industry and has been the subject of many learned papers. The object of this paper is to give a basic overview of the mechanisms involved in the durability of coatings with a particular emphasis on the role of titanium dioxide pigments. It is particularly aimed at newcomers to the industry and describes principles without including many details of the complex physics and chemistry involved.

What is Durability ?

The "throwaway" era is coming to an end. With the increase in awareness of the problems associated with our environment, conservation of materials and resources is now rated high in the list of industrial priorities. Pigmented materials such as plastics and surface coatings have, on the whole, been made to last, but the emphasis towards longer lasting products is on the increase so the subject of durability is becoming more prominent. Thus, a knowledge of the basics of durability must be an advantage to the product formulators.

Paint is normally applied for two reasons: decorative and protective. The relative importance of the two will obviously vary depending on the application. A decorative gloss paint is applied to your front door and you expect it to remain glossy for some considerable time. Redecoration can be expensive and, unless you are a DIY fanatic, it can mean an unwanted job! Maintenance of appearance is the primary concern in this instance, but remember – whilst the paint is intact it is protecting the door. An example where the protection is of most consequence is a marine coating for the hull of a ship. Appearance is not the primary concern in this case!

The durability of a coating can be considered as the extent to which it resists degradation caused by the surrounding environment. In the context of this article, degradation is considered in terms of the effect of weathering on properties such as gloss and chalking, as the effect of pigmentation with titanium dioxide is more pronounced in these areas. Mechanical breakdown (cracking, blistering, etc) and specialised products (eg anti-corrosive paints) are not considered.

Degradation of unpigmented films

Before considering how titanium dioxide influences the degradation processes it is useful to see what happens in its absence.

The biggest contributor to degradation is ultra violet light. This is radiation emitted by the sun which is sufficiently strong to be able to break down chemical links in polymers.

Figure 1 shows the amount of radiation reaching the surface of the earth from the sun in the ultra violet and visible regions of the spectrum. This varies from place to place and is dependent on factors such as the local weather conditions and the time of year. However, it gives an indication of the relative intensities of the different wavelengths. It can be seen that there is a lot less ultra violet than visible light. It is important to remember that the lower the wavelength, the higher is the energy. So, whilst there is less ultra violet present, the energy of what is there is sufficient to be the major cause of degradation.

The earth's atmosphere absorbs a large quantity of the radiation from the sun and Figure 2 shows the relative amounts above the atmosphere compared with that at the surface of our planet. Virtually all the radiation below 300 nanometres is filtered out and a vast proportion above this wavelength as well. Without this filtering, problems with durability would be much greater.

Figure 1
Relative intensity of sunlight at the surface of the earth

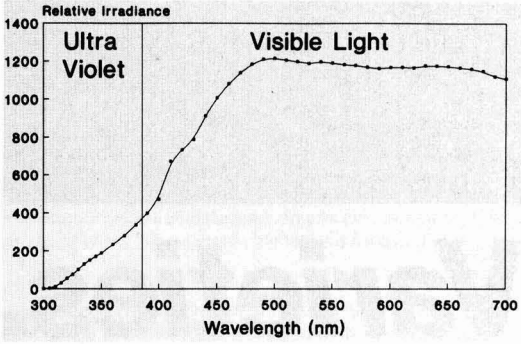


Figure 2
Relative intensity of sunlight

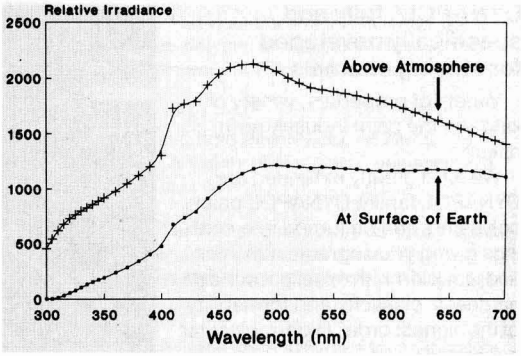
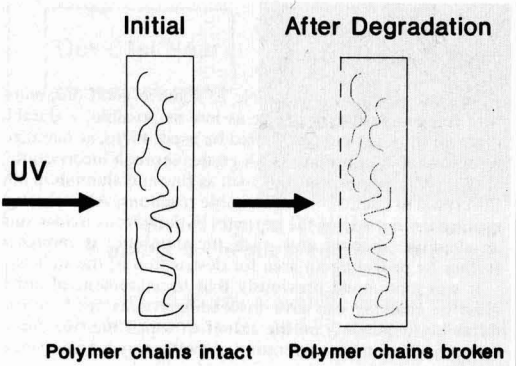


Figure 3 is a schematic diagram which shows that, in an unpigmented film the ultra violet radiation is absorbed by the polymer forming the medium and the chains are destroyed. As a result, the film is broken down. This effect is known as photochemical degradation. The energy required to break

down a polymer bond depends on the strength of the bonds so that the rate of degradation, in turn, depends on the chemical type of the binder. In order for degradation to occur in this way the binder must absorb the ultra violet. If no absorption takes place, the radiation merely passes through causing no damage. Thus, the rate of degradation depends on the wavelength (shorter wavelengths having a greater effect) and amount (intensity) of the ultra violet as well as the absorption and chemical nature of the binder.

Figure 3
Effect of UV light on unpigmented film



This principle of absorption being required for degradation is illustrated in Figure 4. This shows the amount of ultra violet which is absorbed by a certain alkyd film before and after exposure to weathering. Initially there is a high absorption which means there is the possibility for degradation. After exposure there is much less absorption, indicating that chemical changes have taken place. This type of alkyd resin is known to give poor durability.

Figure 4
Absorption of alkyd film before and after exposure

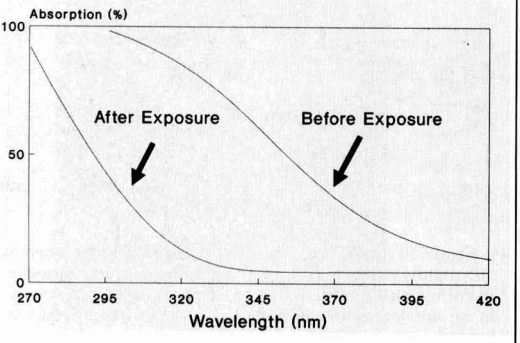


Figure 5 is similar except that it is for an acrylic resin. There is very little absorption before or after exposure. This acrylic is noted for its good durability.

Degradation of films pigmented with titanium dioxide

Through absorption of the ultra violet radiation by the medium, degradation can occur by the photochemical mechanism described above. If, however, some material is incorporated into the system which preferentially absorbs the harmful radiation the medium is protected and will be less susceptible to degradation.

Figure 5
Absorption of acrylic film before and after exposure

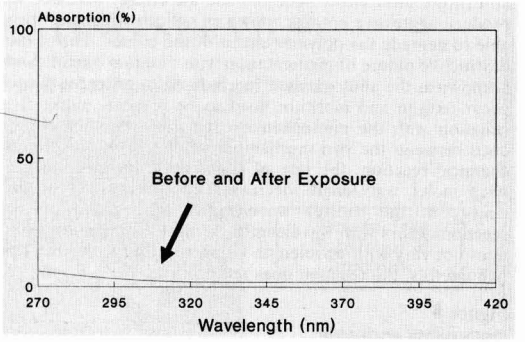


Figure 6
Protection of the medium by UV absorption

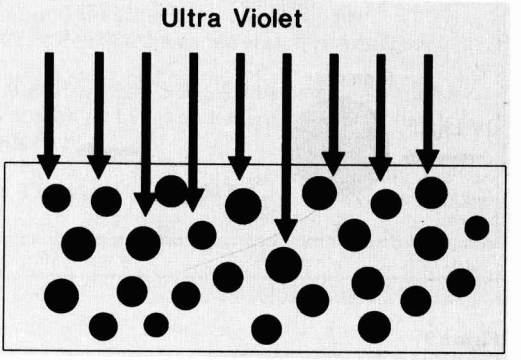
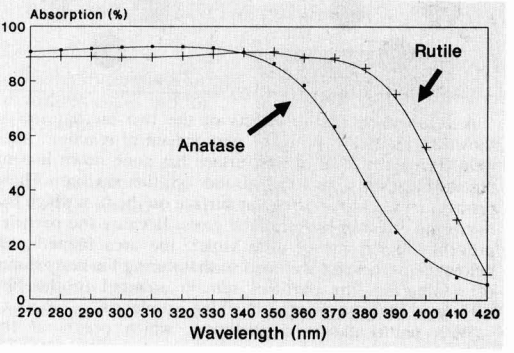


Figure 6 is a schematic representation which shows a paint film containing titanium dioxide pigment which does absorb. Thus, in the absence of other effects to be dealt with later, the pigment absorbs the ultra violet light and protects the binder.

Figure 7 shows how effective titanium dioxide is at doing this. It shows the absorption for two crystal forms of the pigment, anatase and the more commonly used rutile. Throughout most of the ultra violet region the pigments are effective absorbers, especially the rutile which is the more durable of the two.

Figure 7
Absorption of titanium dioxide



There is, however, another mechanism which can occur, schematically described in Figure 8. The titanium dioxide can absorb the ultra violet light and use the energy obtained to produce aggressive entities known as radicals which are then able to degrade the polymer chains of the binder. This is the destructive nature of pigmentation with titanium dioxide and is known as the photocatalytic mechanism. In order for this to occur oxygen and moisture need to be present, unlike the situation with the photochemical reaction. Another difference between the two mechanisms is that, with the photochemical reaction, the rate of degradation increases as the ultra violet wavelength decreases because of the higher energy at the shorter wavelengths. The photocatalytic reaction occurs with wavelengths below 405 nanometres but does not vary with wavelength below this figure. Above 405 nanometres, the reaction does not occur.

Figure 8
Photocatalytic mechanism

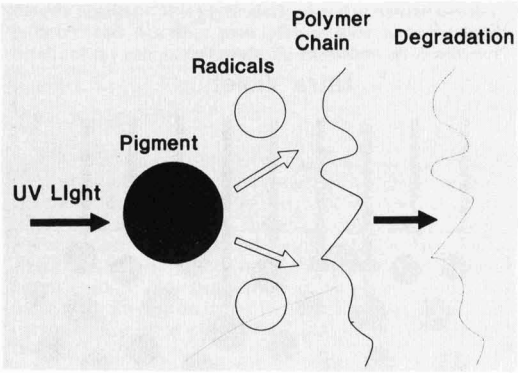
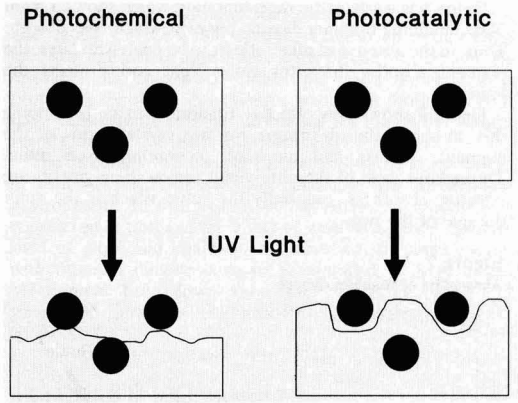


Figure 9
Comparison of degradation mechanisms



A comparison of the effects of the two mechanisms is shown in Figure 9. With the photochemical reaction, after weathering, the level of the surface has gone down leaving pigment particles standing proud on the surface. These protrusions lead to an irregular surface on the film which has the visual effect of lowering the gloss. Because the particles prevent the passage of ultra violet, the area immediately below the particles is screened so that during the early stages of weathering, the particles remain adhered to the film surface so that the onset of chalking is delayed.

With photocatalytic degradation, which occurs at the surface of the pigment, the area around the particle is affected and, because the surface remains fairly flat, the effect on gloss is not so pronounced and remains higher than with the other mechanism. However, because the photocatalytic reactions occur all the way round the particles they are no longer adhered to the surface and consequently will show a degree of chalking earlier in the degradation process than with the photochemical mechanism.

Obviously, in most real systems both mechanisms will operate. Which of them predominates, if either, will depend on the choice of pigment and resin and various other factors to be dealt with later.

Effect of pigment grade

In cases where it is desirable to achieve good durability, with the photocatalytic effect as low as possible, a durable grade of titanium dioxide should be used. Pigment manufacturers are able to produce such grades through incorporating very small amounts of metals such as zinc and aluminium into the crystal to quench the undesirable reactions. Also, through coating the outside of the particles with hydrous oxides such as alumina, zirconia and silica the durability is improved further by providing an area for destruction of the radicals.

It was mentioned previously that measurement of either gloss or chalking can give misleading results for assessing durability depending on the extent to which the two breakdown mechanisms are operating. A better way for assessment is to measure the weight loss of a film which acts as a useful means for measuring the total effect.

