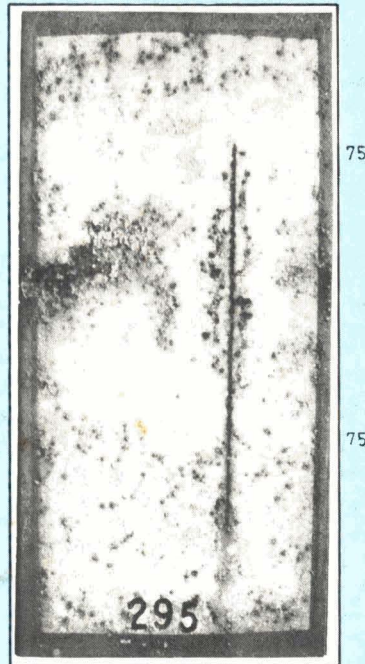




**J
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A**

WATER SOLUBLE CONTAMINANTS AT THE STEEL COATING INTERFACE

THICKNESS : 75 μ m



ALSO IN THIS ISSUE:

- Control of organic pigment dispersion
- Novel paint driers
- President's Page
- London Section 50th Anniversary Dinner

OIL AND COLOUR CHEMISTS' ASSOCIATION



MONOGRAPH No 4



A critical review of the

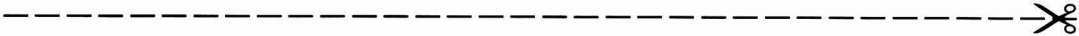
The determination of the solid content of paint (by volume)

by E. G. Elphinstone, International Paint, Gateshead, U.K.

The important factors affecting the determination of the solid content of paint by volume are discussed. Normal paints (those with pigment content below the critical pigment volume concentration) are the subject of greatest scrutiny with metallic zinc primers (pigment content above the critical volume concentration) also receiving attention.

The published methods for *normal paints* (ASTM D2697, BS 3900 Part A10, DIN 53219, ISO 3233) are all very similar, but the ASTM method is the focus of this investigation. Measurement of a wet volume of paint and its corresponding dry volume show that the conversion of wet to dry volume is not without complications because of time, temperature and film thickness dependence. This study has shown the extent to which results are affected by these variables. It is proposed that a definite film thickness (thickness specified in practice) and a specified drying schedule (temperature, humidity and time) should be used in any standard method for the determination of paint solids.

The modification of method D2697 suggested by ASTM to accommodate *paints containing zinc pigment* is examined and compared with other methods. The tungsten disc, mercury displacement method gives results which agree with results obtained in a method relying upon the use of a sensitive stylus trace for the determination of the dry film thickness. The latter method is recommended because of the advantage of giving extra information on the topography of the dry film surface and in avoiding the use of a tungsten substrate and mercury. A centrifuge method, although convenient and reproducible, gave results which do not always reliably agree with those obtained by other methods.



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JOURNAL OF THE

OIL AND COLOUR CHEMISTS' ASSOCIATION

VOLUME 71

1988



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Editorial correspondence should be
addressed to the Hon. Editor, JOCCA,
Oil and Colour Chemists' Association
at the address below.

General correspondence should be
addressed to:
C. Pacey-Day

General Secretary
Oil and Colour Chemists' Association
at the address below.

Advertisement Manager:
F. Craik

Assistant Editor:
P. J. Fyne, MA, DPhil

Tel: 01-908 1086
Telex: 922670 (OCCA G)

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OIL AND COLOUR CHEMIST'S ASSOCIATION

Priority House, 967 Harrow Road, Wembley, Middlesex HA0 2SF England

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President's Page

The first four months of my term of office as President have passed very quickly. This is possibly because much activity has been required following the retirement of our previous Director and General Secretary, Robert Hamblin.

Robert was due to retire immediately after the Eastbourne Conference in June this year when he had planned a well earned holiday after completing 36 years' sterling service with the Association. However, there was a hitch concerning the appointment of his successor and in his usual unselfish and conscientious manner he filled the gap for a further few weeks. We are extremely grateful to Robert and indeed all the Priory House staff for maintaining the Association's affairs in good order from June to September 1987. I am sure we all wish Robert a long and happy retirement but still hope to see him in future at many of the Association's meetings and events.

We also extend a warm welcome to our new General Secretary, Chris Pacey-Day, and hope that he will have a long and happy career with the Association. Prior to joining OCCA Chris was previously employed by the Institution of Chemical Engineers as Director – Membership Services. Chris and his staff at Priory House are currently very busy implementing the modernisation programme. The first phase of this is to computerise the membership register, including printing of address labels for the Journal, subscription notices and accounts. Word processing is also being introduced particularly for production of the copy for the Journal and other OCCA publications. The staff have taken up this challenge and I compliment them on the excellent progress they have made towards mastering the changes involved.

I am grateful to my predecessor, Frank Redman, for the excellent groundwork he prepared for the new organisation to take place smoothly. However I am sure Frank will be best remembered for his leadership of the working party and the successful negotiations he concluded which led to the formation of OCCA New Zealand as an independent body in January 1987.

In the international sphere generally I am pleased to confirm that OCCA prestige remains high. I was able to judge this personally in October 1987, when I made a round the world journey, mainly on OCCA business, accompanied by my wife, Hilary. Our journey took us first to the United States where we attended the FSCT Paint Show and Conference in Dallas. Later we visited Japan as guests of the Japan Society of Colour Material for their 60th Anniversary Conference. Finally we stayed in Singapore where I had discussions with some OCCA members about the possibility of forming a branch there. We were given a very warm welcome by the Presidents of FSCT and JSCM and I took the opportunity of having useful discussions with them as well as the Presidents of FATIPEC and SLF and delegates from OCCA Australia and New Zealand.

At home I think OCCA needs to become more outward looking and co-operate more with other associated organisations to the mutual benefit of our industries and members. Recently an important joint meeting was held between representatives of the Paintmakers' Association, Paint Research Association and OCCA concerning cooperation in Education Training and Professional Qualifications. It was established that each Association has an important part to play and a working party has been set up to establish what requires to be done, this augurs well for the future.

I believe the real strength of OCCA is in its sections. What we really need to encourage is far more **active** participation by **more** members in OCCA section activities. Chris Pacey-Day and the staff at Priory House are very willing to assist in any way possible to co-ordinate such activities so that the membership as a whole may benefit. The best example of this to date is the Exhibition which was rejuvenated when held