Figure 10
Effect of pigment grade on durability

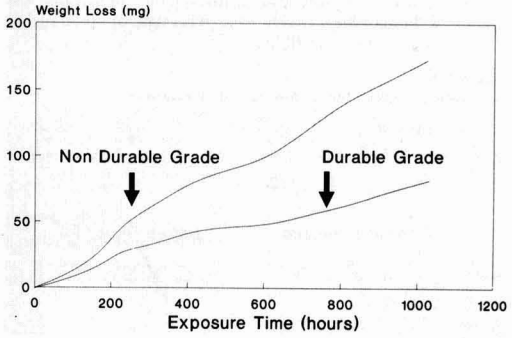


Figure 10 shows the difference in performance between two alkyd gloss paints, one pigmented with a durable grade and the other with a non durable grade. The paints were exposed in an accelerated weathering machine and the weight loss from the panels measured at regular intervals. It can be seen that the durable pigmented paint is lasting much longer, the rate of loss of weight being less than half that of the other paint. To give some perspective to the numbers, the durable paint lost about 10% of its total weight and the non durable one about 25%. This also demonstrates the considerable influence that the photocatalytic degradation can have as, in this case, the protective nature of the pigments in terms of the photochemical effect is likely to be similar in the two paints.

As well as the type of pigment used, it is important to consider the effect of concentration. The effect depends on which mechanism is predominant. Within certain limits, if the pigment is acting as a protector, the higher the concentration,

the better will be the durability. On the other hand, if the pigment is acting as a destroyer, the reverse will be true.

Effect of film thickness

Another factor affecting durability is the initial film thickness. An example is shown in Figure 11 where films of different thickness were weathered and the weight loss determined. In this case, an alkyd gloss paint pigmented with titanium dioxide, the rate of loss of weight was proportional to the initial thickness up to a thickness of 20 microns, above which the rate of weight loss became independent of thickness.

Figure 11
Effect of film thickness on durability

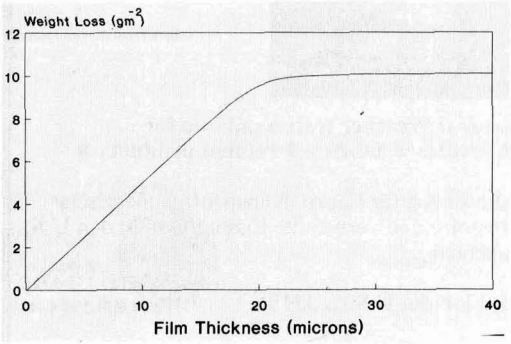
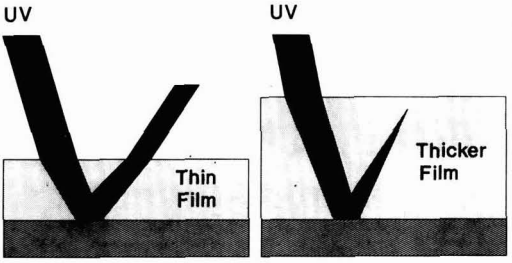


Figure 12
Effect of film thickness on UV absorption



This effect is related to the path length of the ultra violet within the film. In the diagram, Figure 12, with the thin film, after the radiation has entered the film, some of it is absorbed by the pigment and resin whilst the rest may be reflected by the substrate and, after further absorption, emerge from the surface again. However, with the thicker film, nearly all the radiation is absorbed and is consequently available to do more damage. Any further increase in thickness will have little effect as there is no more radiation left to be absorbed. The limiting film thickness, above which the degradation rate is independent, is related to the intensity of the radiation, the absorptivity of the medium, the nature of the substrate and the amount absorbed and reflected by the pigment.

Effect of pigment dispersion

Another important aspect is the state of pigment dispersion. When pigment is initially incorporated into a resin, the pigment particles are subjected to forces designed to break them down into individual crystals during the milling operation. This ideal situation is never achieved. There are always some aggregates present. However, if the dispersion is poor and the number of aggregates and agglomerates is large, they will protrude through the surface of the film giving poor gloss. When the surface is eroded during weathering, this effect becomes more pronounced and the rate of loss of gloss becomes much greater than with a paint in which the pigment particles have been efficiently dispersed. Also, if agglomerates are present which contain clumps of pigment, with the individual particles incompletely wetted out, these can form the centres for mechanical breakdown because of poor film integrity. Normally, this kind of poor dispersion is a result of inefficient milling conditions or insufficient milling time and can be simply checked by examining the paint on a fineness of grind gauge.

A more subtle problem is that associated with pigment flocculation. In this situation, the pigment may be well dispersed initially but the particles come together to form loosely bound clusters known as flocculates. This is a very common phenomenon but is more difficult to detect. A fineness of grind gauge is unlikely to determine whether a paint is flocculent or not. A technique is available for quantifying this effect whereby measurement of a value known as a flocculation gradient is useful. The higher the value, the greater is the degree of flocculation.

Figure 13
Effect of flocculation on gloss retention

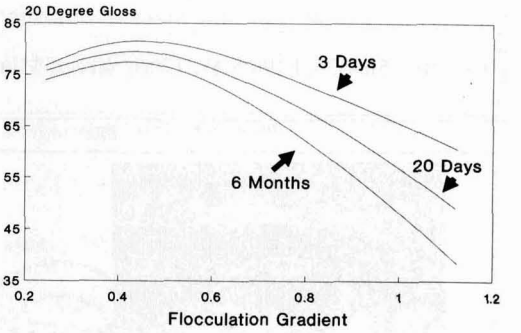


Figure 13 shows the effect of flocculation on gloss retention. To put the numbers in perspective, the range of flocculation gradient values found in commercially available white gloss paints lies between about 0.2 and 0.9. The figure shows what happens to three gloss paints with differing degrees of dispersion in the mildest of exposure conditions. The panels were left in a laboratory under controlled temperature (20°c) and humidity (50%) and the gloss measured after three days, 20 days and six months. Even under these mild conditions the loss of gloss with increasing flocculation was very significant.

As is predictable, the effect under more severe conditions is more dramatic. Figure 14 shows what happens to paints with differing levels of flocculation during exposure in an accelerated weathering machine for 1000 hours. In this case the paints have flocculation gradients between 0.29 and 0.50, in other words they represent the "top of the range". There is a sharp decline in gloss over the first 300 hours with the most flocculent of the paints losing nearly 50 units and the best losing about half this value. The curves flatten out after longer exposure times because, especially with more flocculent paints, there is little gloss left to lose. This loss of gloss occurs when the degradation begins and the particles become exposed and protrude from the surface. With the poorly dispersed paint, the flocculates are large and, after degrada-

tion begins, they protrude to a greater extent and the rate of loss of gloss is much greater.

It was mentioned earlier that film thickness can have an effect. Figure 15 shows that the effect of decreasing the film thickness causes a greater reduction of gloss with flocculent than with dispersed paints. This particular graph refers to initial gloss before any weathering has taken place. The results are to be expected because flocculates will protrude to a greater extent from a thin film than from a thicker one. However, when degradation takes place this difference becomes even more exaggerated and a combination of a flocculated pigment and a thin film will lead to a rapid rate of loss of gloss.

Figure 14
Effect of flocculation on gloss during weathering

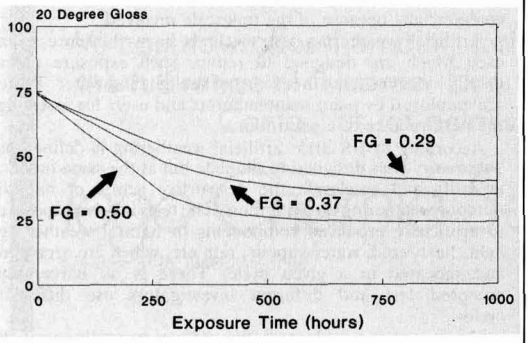


Figure 15
Effect of film thickness and flocculation on gloss

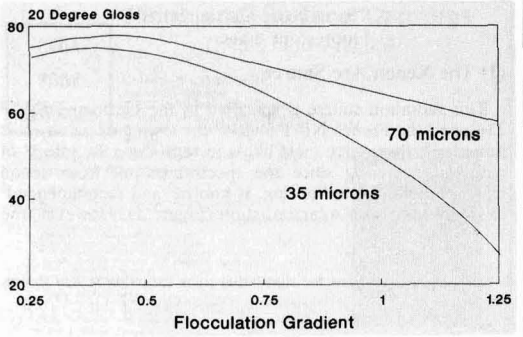
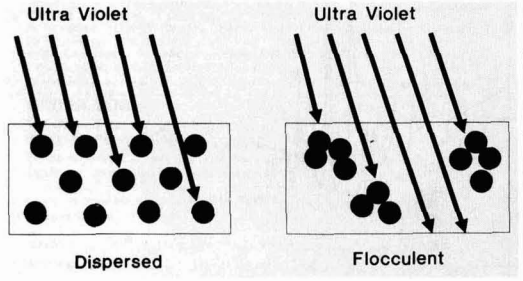


Figure 16
Effect of flocculation on UV absorption



The overall effect of flocculation will depend on which of the two degradation mechanisms is predominant. In Figure 16, the paint on the left side is well dispersed with the particles well separated. As a result, they are able to absorb the incident ultra violet efficiently. In terms of protecting the surrounding resin this is a healthy situation but if the photocatalytic mechanism is operating then there are more sites available for degradation to begin. With the flocculent paint, the individual pigment particles are clustered together and absorb the radiation less effectively. Thus, they are not as efficient at protecting but offer less sites for the onset of photocatalytic degradation. The overall effect is also dependent on the susceptibility of the resin to the two forms of attack.

This is, however, a theoretical situation. In practice flocculation should be minimised as it is wasteful not to do so. When high durability is required a well dispersed durable pigment is the answer.

Conclusion

The concepts of durability of paint films have been considered with respect to the effects of pigmentation with titanium dioxide. Various factors influencing these concepts have been discussed.

Acknowledgement

The author would like to acknowledge the permission of the directors of Tioxide UK Ltd to publish this paper. ■

European artificial weathering standards

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Abstract

Artificial weathering methods are widely employed by paint manufacturers and users to test the durability of surface coatings. In outdoor conditions sunlight or its ultra-violet component is the principal factor responsible for the destruction of materials while water enhances the breakdown process. The manipulation of the intensified components of sunlight together with water in a cyclical process therefore form the fundamental features of weathering devices designed to accelerate the degradation process.

A number of standard procedures are available for assessing the performance of a paint film in artificial environments. The four main specified radiation sources discussed include the carbon arc, xenon arc, mercury arc and fluorescent tubes in BS 3900:Part F3, DIN 53 231, NF T 30-049 and SAE J 1960, BS AU 148:Part 12 and the ASTM G53 standards respectively. However, none of these methods have gained universal acceptance, although the US based SAE J 1960 and ASTM G53 are popular in Europe because of their extensive support in the US.

The use of such diverse range of radiation sources can be attributed to the difficulty in correlating paint degradation data between artificial tests and natural weathering. The goal for a universal method which accelerates and reproduces the degradation occurring in natural conditions is yet to be achieved. However, in the meantime there is a requirement for a generalized European standard which accommodates the various light and water cycles.

The surface of this planet is continuously being subject to the natural elements of sunlight, rain, wind and snow and these are the principal factors responsible for deterioration of most materials including surface coatings. Subsequent loss of the protective function of such coatings makes the substrate more vulnerable to corrosion and erosion, which together with an unacceptable change in appearance reduces the service life of the coated object. Sunlight and especially its ultra-violet component is known[1] to be the single most important agent in causing degradation in coating materials – a process enhanced in the presence of water.

The introduction of a new paint product therefore requires the acquisition of data on its 'durability' which is defined[2] as 'the degree to which surface coatings withstand the destructive effects of the weather'. The most reliable method of testing durability is prolonged exposure of the material to the natural elements. However, this method is rarely viable commercially because of the timescale involved.

Artificial weathering test methods have therefore been used which are designed to reduce such exposure times mainly by intensifying the effects of sunlight and water. These are employed by paint manufacturers and users for assessing the performance of a paint film.

According to BS 2015[2] artificial weathering is defined as 'laboratory tests designed to simulate but at the same time to intensify and accelerate the destructive action of natural outdoor weathering on paint films. The tests involve exposure to artificially produced components of natural weather eg light, heat, cold, water-vapour, rain etc, which are arranged and repeated in a given cycle. There is no universally accepted test and different investigators use different cycles'.

The four main popular radiation sources currently specified in national standards for artificial weathering devices are:

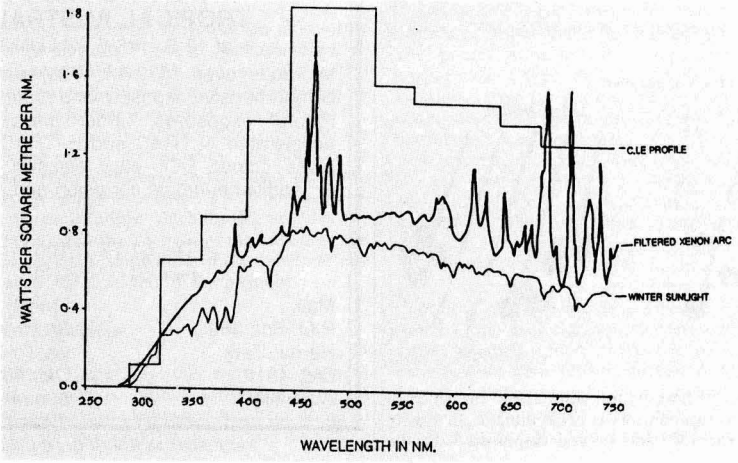
(1) The xenon arc source
(2) The carbon arc source
(3) The mercury arc source
(4) Fluorescent tubes

(1) The Xenon Arc Source

This radiation source is specified in the German DIN 53 231[3], and the French NF T 30-049[4] for testing of paints. It is potentially the source most likely to reproduce the effects of natural weathering since the spectral output from xenon lamps, after suitable filtering, is known[5] and recommended[6] to simulate natural solar radiation (Figure 1). However, the

Figure 1

Spectral energy distribution of winter sunlight and xenon arc compared. CIE recommendations for simulating solar radiation is also shown.



requirement for high humidity and the prescribed cycles of light and rain phases in the DIN and NF standards (Tables 1 and 2 respectively) will need further supporting evidence regarding correlation with natural weathering before either could be considered for adoption as an ISO standard.

Many European automotive manufacturers following US practice use the US BASED SAE J 1960[7] (Table 3) which similarly specifies the use of xenon lamps. This differs from the German and French standards in the proposals for light and rain cycles.

(2) The Carbon Arc Source

The carbon arc is employed in weathering machines complying with BS 3900:Part F3[8] which was proposed after extensive studies carried out by the Joint Services Research Committee on Paint and Varnishes. Although the spectral profile of the enclosed carbon arc differs from that of natural radiation, this method (Table 4) has been shown to be particularly suitable for testing oil modified-alkyds and oleoresinous paints, the effects correlating well with those occurring on prolonged outdoor exposures. While this procedure will break down other resin systems, the resulting degradation often correlates poorly with natural exposures.

Another variation of BS 3900:Part F3, the twin carbon arcs according to the Japanese JIS B 7752, is specified by Nissan for testing resistance to artificial weathering of vehicle paints.

(3) The Mercury Arc Source

This source was specified in BS AU 148:Part 12[9] after an exhaustive study of enamels for automotive applications by the Society of Motor Manufacturers and Traders Ltd. The methods recommended (Table 5) are varied to produce required degradative changes depending on the choice of test.

(4) Fluorescent Tubes

Irradiation by this type of source together with the use of water as condensation, form the basis of artificial weathering devices conforming to ASTM G 53[10]. The fluorescent/condensation machines are particularly favoured by paint manufacturers and users for testing purposes because of their cheapness in running and maintenance costs. Furthermore,

Table 1
Outline of DIN 53 231 procedure

	DIN 53 231
Radiation Source:	Filtered xenon arc light
Irradiance:	280-800 nm – 550 w/m^2 Relative Irradiance: 280-400 nm – 11 ± 2% 280-800 nm – 100%
Method:	2 h (120 min.) cycle of: (1) 18 mins water spray with light on (2) 102 mins dry period Relative Humidity during dry phase: 60 – 80% Black Standard Temperature during dry phase: 63-67°C

Table 2
Outline of NF T 30-049 Procedure

	NF T 30–049
Radiation Source:	Filtered xenon arc light
Irradiance:	300-400 nm – 65 w/m^2 300-800 nm – 550 w/m^2
Method:	4 h (240 mins) cycle of: (1) 30 mins water spray in the dark (2) 60 mins freezing at −20°C in the dark (3) 60 mins RH at 95% ± 5% and a temperature of 55°C ± 2°C in the dark (4) 80 mins light phase at 60°C ± 2°C and RH of 20% ± 5% 10 mins to stabilise the machine for repeating the cycle.

Table 3
Outline of SAE J 1960 procedure

	SAE J 1960
Radiation Source:	Filtered xenon arc light
Irradiance:	0.55 w/m^2 at 340 nm
Method:	3 h (180 mins) cycle of: (1) 40 mins light only (2) 20 mins light and front specimen spray (3) 60 mins light only (4) 60 mins dark with back specimen spray Black panel temperature: 38°C (dark phase), 70°C (light phase). RH 95% ± 5% (Dark phase), 50% ± 5% (light phase).

Table 4
Outline of BS 3900:Part F3 procedure

	BS 3900:Part F3
Radiation Source:	Filtered and enclosed carbon arc
Method:	23 h cycle + 1 h for maintenance (1) 4 h water spray with light (2) 2 h light phase only (3) 10 h spray with light (4) 2 h light phase only (5) 5 h water spray with light

Table 5
Outline of BS AU 148:Part 12 procedure

	BS AU 148:Part 12
Radiation Source:	Mercury arc and infra-red lamps.
Method 1:	9 h cycles of: (1) 4 h infra-red plus UV emission plus water spray (2) 1 h water-spray only (3) 4 h infra-red plus UV emission plus water spray Ambient temperature: 74°C ± 1°C Repeated three times (total test time 27 h) to produce chalking, gloss and colour changes.
Method 2:	24 h cycles of: (1) 6 h infra-red plus UV emission plus 45%-85% RH (2) 2 h dark and dry phase (3) 7 h infra-red plus UV emission plus 45%-85% RH (4) 9 h dark and dry phase Ambient temperature: 60°C ± 1°C Repeated ten times (total test time 240 h) to produce micro-checking.

flexibility in the choice of lamps permits the use of tubes with output in different UV regions (Table 6). In Europe, the DIN 53 384[11] allows lamps with visible light unlike the restriction to UV light in ASTM G 53. The UK based BS 2782:Part 5: Method 540[12] also specifies the use of the fluorescent/condensation type of machine. However, both the DIN 53 384 and the BS 2782 methods are designed to test plastics.

This type of machine is very likely to be more widely applied in paint testing in the future as further evidence is acquired on correlation with natural weathering. For instance, the recent draft BS 1722:Part 16 specification[13] for powder coatings applied to fence components requires the use of a fluorescent/condensation type apparatus specified in BS 2782:Part 5 for testing resistance to artificial weathering.

Conclusions

The existence of such diverse methods for standards for artificial weathering in Europe can largely be attributed to the difficulty in correlating data from these sources with natural weathering. However, while considerable support is found for adopting the ASTM G 53 machines, the xenon arc is preferred because of its closer approximation to natural sunlight. Although the International Organisation for Standardization (ISO)[14] has yet to decide on a particular radiation source, the German members of ISO are, however, pursuing the use of xenon arc adopted in the DIN 53 231 as an international standard.

While a universal artificial weathering test for predicting and accelerating the effects on all paints to all types of natural exposures may never exist, there is a requirement for harmonisation of the various radiation sources and the methods used in this field as one generalized European standard.

Table 6
Fluorescent lamp characteristics

Lamp Type	Peak Output
A	317 nm
B	355 nm
C	374 nm
D	365 nm
E	313 nm

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Continued on p.492

Weathering studies: Coating evaluation in the 90's

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Abstract

Over the past decade, the number of coatings manufacturers in the world has steadily declined. Large coatings companies have purchased smaller ones. Some of the small companies have merged to make new units, or simply shut their doors. During the same period of this decline, the methods for evaluation of weather related failures have been rewritten and redefined sometimes within the new companies, and sometimes in large associated groups such as ASTM, BIS, ISO, and DIN.

As the European Community readies for 1992, new standards are being written almost daily. There is a need to consider some common reporting scales and definitions for rating performance of materials exposed to weathering so that there can be an overall enhancement in the exchange of information from company to company, and from market to market. The following article is meant to explore some of those scales and practices presently used, and offer some recommendations for common ground in reporting. The article is not to be considered an end all but rather a beginning for further consideration as committees sit down to write new standards.

Introduction

The last two decades has underscored the name given the '90s as the "Age of Information". With computers, telefax, television, and ovenight delivery of small parcels virtually world wide, our expectation for immediate answers has become commonplace. In this same period, the demands on materials for longer durability and higher quality has forced coatings manufacturers to test their products for longer periods documented by more detailed reports. As the exchange of this information becomes more global and data base oriented, there is a need to examine some of the standard practices of weathering and reporting material performance.

The best method for evaluating the performance of a material during exposure is for the interested party to actually see the specimen as it is being tested. In many instances, however, the person most interested in the durability of the material is not the one conducting the exposure test, and seeing the specimen becomes impractical. Therefore, it is necessary to trust the examination of the test material to an individual who may not be directly related to the product development.

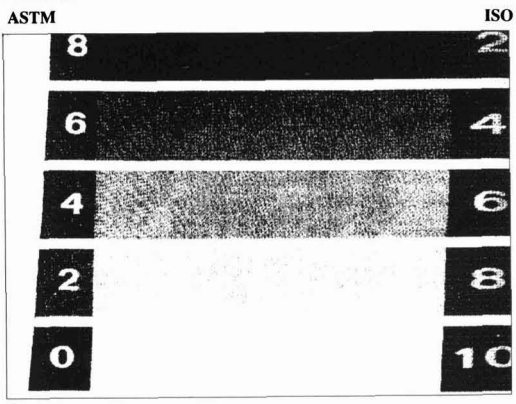
Everyone placed in the position of having to rely on another's judgment for the evaluation of a material's performance must be aware that differences will exist in ratings by one observer at different times or between different observers at the same time. In order to minimize the differences, certain criteria may be established, such as specific training and color aptitude tests for inspectors, standard viewing conditions, examination procedures, and photographic references. Even then much confusion may exist due to differences in rating scales and interpretation of the scales and terminology used.

While the American Society for Testing and Materials (ASTM), the Federation of Societies for Coatings Technology (FSCT), and the International Organization for Standardization (ISO), have excellent examination procedures, not all the systems have a common terminology with definitions to match the ratings of many of the failure modes.

Rating scales

A good example of this problem may be found in ASTM D 4214, Evaluating the Degree of Chalking of Exterior Paint Films[1]. The ASTM scale lists 10 as no development and O as failure, while the Paint Research Institute, TNO scale is the inverse, O to 10. The ISO scale is one half of these two scales and reads O as unchanged and 5 as severe (see Table 1). Look further into D 4214 and one finds a table for reflectance readings for the instrumental measurement of chalk and the scale is expanded to have 21 rating points from 10 to O with half steps between each number, e.g., 9.5, 8.5, 7.5, etc.

Table 1



A similar difficulty may be found when assessing color change visually. In ASTM D 1727, Visual Evaluation of Color Differences of Opaque Material[1], the report section instructs the observer to report "observed direction and magnitude of each of three components of color difference". Other than this remark, no scale is given. Further along, in this volume of ASTM standards, the reader will find D 1150, Single- and Multi-Panel Forms for Recording Results of Exposure Tests of Paints[1]. The scale here is the same 10 to O found in D 4214, but in D 1641, Standard Test Method for Exterior Durability of Varnishes, the scale is 10 to 1. While this is not a real significant difference it forces the reader of any report to know that the failure point for paint is O and for varnish it is 1.

Turn to D 2616, Evaluation of Visual color Difference With a Gray Scale[1] and the designations are in a 5 step scale with intermediate ratings listed as two numbers (see Table 2)[1]. While this is yet another scale, it is common practice to turn the AATCC step designations into a 10 point scale with 5 becoming a 10, the 4-5 rating a 9, and so on.

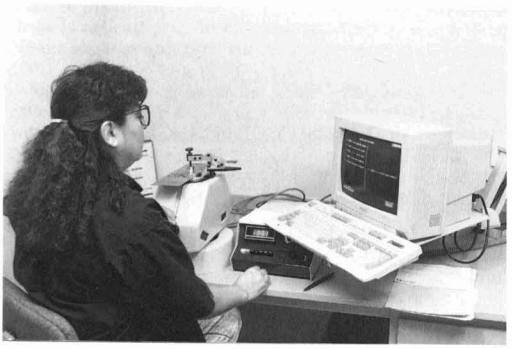
Where using a single numbering system for rating becomes useful is for those who evaluate the materials and subsequently read reports in which several types of coatings may be included in the population. ASTM D 1150, promotes a form that has several evaluation criteria on a single sheet covering a period up to 60 months. If this type of reporting form is to be used, the balancing of evaluation scales becomes

all important. For the computer user, the space savings for a single field can be significant for a large population over time. Reporting color using the gray scale requires three bytes where the 10 to O scale uses only two.

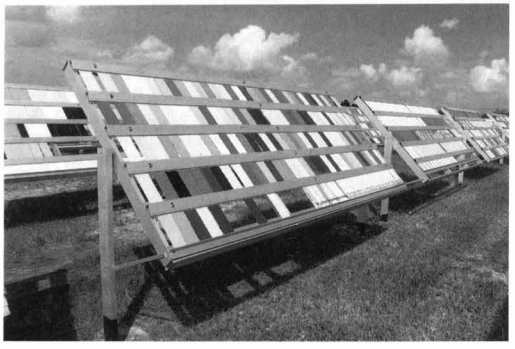
Table 2
Gray scale characteristics

AATCC Step Designations	CIELAB (DELTA E) Units Color Difference	Tolerance (±)
5	0	0.2
4-5	0.8	0.2
4	1.7	0.3
3-4	2.5	0.3
3	3.4	
	0.4	
2-3	4.8	0.5
2	6.8	0.6
1-2	9.6	0.7
1	13.6	1.0

Computerized instruments enhance efficiency and accuracy



Replicate exposures reduce error in long term testing



If a single scale is used in reporting, the only real difference becomes whether it is qualitative or quantitative. The quantitative portion is divided into groups of the observer's estimated degree or amount of difference to a given reference or an actual measurement such as gloss, color or distinctness of image.

For each of the categories there are descriptive terms. The qualitative terms are: As Received, Very Good, Good, Fair, Poor, and Very Poor (failed). The first set of quantitative terms consist of: None, Slight, Moderate, Pronounced, Severe, and Very Severe (failed). The second group of quantitative terms are: None, Very Few, Few, Medium, Medium Dense and Dense (see Table 3)[9].

An example to support the suggestion that a single common scale be used, may be found in the Jordon Award Paper by 1. Melville and L. A. Simpson entitled "Wood finished – field and laboratory tests"[6]. This excellent article describes the exposure and evaluation of a series of exterior finishes. "The exposed panels were tested for gloss, color, dirt retention, erosion, cracking, flaking, chalking, and porosity". The authors are careful to enumerate each of the categories and their respective scales. Values for chalk, dirt retention, flaking, cracking and erosion are all rated from 10=no failure to O=severe failure. Porosity was rated on a scale of 1 to 5 and 60 degree gloss readings 100 to 0. But when it came to color the authors used two scales.

"Colour was assessed in two ways. For white finishes the rating was made against ceramic standards which vary from a very blue-white at the rating of 12 to a brown-white at a rating of 1. For non-white samples standards were kept unweathered for comparison. The exposed panels were compared with the internal standards and rated as being darker, slightly darker, similar, slightly lighter, or lighter than the standard, and according to these ratings assigned values from O to 4 respectively"[6].

The result of this introduced scale finds the authors commenting that "Colour measurements are difficult to compare because two different scales were used for white and non-white samples". In reality, the introduced color scales make it difficult to compare color to any of the other ratings, because the summarized data of averaged performance of the white panels have three fold importance in color ratings over the non-white and no real relationship to a 10 to O scale used to rate the other categories.

The support for a common reporting scale can be found in numerous articles. A summarization of this view is found in the July, 1987 Versailles Project on Advanced Materials and Standards[8].

"Standards play a key role in the computerization process of materials data for two reasons:

1. The performance properties and designation of engineering material are generally not defined by scientific laws and principals but rather by procedures and practices that have been codified as standard tests. Consequently, the information related to these standard tests must be adapted to computerization.

2. The interconnection of computer equipment and software has been so hampered by proprietary concerns that mutually agreeable practices have become necessary"[8].

As the use of LIMS, Laboratory Information Management Systems, expands, the enhancement to understanding the changes in weathered specimens is all too obvious. Data transfer may be done by disk or modem. Depending upon the design of the system, additional information about formulas, past exposures, and new and old systems can all be compared.

For studies reviewing the influence of a specific environment, or correlation of laboratory accelerated exposure to real time weathering, the advantages are endless. Chalk ratings may be plotted against solar irradiance and wet time, or as a function of temperature. Top coat cracking studies may be reviewed to see which type of exposure gives the greatest acceleration with highest correlation to service use. Real time weathering data may be used to program accelerated laboratory equipment for specific in service environments. To make the advantages of computer based reporting pay back at the highest level of return, common standards and scales become a must.

Viewing conditions

Assuming that single scale for reporting is desirable, it is equally important to look at the differences in viewing conditions. Good examples are ASTM D 1729, Visual Evaluation of Color Differences of Opaque Materials[1]; Society of Automotive Engineers, (SAE), method J361, Method for Determining Visual Color Match to Master Specimen For Fabrics, Vinyls, Coated Fiberboards, and Other Automotive Trim Materials[4]; and ISO method 3368, Paints and Varnishes – Visual Comparison of the Colors[3]. Each of these methods list specific Munsell System values for the background and surrounding of the viewing conditions. ASTM D 1729 shows general background as N5/ to N7/, SAE J361 as N7/7, and ISO 3668 as N/4 to N/5. Literally, we are talking about shades of gray, but the hope would be that

Table 3

South Florida Test Service Inspector's Manual: SFTS – ASTM/FSCT – ISO Rating Scales and Terms

The following correlations have been established to assist the reader in converting SFTS terms and rating scales into proposed ASTM, FSCT or ISO equivalents. It must be remembered that the tables presented in ISO 4628/1 and many of those used in this manual were developed for evaluating the degradation of paint coatings only, even though we apply them to other materials.

SFTS	FSCT	ISO
QUALITATIVE		
10 As Received	Perfect	ISO 4628 does not present a table which applies
8 Very Good	Good	
6 Good	5 Intermediate	
4 Fair		
2 Poor	Poor	
0 Very Poor (failed)	Poorest degree conceivable	
QUANTITATIVE	**FAILURE**	**INTENSITY OF CHANGE**
10 None	Absent	0 Unchanged
8 Slight	Slight	1 Very Slight
6 Moderate	5 Intermediate	2 Slight
4 Pronounced		3 Moderate
2 Severe	Bad Failure	4 Considerable
0 Very Severe (failed)	Complete Failure	5 Severe
FREQUENCY		**QUANTITY OF DEFECTS**
10 None		0 None
8 Very Few		1 Very Few
6 Few		2 Few
4 Medium		3 Moderate
2 Medium Dense		4 Considerable
0 Dense		5 Dense
	AREA (PER CENT)	
10 None		0 None
8 0.1		1 0.05
6 1.0		2 0.5
4 10.0		3 1.0
2 33.0		4 8.0
0 50.0+		5 40/50.0
SIZE		**SIZE OF DEFECTS**
10 None		0 Not visible under 10X magnification
8 Smallest size easily seen with unaided eye		1 Only visible under up to 10X
6 1 mm		2 Just visible with corrected vision
4 2-3 mm		3 Clearly visible up to 0.5 mm
2 4-5 mm		4 Range 0.5 up to 5 mm
0 Greater than 5 mm		0 Larger than 5 mm

when one method lists another society practice as a reference as is the case in ISO 3668, the numbers would be the same.

The same methods listed above contain differences in illumination. The ISO method lists a minimum of 6504 K (CIE Standard Illuminate D65), while both the ASTM and SAE methods agree upon 2300 K. Aside from measuring the intensity of the light in the viewing area, the caution found in ISO 3668 is well founded.

"Specification and control of illuminates present difficulties and there is as yet no internationally agreed method for checking that the illumination in a viewing cabinet has a spectral composition sufficiently close to that of CIE Standard Illuminate D65"[3].

Add to this caution the news that committees like ISO TC61 are reviewing a new CIE standard number D85 to replace D65, working to keep the observer informed and in the correct viewing conditions for evaluating samples becomes an ongoing process.

Specular gloss measurements

With the introduction of more computerized instruments into the laboratory environment, the addition of direct reading gloss instruments was only a matter of time. The increase in efficiency and accuracy is an obvious benefit, but there is at least one development that deserves reporting. Newer gloss instruments will read and report readings over 100.

Since specular gloss is the relative luminous fractional reflectance of a specimen at a specular direction, the gloss value is actually a percentage reflectance from a specularly perfect reflector. Referring to Table 2 of ASTM D 2467, Method for Specular Gloss of Plastic Films[1], one can see that the value for a perfect mirror at 20 degrees is double the value at 45 or 60 degrees.

Routinely, the instrument calibrated for ASTM D 523, Method for Specular Gloss[1], is tied to a specified geometry, and operated according to the manufacturer's instructions. Measured gloss ratings by this test method are obtained by comparing the specular reflectance from the test specimen to that from the black glass standard used in the calibration process. Test surfaces having greater specular reflectance than that of the standard may result in readings of greater than 100. While values of above 150 may be suspect, the manufacturers represent that the readings are linear to this level.

To insure that these higher numbers are representative of the specimen's condition, it is helpful to check the suggested uses of the different geometries. The 60 degree geometry is used for intercomparing most specimens and for determining when the 20 degree geometry may be more applicable. The 20 degree geometry is for comparing specimens having 60 degree gloss higher than 70. The 85 degree geometry is used for sheen or near-grazing shininess, and is most frequently applied when specimens have 60 degree gloss values lower than 10. For surfaces having a very high relative luminous reflectance factor, other options may be suitable, such as ASTM 4039, Method for Reflection Haze of High-Gloss Surfaces[1], or E 430, Method for Measurement of Gloss of High-Gloss Surfaces by Goniophotometry[1].

The scale of earlier analog gloss meters was 0-100, and any test specimen having a specular gloss of a higher value merely peaked the meter. The new digital meters capable of reading the over 100 gloss units should not be confused with historic data.

Specimen size

The next challenge for the observer is one that has come from growing consumer demands for more durable products. By and large, exposure periods for weathering have been getting longer. It is common for a coil coat specimen to be on exposure in southern Florida for 10 years at 45 degrees. This is approximately twice the time this product was tested only 10 to 15 years ago. Automotive finishes are now exposed for 2 to 5 years, PVC building products see 4500 hours of Weather-Ometer® exposure, and textiles used in automobiles have more than doubled the length of time they are exposed to sunlight testing.

The size of the specimen for most coatings studies has not changed, nor has the frequency with which evaluations are made. Where chalk ratings, color evaluations, specular gloss readings, and other categories such as flaking, and adhesion are all taken from one specimen with any regularity, lengthening the test builds in inaccuracy. There simply isn't enough room on the panel to do all of the ratings.

ISO 3668 identifies the preferred size of the test panel to be at least 150 x 100 mm. ASTM D 1729 lists the size to be 90 x 165 mm, while D 1006, Conducting Exterior Exposure Tests of Paints on Woods[1] has 150 x 915 mm. SAE J361 recommends 215 x 280 mm and the European Coil Coaters Association, ECCA[2], call for a 200 x 150 mm blank before any bends are put on the specimen. Even with all of these listed recommended sizes, the majority of the exposures in Florida are either 152 x 305 or 102 x 305 mm. What begins to place pressure on the observer to maintain accuracy in his reporting is the number of evaluation categories demanded of one coated specimen. Add to this the practice of washing one half of most of the automotive coatings being tested, the area needed for chalk is going to overlap with gloss areas or the area needed for cross hatch adhesion after several readings. If tape chalk, (ASTM D 4212), is used in favor of circular finger and velveteen method found in D 659, the available space is reduced even further.

From the examples in Figure 1, it becomes clear that there must be some further consideration made to the recommended sizes listed in ISO 3668 and ASTM D 1729 particularly if the specimens are to be weathered in natural environments. Because of the design and geometric limitations of accelerated equipment, sizes cannot be increased in many laboratory studies. But even the largest specimen can pose a problem for the observer. If the 150 x 915 mm area of the exterior coatings study is covered by 10 coatings, the effect is just the same as if the panel were reduced to less than 91.5 mm in width.

The answer is to expose either larger panels or larger populations of the same study. Short of this, the observer should include in his report that the frequency of a physically disruptive rating like chalk or adhesion has overlapped with other areas being rated. While this may not completely explain variations in ratings over long exposure periods, it may help to encourage those preparing the specimens to look at the design of the study and insure there is enough area to rate over the length of the test.

The economics of this suggestion may frustrate better designs for weathering studies. The cost of preparation, shipping, exposure, reporting, and return must be kept in some balance, but if decisions are being made to produce new formulations of coatings that are to go into world markets, it is equally important to insure the best possible study for the building of data. Taking chalk ratings over the same small panel for a 10 year period may only tell the producer there was enough paint on the test specimen to endure the repeated ratings over the period of the test.

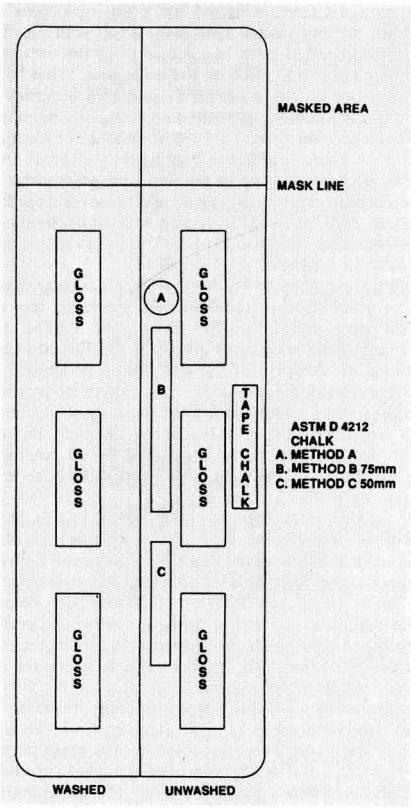
Conclusion

The need to insure a common "language" for evaluating

coatings specimens seems to have intensified over the past few years, and the purpose of this article was to point out some of the more obvious places that there is need to make common scales more universal. Experience has shown that there is even a need within larger corporations. Where multinational operations and even large single site corporations have labs making and reporting on the same or similar materials, often there is no knowledge of weathering programs and ratings scales used by the other department.

However, in some of these corporations, there are remarkable examples of intra-company guides with pictorial examples defining and illustrating the categories of failure[7]. Some of the pictorial standards developed within groups like ASTM and TNO have achieved the same results and it is recommended that when reporting is done by an observer acting upon instructions from the author of the study who cannot see the specimens, some effort should be made to list the standard practices and methods which promote a common understanding.

Figure 1



Acknowledgment

The author wishes to acknowledge the patience and help of members of the South Florida Test Service staff. Thank you to Larry Bond for listening, John Scoff for encouraging, Lesely Crewdson and Alice Hopper for reading and rereading, and Silvia Charafardin for typing. A special thanks to Alan Wootton of Alplas Technology for his introduction to the people at JOCCA.

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Based on paper presented to the 1990 PRA Symposium on "Harmonization of Standards and Regulations in Europe".

Analytical method for dibutyltin and tributyltin contents in antifouling paint by gas chromatography

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Abstract

This analytical method covers the simultaneous determination of dibutyltin (DBT) and tributyltin (TBT) contents in antifouling paint by gas chromatography with flame photometric detection (GC-FPD). The method is based on the dilution of butyltin compounds in antifouling paint with toluene, followed by the gas chromatographic determination of their hydrides converted by sodium borohydride. The recoveries of dibutyltin dichloride (DBTC) and tributyltin chloride (TBTC) from antifouling paint samples were 92.3-102.9% and relative standard deviations were 2.5-5.1%. The proposed method is suitable for the routine analysis of DBT and TBT contents in antifouling paints.

Introduction

The triorganotin compounds (R_3 SnX), e.g. tributyltin (TBT) and triphenyltin (TPT) compounds, have been shown to be active against various marine organisms, and are used as an antifoulants in antifouling paints which are applied primarily to prevent the attachment of marine fouling organisms on ship and boat hulls[1-3]. The tributyltin (TBT) compounds used in antifouling paints has been described as 1% in minimum, and 15% by weight[4-5]. The authors [6-10] have already reported the leaching mechanisms of copper, tributyltin (TBT) and triphenyltin (TPT) compounds from conventional and self-polishing copolymer (SPC) antifouling paints into seawater.

An analytical method for tributyltin (TBT) and triphenyltin (TPT) contents in antifouling paints must be essential for both research and production control in the paint manufacture. In the previous paper[11], the author described the simultaneous determination of tributyltin compound and its degradation product, dibutyltin (DBT) compound, leached into seawater from self-polishing copolymer (SPC) antifouling paint by gas chromatography with flame photometric detection (GC-FPD). This method was based on the extraction of organotin compounds as their chlorides from the seawater with toluene, followed by the gas chromatographic determination of their hydrides converted by sodium borohydride.

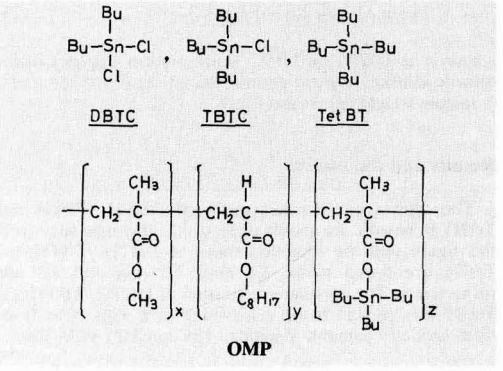
In this paper we describe an analytical method for the simultaneous determination of dibutyltin (DBT) and tributyltin (TBT) contents in antifouling paint (A/F) by gas chromatography with flame photometric detection (GC-FPD).

Experimental

Apparatus: The gas chromatograph used was a Shimadzu Model GC-14A (Shimadzu Co, Kyoto, Japan) equipped with a flame photometric detector (FPD) with a 610nm interference filter to monitor the emission intensity of the SnH molecular species in a hydrogen-air fuel-rich flame[12]. The output signal from the FPD was recorded on Shimadzu Chromatopac C-R6A data processor. The column used was a wide bore fused silica capillary column, Shimadzu HiCap-CBP1-W25-300 (cross-linked methyl silicone gum, 25m x 0.53mm i.d., df=3.0 μm). The working conditions for GC-FPD are shown in Table 1.

Reagents: Dibutyltin dichloride (DBTC: $(C_4H_9)_2SnCl_2$), tributyltin chloride (TBTC: $(C_4H_9)_3SnCl$) and tetrabutyltin (TetBT: $(C_4H_9)_4Sn$) were obtained from Tokyo Kasei Kogyo Co Ltd (Tokyo, Japan). The structures of butyltin compounds are shown in Figure 1. The standard stock solutions (1000 μg ml^{-1}) of butyltin compounds in ethanol were prepared and kept in a refrigerator. The standard solutions in a range of 0-4.0 μg ml^{-1} were prepared by dilution of the stock solution with ethanol.

Figure 1
The structures for DBTC, TBTC, TetBT and OMP (Bu=C_4H_9–)



A 2.5% (w/v) sodium borohydride ($NaBH_4$) ethanol solution was freshly prepared before each use by dissolving 1g of sodium borohydride in 40ml of ethanol.

Organic solvents, e.g. hexane, toluene and ethanol, were of pesticide analytical grade from Katayama Chemical Industry Co Ltd (Osaka, Japan).

Standard preparation: The butyltin compounds as their chlorides were hydrogenated into dibutyltin hydride (DBTH: $(C_4H_9)_2SnH_2$) and tributyltin hydride (TBTH: $(C_4H_9)_3SnH$) by an ethanol solution of sodium borohydride, and these hydrides were extracted with hexane as follows[13]: DBTC and TBTC were dissolved in ethanol in a concentration range of 0-4.0 μg ml^{-1}. A 5ml aliquot of each solution was mixed with 2ml of 2.5% (w/v) sodium borohydride ethanol solution and the mixture was mildly shaken for 10 min. 20ml of water and 5ml of hexane were added to the mixture, and the mixture was shaken on a mechanical shaker for 10 min and allowed to stand for 10 min. The hexane extract was used as a standard solution of DBTH and TBTH. 2 μl of the standard solutions were used for constructing working curves. Standard solution of TetBT was prepared to dissolve in hexane.

Samples: The antifouling paints (A/F) used for this study were produced with tributyltin fluoride (TBTF: from Nitto Kasei Co Ltd, Osaka, Japan) and organometallic copolymer (OMP) as an antifoulant. The organometallic copolymer used in self-polishing copolymer (SPC) antifouling paint is described as poly(methyl methacrylate – octyl acrylate – tributyltin methacrylate) (Figure 1)[14].

Analytical procedure: A 50-100mg amount of an antifouling paint sample, accurately weighted, was transferred into a 100ml flask and diluted to 100ml with toluene, shaking thoroughly to obtain the complete dilution. 1ml of 2.5% (w/v)

sodium borohydride ethanol solution was added to the toluene solution in order to convert the butyltin compounds into their hydrides. This mixture was mildly shaken for 10 min and stood for 1 hour to precipitate the pigments and cuprous oxide in antifouling paint. 10ml of toluene solution was diluted to 100-500ml with toluene as the sample for gas chromatographic determination. A 2 µl of the toluene solution was injected into the gas chromatograph, and the concentrations of DBTC and TBTC were determined from the working curves. From the concentrations of DBTC and TBTC obtained, the DBT and TBT contents (%) in antifouling paint were given by the following equations:

$$\text{DBTC, TBTC (\%)} = \frac{A \times 100 \times B \times 10^{-6} \times 100}{C}$$

$$= \frac{A \times B \times 10^{-2}}{C} \quad (1)$$

$$\text{DBT (\%)} = 0.76 \times \text{DBTC (\%)} \quad (2)$$

$$\text{TBT (\%)} = 0.89 \times \text{TBTC (\%)} \quad (3)$$

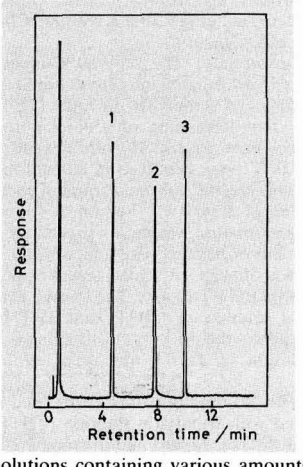
where A is DBTC or TBTC concentration (µg/ml) in the toluene solution, B is the dilution parameter (x 10-50), and C is sample weight (g), respectively.

Results and discussion

The typical gas chromatogram of DBTH, TBTH and TetBT in hexane are shown in Figure 2. It can be seen from this figure that the retention times of DBTH, TBTH and TetBT are found to be 4.4 min, 7.5 min and 9.7 min respectively. The complete separation of DBTH, TBTH and TetBT was proved to be achieved with a wide bore fused silica capillary column, Shimadzu HiCap-CBP1-W25-300.

Figure 2

Typical gas chromatogram of DBTH, TBTH and TetBT. Peaks (1)=DBTH (1.04 ng as DBTC), (2)=TBTH (1.10 ng as TBTC) and (3)=TetBT (0.95 ng as TetBT)

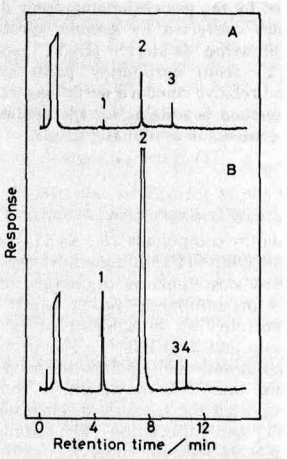


Standard solutions containing various amounts of DBTH and TBTH (0-4.0 µg ml^{-1} as DBTC and TBTC) in hexane were prepared from a standard solution of DBTC and TBTC in ethanol. The working curves are made by plotting amount of DBTC and TBTC against corresponding peak areas in the chromatograms. Under these GC conditions, it can be seen that the working curves are linear in the working concentration ranges of about 0-4.0 µg ml^{-1} DBTC and TBTC.

Figure 3 shows the gas chromatograms obtained from the antifouling paint sample with and without addition of sodium borohydride ethanol solution. From this figure, no GC peaks attributed to the butyltin chlorides such as DBTC and TBTC were observed probably because of their adsorption on the capillary column in the gas chromatograph, but those attributed to their hydrides such as DBTH and TBTH were observed in the chromatogram. In addition, a trace amount of TetBT was determined in most antifouling paints, as shown in Figure 3.

Figure 3

GC-FPD chromatograms obtained from the antifouling paint sample. (A)=no $NaBH_4$ added, (B)=$NaBH_4$ added. Peaks (1)=DBTH, (2)=TBTH, (3)=TetBT and (4)=unknown



A recovery test was conducted using the antifouling paints containing 1.3-20.3% DBTC and TBTC. As shown in Table 2, DBTC and TBTC were determined with 2.5-5.1% relative standard deviation (RSD) in the range 1.3-20.3% as their chlorides, and the precisions expressed as relative standard deviation (RSD) were very good.

The proposed method was applied to the determination of DBT and TBT contents in antifouling paints. Table 3 shows the analytical results for DBT, TBT and TetBT contents in several antifouling paints (A/F) and organometallic copolymers (OMP). For comparison, the calculated contents of TBT are also tabulated. From this table, the TBT contents obtained by this analytical method were in good agreement with the calculated contents.

This method should be useful in determining the DBT and TBT contents in antifouling paints (A/F) and organometallic copolymers (OMP). In comparison with the atomic absorption spectrometry described previously[15], the gas chromatographic determination of each butyltin compounds in antifouling paint (A/F) and organometallic copolymer (OMP) was rapid, simple and sensitive. This method may be applied to routine analysis for DBT and TBT contents in antifouling paints without difficulties.

Acknowledgements

The authors wish to thank the management of Kanae Paint Co Ltd for permission to publish this work.

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Table 1
Working conditions for GC-FPD

Column	Wide bore fused silica capillary column, Shimadzu HiCap-CBP1-W25-300 (25 m x 0.53 mm i.d., df=30.0μ)
Detector	FPD with a 610 nm Interference filter Air: 0.7 kg/cm^2 H_2: 1.8 kg/cm^2
Temperature	Injection: 250°C Detector: 300°C Column: 70°C (1 min)−15°C/min−240°C
Carrier gas	Nitrogen 1.0 kg/cm^2
Sensitivity	8
Split ratio	Splitless
Chart speed	5 mm/min

Table 2
Recoveries of DBTC and TBTC from antifouling paints

Compound	Added (%)	Found(%)[a] Aver.± SD[b]	RSD(%)[c]	Recovery (%)
DBTC	1.3	1.2 ± 0.04	3.3	92.3
	5.1	5.1 ± 0.20	3.9	100.0
	10.1	9.9 ± 0.25	2.5	98.0
TBTC	4.9	4.8 ± 0.17	3.5	97.9
	10.1	10.4 ± 0.53	5.1	102.9
	20.3	20.5 ± 0.82	4.0	101.0

a) Average for five runs.
b) Standard deviation.
c) Relative standard deviation.

Table 3
DBT, TBT and TetBT contents in the antifouling paints and organometallic copolymers

Sample	Type	Contents (%)[a] DBT Calcd.	DBT Found	TBT Calcd.	TBT Found	TetBT Found
A	A/F[b]	——	0.20 ± 0.01	7.6	7.5 ± 0.3	trace[d]
B	A/F	——	0.27 ± 0.01	8.7	9.1 ± 0.5	trace
C	A/F	——	0.28 ± 0.01	10.9	11.3 ± 0.4	trace
D	OMP[c]	——	0.45 ± 0.02	23.2	23.0 ± 0.6	trace
E	OMP	——	0.48 ± 0.02	24.5	25.7 ± 1.2	trace

a) Average ± standard deviation for five runs.
b) Antifouling paint.
c) Organometallic copolymer.
d) Trace means <0.02%.

Manchester Section

Pigment dispersion

Manchester Section's 1990/91 session had an earlier start than usual, with an extra printing ink lecture being included in the programme at short notice. The meeting was held on Monday,17 September, at the "Silver Birch", near Warrington, where a paper entitled "Pigment Dispersion - Its Impact on the Manufacture of Lithographic Printing Inks", was presented by Mr Elun Stenfeldt Madsen, of ATL offset, 53 members and guests being present at the meeting.

Pigment dispersion is the combination of mechanical grinding, physico-chemical wetting and stabilisation. It is vital that mill base rheology should be adjusted in relationship to the grinding equipment used, comparative requirements of 3 roll mills and bead mills being made. Good pigment wetting is very important, yet the trend towards faster and faster setting inks is resulting in a move towards poorer wetting, and less compatible resin systems. Temperature is important for compatibility, and therefore temperature control is vital in the dispersion process. The effectiveness of hyperdispersants in promoting pigment wetting was outlined, unsatisfactory wetting giving very high thixotropy in the mill, resulting in poor dispersion, and therefore poor transparency and colour strength in the ink.

The lecture was followed by an extensive and lively question and answer session, the vote of thanks was proposed by Mike Nixon, and the meeting concluded with a buffet sponsored by Tennent - KVK.

M G Langdon ■

Biocides

Manchester Section's first paint lecture of the 1990/91 session was held at the Mechanics Institute Manchester on Monday, 1 October 1990. The meeting was attended by 44 members and guests, and a paper entitled "The Growth and Control of Micro Organisms on Applied Surface Coatings", was presented by John Gillatt of Thor Chemicals (UK) Ltd.

The lecturer commenced by outlining the place of micro organisms in evolution. Three main groups affect paint, these being Bacteria, Fungi and Algae, and the circumstances under which they will grow in, or on paint, were outlined.

Bacteria organisms were described, their growth being very rapid under ideal conditions, with 500 becoming 16,000,000 in 5 hours. The way in which bacteria can be counted and identified was illustrated, and the problems caused by contamination by these organisms in paint detailed. The numerous varieties of fungi which can effect paint were also described. These were slower growing than bacteria, but cause similar problems in the wet state, however, unlike bacteria, fungi will also grow on paint in the dry film state.

Sources of contamination by micro organisms are, raw materials, production plant, environment, and container, and this contamination can result in micro biological growth in both plant and container. The areas where micro biological growth is likely to occur in mixing vessels was outlined, and the problematical organisms listed. Contamination can be controlled by improved plant hygiene and design, by checking, and, if required, treating the water supply, checking raw materials, and incorporating a broad spectrum biocide into the coating.

The two main functions of surface coatings are protection and decoration. Surface coatings can deteriorate due to attack from micro organisms, the types of breakdown which could be expected were outlined, the organisms found growing on interior coatings were detailed, and the various preventative measures outlined. Algae are the primary problem on the exterior of buildings (they require light for growth), the various types which can be found were described, their effect on exterior coatings illustrated, and methods of prevention outlined.

The lecturer concluded his talk by outlining the various types of biocides available, the properties and effectiveness of the different types, and the changes taking place on environmental and toxicity grounds.

The lecture was followed by a short question and answer session, the vote of thanks was proposed by Mike Langdon, and the meeting closed with a sponsored buffet and bar, courtesy of Thor Chemicals.

M G Langdon ■

Natal Section

Working with mercury

It is well documented that mercurials are toxic to human and animal life. That mercurial derivatives can be manufactured and used safely if a responsible attitude is adopted was stated by Mr P J Strassburg of Thor Chemicals SA (Pty) Ltd at a well attended and informative meeting held at the MBA Building, Westville on 5 September 1990.

Mr Strassburg commented that there is a demand for mercury and its chemical derivatives. Mercury metal is used in dentistry, scientific instruments and certain electrical apparatus. The chemical derivatives of mercury are used in pharmaceutical products,electrical storage batteries,bacteriacides, fungicides and algicides used in aqueous polymeric systems. Indeed, were all trade in mercurials banned internationally, mankind would lose considerable benefit.

Mercury occurs naturally in many foodstuffs like butter (141 parts per billion), cheese, margerine, egg yolk, brazil nuts and bananas (down to 31 ppb). These levels seem high when compared to the World Health Organisation's standard of 1 ppb for drinking water. Mercury is ingested by mankind every day because mercury occurs naturally. Other substances, such as alcohol, caffeine and nicotine, are toxic. Nevertheless their use is socially acceptable.

Chemical derivatives of mercury are used in eye ointments, eye drops, haemorrhoid preparations and antiseptic solutions.

Mr Strassburg dealt in detail with the chemistry and toxicity of mercury. He explained how his firm acts responsibly in the handling of mercury.

The vote of thanks was proposed by Mr Mike Eveleigh. He complimented Mr Strassburg for attracting a record attendance and for a record number of questions. The lecture had created a better insight into the handling of mercury. Thor Chemicals were also thanked for their hospitality.

E Puterman ■

Newcastle Section

Moisture cured polyurethanes

The first meeting of the 1990/91 Session was held on 4 October at St Mary's College, University of Durham. Forty-five members and guests attended to hear a lecture entitled "Moisture-cured Polyurethanes" given by Mr R F Stanfield of Liquid Plastics Ltd.

He began by suggesting that "moisture triggered" would be a better description of the products he would be discussing, typical of a newer generation of single-pack urethanes not having the drawbacks of traditional moisture-cured, even two-pack, products. He intended to concentrate on one particular example which was commercialized in 1985 after 3-4 years intensive study and development of the moisture-triggering mechanism including alternative prepolymers and other critical ingredients. From this study emerged a highbuild pigmented roof coating, capable of application in a wide range of conditions, curing, even under water, to give a continuous film free from defects, with outstanding toughness, flexibility and

durability. It was granted an Agrément Board Certificate after commercial demonstration of performance. Overhead slides of coating sections illustrated the widespread vacuole formation (Swiss Cheese effect) often found in traditional urethane thick films cured in moist/wet conditions and the homogeneous pore-free film of the moisture-triggered product. Further slides illustrated the basic chemistry of the various urethane cure mechanisms and it became clear that the product under discussion is an oxazolidine-containing material: here, moisture "triggers" the cure reaction by opening the oxazolidine ring, giving polyamine which reacts very quickly with the isocyanate prepolymer. Curing is equally effective in the dry conditions of Arizona or cool, wet northern European conditions.

A list of drawbacks of traditional roof coatings included the necessity to apply multi-coat thin films to minimise bubbling or vacuole formation in moist conditions as a result of carbon dioxide release by the isocyanate-water reaction: also, the necessity to add pitch and/or aluminium to upgrade water-resistance and reduce water vapour transmission, resulting in dark colours and high heat-gain in strong sunlight. Others were limited shelf-life (gelation/gassing), short pot-life of opened containers, poor aged self-adhesion and acceptance problems of glass-fibre reinforcement with thin coating films.

A similar list of drawbacks common to two-pack urethanes cited the need for close control of mixing, often difficult on-site, and the greater hazard from isocyanate vapour exposure.

Using these lists of disadvantages, Mr Stanfield then set out the main areas selected for improvement in his development work on roof coatings. By far the most important was to avoid reactions leading to carbon dioxide formation: from this would flow the benefits of thick, pore-free, water-resistant polymer films, not requiring fortification with pitch/aluminium and, thus, the possibility of high-reflectance whites/tints (low heat-gain), capable of curing uniformly in the cold, wet conditions typical of UK roofing.

In fact, the coating finally developed met all the conditions set, being capable of application up to 3mm thick on damp substrates and resistant to rain soon after application, actually curing uniformly when immersed in water. The cured polymer film is strong, resisting foot traffic, accepting aggregate for non-slip properties and glass fibre reinforcement for joint-bridging. Applied using airless spray, roller or brush it cures in 5 hours at 22 °C and, with catalysis down to 2 °C, ensuring the wide weather-window of application necessary. A true elastomer, it retains its flexibility, and the absence of vacuoles within the cured film, allied to the use of aliphatic isocyanate prepolymer, results in excellent resistance to erosion and film breakdown on exterior weathering. Mr Stanfield showed typical test-results on film properties (tensile strength, elongation, adhesion to concrete, water vapour transmission) together with accelerated weathering, QUV resistance, flame retardance and Operational Temperature Range (cold flex, freeze-thaw cycling, Durometer hardness etc).

He concluded by showing typical successful applications, the most impressive being a 40,00Om^2 roof of an aero-engine plant in Turkey, sealed and coated after serious rain water leakage. Other examples included protection of polycarbonate or glass roof lights: to prevent surface erosion and preserve light transmission with the former and to improve impact-resistance (shatter proofing) of the latter: in these latter cases a clear, unpigmented version is used.

In question time, Mr Stanfield said that coating smell noted with relatively freshly-applied material is due to isobutyraldehyde which, in practice, is rarely noted. The long usable pot-life results from surface skin formation which prevents further "moisture-triggering" within the bulk of an opened can. This skin becomes extremely thick if the open can is left unused for seven days or more and is almost impossible to remove. Despite the very low free isocyanate, air-fed respirators are necessary in confined spaces-tanks etc. No problems with Union-resistance had been met in Australia and the coating has approval by Det Norske Veritas and Danish authorities. A limited number of "Approved Applicators" had been carefully selected for the UK, which safeguarded against mis-use. He agreed that pigmentation should not include materials which absorbed oxazolidone. Finally he felt that it could be adapted for Structural Steel protection.

The vote of thanks was given by the Chairman, Mr David Neal, after which all present were treated to an excellent buffet with wines and refreshments, courtesy of Liquid Plastics Ltd.

J Bravey ■

West Riding Section

The mysterious world of paint additives

Rob Lewis (West Riding Section Chairman) introduced Lionel Morpeth's lecture on "The mysterious world of paint additives" and mentioned that it was the last Technical Lecture of the season which turned out to be one of the most visual and entertaining of the season and an excellent note to finish on.

Lionel's curriculum vitae started in 1963 when he worked at International Paints of Felling, 1969 Foscolor and 1980 Byk Chemie. Lionel mentioned that this lecture would be a minute waltz through the paint additives industry and when his first video showed a boat trip in Lucerne I thought he was going to take the minute waltz literally. Lionel started by saying that these products are used at less than 1% of the paint formulation and that 0.5% is not the answer to everything.

That is certainly true for buyers who are not interested in saving 10% of 0.5%, rather than 0.5% of 10%. Wetting was explained in detail with the following parameters being considered:

1. Low surface tension. 2. Inter-facial tension. 3. Pigment pore size. 4. Viscosity of resin solution.

Inter-facial tension was majored on showing the different mechanisms of repulsion i.e. aqueous systems - electro-static, organic systems - steric hinderance. Wetting agents are broken down into different categories with explanations given about cationic, anionic and electro-neutral agents. This ensued into a description of controlled flocculation and de-flocculation, the uses being high build/primer systems and top coats respectively. Also three paints were presented showing the difference between soft and hard settlement of an iron oxide in an alkyd/amino system by using controlled and de-flocculation.

An explanation was given showing the difference between classical wetting agents and the new polymeric agents which have multiple weak anchor points giving a cohesive force at the pigment surface. Polymeric agents are close to my heart and I have an affinity for them - sorry about the pun. A comparison of floating and flooding was given, I think to keep Rob Lewis happy and the effects of flocculation when using combinations of multi-purpose mill bases using chromaticity diagrams to explain batho-chromic and hypso-chromic shifts.

Section 2 of the presentation was about silicones. These can be categorised into flow controls and anti-foams, slip agents, defoamers and hammer finish additives in order of ascending molecular weights. Flow aids in general are high boiling solvent mixtures or polyacrylates. Slip aids are normally silicones which are used to control surface tension and are normally used to avoid surface defects in paints.

However, the incorrect use of silicones may lead to problems such as inter-coat adhesion and foaming. Lionel showed in great detail how to select the correct siloxane chemistry to overcome these problems. Lionel's minute waltz turned

out to be a stimulating presentation and certainly was not nearly as boring as watching paint dry.

P R Stanton ■

Wasteful society - An industrial overview of current attitudes to waste production.

The first lecture of the new session took place on Tuesday 18 September at the Roundhay Mansion Hotel,Leeds. The lecture was given by David Humphrey of Sandoz Products Ltd.

David, a Yorkshireman born and bred, is a member of the Institute of Occupational Safety and Health. He joined Sandoz in 1952 and moved to safety and environment 10 years ago after various production positions including chemical plant manager.

The lecturer opened showing all of us as waste producers, examples being the local household waste station and flytipping. It was also shown that landfill sites could eventually be returned to agricultural use therefore having a beneficial environmental effect.

Moving on to industrial waste the lecturer showed how the effects could be air, water and ground pollution, affecting sight, smell and hearing. Using Sandoz in Leeds as an example he discussed the possible problems, starting with the influence on the environment by just being there. He went on to highlight problem areas in the production of solid and liquid wastes before moving on to discuss waste treatment.

The mechanism, use of and possible results obtained by an effluent treatment plant were discussed in some detail. The use of bacteria and a sufficient supply of oxygen was shown to degrade the waste and be capable of adapting to deal with new waste types. An occasional impurity such as MEK in the waste is said to cause a dramatic increase in oxygen usage as bacteria are very fond of such materials. The use of rainwater collection for blending in the runoff stage has shown to reduce impurities in the discharge.

The lecturer discussed noise from frequency of fans on roofs, for example, and indicated insulation was necessary. Of particular concern were noise problems at night or weekends.

Solid waste was shown to be more of a problem, landfill or incineration being necessary. The lecturer showed how thas sort of disposal of solid waste had carried on for centuries, notably in graveyards and crematoriums. The UK was shown to be the world leader in the waste disposal trade.

Mr Humphrey moved on to discuss the tightening of legislation in the future. This, coupled with more rigorous checking by the authorities, will only increase the problems.

The waste disposal and chemical industry and its poor public image was now touched on. The industry has much work to do to clean up its image and change public opinion. When we consider the work done by archaeologists sifting through ancient rubbish tips, the question - what will future archaeologists think of us? - can validly be asked.

A very rigorous question session followed with a large range of enquiries from the mechanism and choice of effluent bacterial sludge to how the industry can fight back and turn public opinion.

Bob Hemmingway of Kalon proposed the vote of thanks which was followed by a sponsored buffet and bar courtesy of Sandoz Ltd. The lecture was attended by 21 members and 6 nonmembers.

S Birkett ■

Letters

Silicone Emulsions

Dear Honorary Editor,

Referring to Monsieur J.M.Pouchol's transaction on "Silicone Emulsions: Binders for high performance facade coatings" (*JOCCA* 1990, **73**(9), 370),and particularly to references to Pliolite paints.

The "ideal" moisture vapour transmission rate (MVTR) for exterior masonry paints could be the subject of a book. From the proven practical durability worldwide of Pliolite® resin-based masonry paints over the last 35 years, we can conclude that the MVTR of these coatings is satisfactory.

Regarding the statement "as for solvent based paints, such as Pliolites, only a few fanatics still use them...." it is a fact that in France Pliolite® resin-based masonry paints cover more square metres per year than any other type of masonry paint. Also Pliolite® resin-based masonry paints have a significant market share in other European countries including the U.K.

I understand in France a "fanatique" is a devotee. If this is M. Pouchol's intended meaning, he is right except for the number of devotees he implies.

Goodyear Chemicals Europe,
Greenfield House,
69-73 Manor Rd,
Wallington
Surrey SM6 0DE

Yours faithfully,
Barry Wood C.CHEM, MRSC

23 October 1990

®Trademark of the Goodyear Tire & Rubber Company, Akron, Ohio, USA. ■

News of Members

Robert Hamblin in ceremony to commemorate Richard III

A significant event took place recently at Leicester in which Robert Hamblin (Honorary Member and the former Director & Secretary) participated.

Richard III was buried at Leicester, but his grave is unmarked. The Richard III Society has sought to redress this by a memorial ledger stone in Leicester Cathedral in 1982 and this year by a plaque commemorating his burial in Greyfriars. The Leicester Plaque depicts a Greyfriars burial and part of the text reads:

Next to this site stood the church of the Greyfriars where the body of Richard III, the last Plantagenet King of England,was interred after his death, aged 32, at the Battle of Bosworth Field 22 August 1485.

The plaque also carries Richard III's emblem of the white boar with his personal motto *Loyaulté me Lie.*

Readers who were privileged to attend this Association's Council Reunion Dinners at the Wax Chandlers' Hall will recall that the Wax Chandlers' Company's Charter shows the only known surviving example of the Royal Arms with two white boar supporters and Richard III's personal motto.

Since the only remnant of the Greyfriars is a small piece of wall, the National Westminster Bank kindly agreed to the siting of the Leicester Plaque in an adjacent but more prominent position on their side wall in Greyfriars.

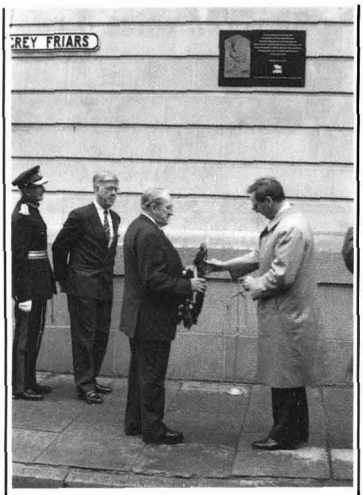
Following his year as Master of the Wax Chandlers' Company, Robert Hamblin was elected Chairman of the Richard III Society, which was founded in 1924 and now has 4000 members. On 30 October, HRH The Duke of Gloucester (Patron of the Society) inaugurated the Leicester Plaque by placing a wreath -a laurel chaplet with Yorkist white roses and purple ribbon- beneath the plaque.

MD of Ferguson & Menzies retires

Mr P F Bridle has retired as Managing Director of Glasgow based Ferguson & Menzies Ltd and Mr J F Rait, former Scottish Director for Kuwait Lubricants, has been appointed the new MD. Mr Bridle has been in the Surface Coatings Industry for over 48 years since joining Thos. Parsons and Sons at Mitcham in March 1942. Mr Bridle will remain with the company for a while on a part-time basis.

Deaths

Alfred William Hall (London Section- Elected to Association 1927)



The photograph shows the scene at the inauguration of the Leicester Plaque. (Left to right) The Lord Lieutenant of Leicestershire (Mr T G M Brooks,JP); Mr G R Waine (Regional Executive Director, National Westminster Bank); Robert Hamblin (Chairman,Richard III Society); HRH The Duke of Gloucester (Patron of the Society).

Photo: Geoffrey Wheeler

Tribute

Mr D R Gray

Cecil Butler writes:

With the death of Denis Roy Gray, OCCA (and the West Riding Section in particular) lost one of its most enthusiastic and hard-working supporters.

Denis Gray started his career in the paint industry with Pinchin Johnson Ltd. at Silvertown. He moved to Ripon in the immediate post-war years to become successively Chief Chemist and Works Director of Messrs T.& R Williamson - where he remained until his retirement in 1974.

Originally a member of the London Section, he was instrumental in founding the West Riding Section in 1953, and was effectively its first Chairman (1953 to 1955). He saw service on the OCCA Council, and held offices of various kinds in the W.R.Section until, during his retirement, he found travelling to Section meetings in Leeds beyond his physical ability.

Denis was essentially a very private man. His main interest in his latter years was in photography - he was an official of the local photographic society, and won many trophies for his excellent slides. He gave sterling service to the Ripon Mens' Forum, and was a very active supporter of the local Methodist Chapel.

He will be remembered as a polite and friendly man who had the ability to express firm opinions - sometimes unpopular ones - without causing offence to anyone.

Obituary

Stuart Allen Cooke BSc (1922 - 1990)

Allen Cooke, a member of the Ontario Section since 1985, passed away on 9 October 1990.

John Ambury writes:

Al Cooke and his charming wife Wilma were loved and trusted friends to all of the people inside and outside the industry who came to know them. They were a beautiful couple and a terrific team. Al was famous for suggesting early breakfast meetings at the Board of Trade Country Club, and for making far-flung sales calls in any weather. Wilma was often there, waiting unobtrusively, only occasionally having to mention to Al, who could be persuaded to reminisce along with business, that they would have to be leaving soon to make their next appointment. He was modest about the value of his knowledge and experience, but was willing to share them if one was interested; if one was interested, and enjoyed a bit of history along with the technology, they were valuable indeed.

Al had been involved with resins and coatings development since his youth. He had worked for several chemical manufacturers, and continued to operate his own raw materials firm, Cooke Chemicals Limited, into this year. He was a long-time member of the FSCT (Toronto Society) as well as OCCA; he attended more meetings of both groups than many of the younger, more mobile members, and always made useful contributions to the discussions. He presented the George Brown lectures on Surfactants, but took no credit for his part in their preparation.

Al Cooke was known and respected for his many contributions to the industry, but perhaps even more for his personal qualities: his loyalty and sincerity, his quiet strength and determination, and his insistence on helping others whenever possible rather than being helped. He will be sadly missed and fondly remembered.

Trent Valley Branch

5-a-side soccer tournament

Trent Valley 'kicked off' their 1990-91 session on Friday September 28th with the third annual 5-a-side soccer tournament. Oak Tree Lane Leisure Centre, Mansfield was the venue for this

Trent Valley 5-A-Side Results

League 1	P	W	D	L	Pts	League 2	P	W	D	L	Pts
Hawley 'B'	4	3	1	0	7	Mebon 'A'	3	2	1	0	5
Steetley	4	2	1	1	5	Hawley 'A'	3	1	2	0	4
Perfectos	4	1	2	1	4	Dacrylate	3	1	1	0	3
Mebon 'B'	4	0	2	2	2	Hopton 'A'	3	0	0	3	0
Hopton 'B'	4	0	2	2	2						

Semi-Finals: Mebon 'A' 4 Steetley 0, Hawley 'A' 0 Hawley 'B' 1
Final: Mebon 'A' 0 Hawley 'B' 0 (after extra-time), Mebon 'A' win on penalties 6-5

years event, sponsored by Mebon, with 11 teams competing for the "Perfectos Trophy". The initial matches were played on a league basis with the top teams progressing to the semi-final knock-out stage. The semi-finals saw the holders, Hawley 'A', eliminated by their rivals Hawley 'B' by a single goal while Mebon 'A' finished with a flourish to beat 'newcomers' Steetley by a convincing 4-0. The final saw two well organised sides fight out a 0-0 draw. Extra time failed to separate the teams and the game moved to penalties, each scoring three from five attempts. The climax was an 'Italia 90' style "sudden death" shoot-out. Hawley 'B' failed with their final kick leaving Mebon 'A' as the new champions. Handshakes all round typified the spirit of the evening, competitive but always fair. Players and spectators retired to the bar where Tony Cox presented the "Perfectos Trophy" to the winners. T shirts and pens, donated by Via Gellia and Joseph Masons respectively, were presented to the runners up and referees. An excellent buffet, prepared by Deborah Howkins, concluded the evening. So, all in all, a successful start to Trent Valley's 1990-91 programme, credit going to organiser Greg Zielinski of Mebon whose splendid efforts should ensure that next years event will be eagerly awaited. Watch this space!

D P Williams ■

Manchester Section

Quiz Night

Manchester Section's ever popular annual quiz night, held at the "Silver Birch", Warrington, on Monday, 15 October 1990 attracted over one hundred contestants in 26 teams. The quiz consisted of six rounds of general knowledge questions, again ably presented by the sections guest quizmaster John Bennett, with this year, a total of 240 points at stake

The winners were "Macpherson B", runners up "Fred Housego Lookalikes" ("Macpherson A") and in third place "Bottom of the Barrel". The booby prize was won by "Top Deck" who also took the prize for the lowest score in any round. The prize for the highest score in any round was won by Macpherson "B", and the prize for the most original name was won by BASF.

The event was excellently organised by Mike Nixon, and the buffet again of the Silver Birch's very high standard rounded off what was yet another very successful and enjoyable event.

M G Langdon ■

Manchester quiz full results

1st	MACKPHERSON "B"	161 Points
2nd	FRED HOUSEGO LOOKALIKES	157 Points
3rd	BOTTOM OF THE BARREL	148 Points
4th	FRIGASM	144 Points
4th	COSHH	144 Points
6th	SPOT COLOURS	142 Points
7th	FULL MONTY	138 Points
8th	BASF	135 Points
9th	JELLY BABIES 6	131 Points
10th	JOLLY JACKS COLOUR MEN	128 Points
11th=	PIG MEATS	127 Points
11th=	HIT MEN AND HER	127 Points
13th=	JOHNSTONES "A" (KR)	126 Points
13th=	FOSCOLOUR	126 Points
15th	THE COWBOYS	123 Points
16th	MANDERS LIQUID INKS (SRSP)	122 Points
17th	BALL MILLERS	121 Points
18th	PROCESS FOUR	115 Points
19th	SWALE SWOTS	114 Points
20th	FOUR COLOUR SET	112 Points
21st=	DUMB DUCKS	111 Points
21st=	JOHNSTONES "B" (ISE)	111 Points

Ontario Section

Third Annual Golf Tournament

The Ontario Section Golf Tournament was held on 18 September 1990 at the Nobleton Lakes Golf and Country Club, north of Toronto.

Over 70 golfers challenged the beautiful and varied but quite difficult par 72 course, on a bright, mild day that was perfect for the sport. (The weather was especially appreciated because it came between a frigid Monday and a dark, rainy Wednesday.) As in the previous two years, the Nobleton Lakes facilities and staff handled the event very smoothly, from the start at the pro shop to the beer and sausage hut after the front nine to the comfortable club house verandah lounge - from which the early finishers could observe the expertise of the later foursomes on the approach and green of the eighteenth hole.

The day finished in style with the awards banquet, at which the trophies were awarded and the gift prizes, donated by a large number of generous suppliers, were presented to all participants as their names were drawn.

David Houston scored a clear win with an impressive 79, to take home the tournament's Low Gross trophy for the second year in a row. The Second Low Gross was captured by his former coach, Bob Houston, with a very respectable 84. The winner of the Low Net was Dave Gray. The winner of the Most Honest

Winking Wienie (for High Gross) was overcome by a sudden attack of modesty and declined to give his name to the press.

The entire Tournament was arranged and coordinated by John Pitt, who started this contest for the Section almost single-handedly in 1988, and who was responsible (along with the enthusiastic participants, the prize donors, the Nobleton Club and the cooperation of the weather) for another outstanding and memorable OCCA event.

J F Ambury ■

West Riding Section

West Riding Golf Trophy

This competition was held on 11 October 1990 at the usual venue of Wetherby. The golf course was in good condition and the Autumn colours made the course look particularly attractive. The damp weather made the course a little slow, but the greens were in excellent if slightly sandy condition.

35 members and guests took part in the competition and we just managed to complete the round before darkness fell. The highest score was guest Colin Brewster with 36 points and the winner of the competition was Martin Smith with 34 points.

The day was finished off with a good meal in the Club House and the presentation of prizes.

G C Alderson ■

Publications Committee visits Herald Press

The OCCA Publications Committee recently convened at the Moat House Hotel, Stratford-upon-Avon for its annual meeting. In the morning the committee discussed the changes in JOCCA over the last year and the editorial programme for 1991. The committee also toured the hotel which will be the venue for SURCON 91. Lunch at the hotel was kindly sponsored by Herald Press and in the afternoon members visited the Herald print works.

Professional Grade

At the meeting of the Professional Development Committee held 25 September 1990 the following admissions were made:

Admitted to Fellowship:

Miller, Eric Roy (*Thames Valley*)

Upgraded from Associate to Fellowship:

Notley, Malcolm Anthony Leslie (*London*)
Troparevsky, Alejandro (*General Overseas - Argentina*)
Whatling, Allan (*Bristol*)

Admitted to Associateship:

Alexander, Paul (*Midlands-Trent Valley*)
Clarke, John Bernard (*West Riding*)
Cowan, Michael Hugh (*Manchester*)
Quintal, Elizabeth(*Ontario*)
Watson, Peter Joseph Huby (*Newcastle*) ■

New Members

Ordinary members

Boyden, J, BSc (*Manchester*)
Classen, C (*Cape*)
Coombes, N A (*Transvaal*)
Davis, W I, BSc (*Zimbabwe*)
Downes, J D, BSc (*Bristol*)
Fisher, C, BSc (*Hull*)
Fowler, R C (*Manchester*)
Frost, K N, BSc (*Transvaal*)
Gadd, F (*Cape*)
Gamble, R J, BSc (*Cape*)
Harpur, D P, PhD, BSc (*Cape*)
Heath, S E (*Manchester*)
Lazarus, C M (*Hull*)
Millar, D, BSc (*Scotland*)
Naidoo, S V (*Natal*)
O'Connell, A (*London*)
Phillips, A, BSc (*Midlands*)
Reck, E, BSc (*Newcastle*)
Retief, F, BSc (*Cape*)
Robinson, M (*West Riding*)
Robinson, P W A (*London*)
Sutheran-Lott, G N, BSc (*London*)
Whittaker, K A (*West Riding*)

Associate members

Car, D I (*Zimbabwe*)
Griffin, D L R (*Zimbabwe*)
Newell, E T (*Zimbabwe*)
Oliver, C C (*Zimbabwe*) ■

Classified continued on Inside Back Cover

List of successful candidates

As laid down in the report of the Working Party on Education, Training and Qualifications which was adopted on the institution of the Professional Grade, a list of those members in the Grade is published in the December issue of the *Journal* each year. The 19th such list appears below and includes the names of members resident in 35 countries.

The sections to which members are attached are given in italics.

Fellows

Addenbrooke, Brian John (*Midlands*)
Anneveldt, Jan Johan Willem (*Natal*)
Apperley, Thomas William James (*West Riding*)
Arbuckle, Kenneth Harold (*London*)
Archer, Harold (*Manchester*)
Ashworth, Norman (*Manchester*)
Astfalck, Anthony Noel (*Transvaal*)
Atherton, Donald (*Scottish*)
Bailey, John Noel (*Newcastle*)
Banfield, Thomas Arthur (*London*)
Barrett, Ronald Leon (*Scottish*)
Bayliss, Derek Arthur (*London*)
Berberi, Anwar (*General Overseas - Lebanon*)
Bester, Lawrence Percy (*Natal*)
Bhumkar, Chidanand Jayram (*General Overseas - India*)
Birrell, Peter (*Ontario*)
Bishop, Eric Harold Abbott (*West Riding*)
Boroky, Joseph Stephen (*General Overseas - Australia*)
Bose, Sunil Kumar (*London*)
Bosman, Herman Izak (*Transvaal*)
Bourne, John Robert (*Midlands - Trent Valley Branch*)
Boxhall, John (*Thames Valley*)
Bridle, Peter Frederick (*London*)
Brooks, Leo James (*London*)
Brown, Arthur Ernest Girdlestone (*London*)
Burwell, Barry (*Manchester*)
Butcher, George Alfred (*Midlands*)
Butcher, Kenneth William George (*Manchester*)
Butler, Cecil (*West Riding*)
Byrns, Arthur Robin (*Cape*)
Caffery, George Francis (*London*)
Campbell, George Alexander (*Manchester*)
Canterford, Barry Albert (*London*)
Carter, Eric Victor (*Newcastle*)
Churchman, Anthony Edward (*London*)
Clarke, Harry James (*Midlands*)
Clement, Donovan Harry (*Midlands*)
Cole, Derek (*General Overseas - Australia*)
Collier, Claude William (*Midlands - Trent Valley Branch*)
Collings, Arthur Geoffrey (*London*)
Coupe, Raymond Richard (*London*)
Courtman, Frank (*Manchester*)
Coverdale, Peter Frederick Muir (*Midlands*)
Cowie, Edward Bruce (*General Overseas - Singapore*)
de Jong, Jan Lauwrens (*Transvaal*)
Dowsing, George Frederick (*London*)
Draper, Patrick Albert (*Natal*)
Duligal, Eric Arthur (*Transvaal*)
Dunkley, Frederick George (*Midlands - Trent Valley Branch*)
Easton, James Douglas (*General Overseas - USA*)
Eglington, Roland Alexander (*Natal*)
Ekeh, Godwin Chukoemeka (*General Overseas - Nigerian Branch*)
Finn, Stanley Russell (*London*)
Fisher, Leslie Alexander (*Natal*)
Froggatt, Joshua John (*London*)
Furuhjelm, Victor Henrik (*General Overseas - Finland*)
Garratt, Peter Garth (*General Overseas - Austria*)
Gate, Peter Atholl Jackson (*Transvaal*)
Geddes, Kenneth Raymond (*Manchester*)
Giesen, Mathias Franz (*General Overseas-Germany*)
Gilliam, Brian Frederick (*London*)
Gillian, James Graham (*Manchester*)
Gollop, Percy Lionel (*London*)
Goyal, Ramkumar Bhanwarlal (*General Overseas - India*)
Graham, Thomas (*Manchester*)
Grainger, William Alan (*Irish*)
Gray, Alan (*Ontario*)
Greenfield, Eric (*Midlands*)
Grey, James (*Ontario*)
Haken, John Kingsford (*General Overseas - Australia*)
Hamburg, Herman Rudolf (*London*)
Hanson, Robert Philip (*Newcastle*)
Hill, Derek Alfred Wheeler (*London*)
Hill, Gilbert Victor Geoffrey (*Thames Valley*)
Hill, Lawrence Albert (*General Overseas* - Australia)
Hipwood, Hubert Allan (*London*)
Hodgson, Kenneth Vickerson (*London*)
Holbrow, Gordon Leonard (*London*)
Hopgood, Arthur Thomas Robb (*London*)
House, Dudley James (*Transvaal*)
Hutchinson, Geoffrey Herbert (*London*)
Inshaw, John Lesley (*Thames Valley*)
Iyengar, Doreswamy Raghavachar (*General Overseas - USA*)
Jacob, Basil (*Thames Valley*)
Johannsen, Ralf Peter (*Cape*)
Johnsen, Svend (*General Overseas - Denmark*)
Jolly, Anthony Charles (*Manchester*)
Kalewicz, Zdzislaw (*General Overseas - France*)
Kane, Joseph Richard (*London*)
King, Raymond John (*Midlands*)
Kotwal, Hoshidar Peshotan (*General Overseas - Pakistan*)
Kut, Sigmund (*London*)
Landmann, Axel Wolfgang (*London*)
Lasser, Howard Gilbert (*General Overseas - USA*)
Lewis, Fred (*Manchester*)
Ley, John Barry (*Thames Valley*)
Lomas, Harold (*Ontario*)
Lunt, Walter Richard (*West Riding*)
Martin, Christian Pierre (*General Overseas - France*)
McLean, Angus (*Scottish*)
McMillan, Stanley (*Newcastle*)
McQuirk, Peter John (*Midlands*)
McRobbie, David Martyn (*London*)
Miller, Eric Roy (*Thames Valley*)
Mitchell, John Edmund (*Manchester*)
Mitchell, Seward John (*Midlands*)
Monk, Cyril James Henry (Bristol)
Moon, William Robert (*Manchester*)
Morgans, Wilfred Morley (*General Overseas – Spain*)
Morpeth, Frederick Johnson (*Manchester*)
Morris, David (*Manchester*)
Munn, Raymond Henry Edward (*London*)
Munro, Hugh Anderson (*Scottish*)
Myers, Gordon (*Transvaal*)
Newman, Derek George (*Thames Valley*)
Newton, Dennis Sydney (Bristol)
North, Albert Graham (*London*)
Notley, Malcolm Anthony Leslie (*London*)
Pace, Graham (*Thames Valley*)
Patel, Shirish Manibhai (*Ontario*)
Pettit, Michael Robert (*London*)
Piggott, Kenneth Elliot (*Natal*)
Polaine, Sidney Alan (*London*)
Porteous, Barry Milroy (*Ontario*)
Prigmore, Maurice Henry (Bristol)
Raaschou Nielsen, Hans Kristian (*General Overseas - Denmark*)
Rampley, Dennis Neil (*London*)
Ray, Stanley Arthur (*Midlands*)
Rechmann, Heinz (*General Overseas - Germany*)
Redman, Frank Benson (*Manchester*)
Reid, John Rodney Stanford (*Transvaal*)
Rileigh, Albert Kenneth (*General Overseas - Australia*)
Robson, Gordon Reginald (*Manchester*)
Roe, David Edwin (*London*)
Rouse, Robert Earnshaw (*Transvaal*)
Rubin, Wallace (*London*)
Rudram, Arthur Thomas Stephen (*London*)
Saunders, Laurence Frederick (*Transvaal*)
Seymour, Norman Henry

(*Manchester*)
Sharpe, Eric Edward Victor (*Transvaal*)
Simon, Raymond (*Irish*)
Simpson, Leslie Ainsley (*Newcastle*)
Slade, Harold Aitken (*London*)
Smith, Eric (*West Riding*)
Smith, Francis Mark (*Manchester*)
Smith, John George Nixon (*Newcastle*)
Stead, Graeme Rupert (*Transvaal*)
Stoyle, Francis Wilbert (*Irish*)
Stretton, Elizabeth (*Manchester*)
Talbot, Ernest Alexander (*Newcastle*)
Tate, Donald Howarth (*Newcastle*)
Taylor, Alexander Legg (*Irish*)
Taylor, John Roberts (Bristol)
Thukral, Prem Sagar (*London*)
Tooth, John Henry Collins (*London*)
Troparevsky, Alejandro (*General Overseas - Argentina*)
van Londen, Anton Matthijs (*General Overseas - Netherlands*)
Walker, Peter (*Thames Valley*)
Watkinson, Leonard James (*West Riding*)
Weineck, Terrence Graham (*General Overseas - Portugal*)
Whatling, Allan (Bristol)
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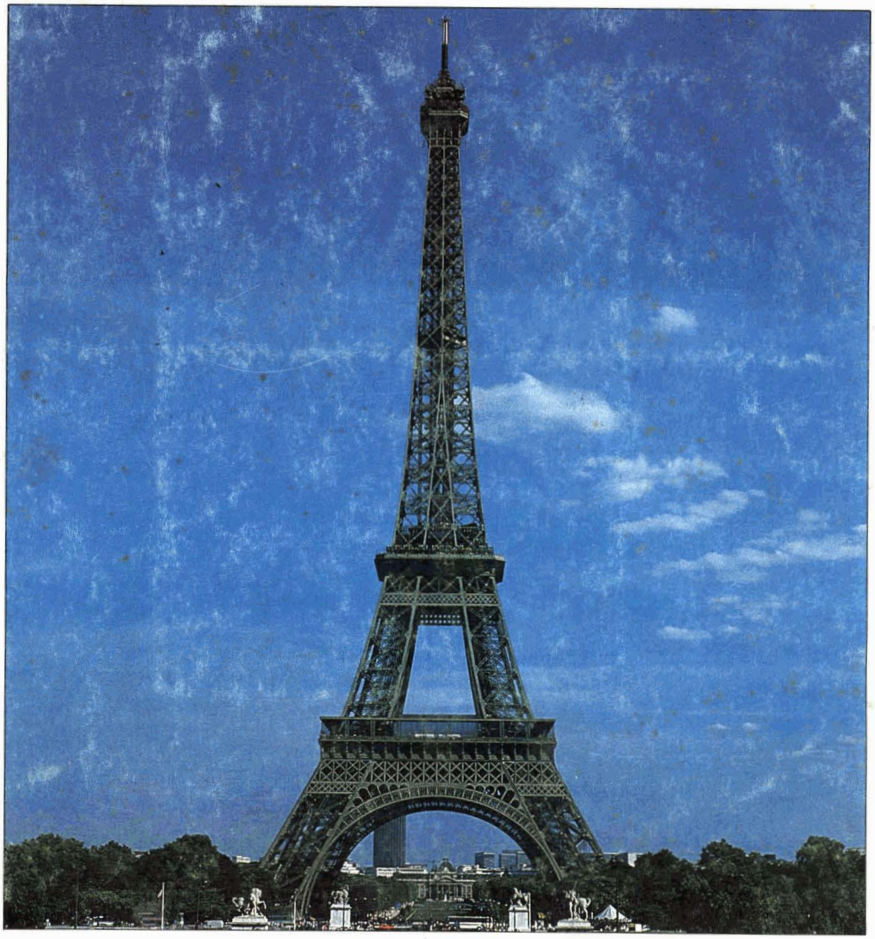

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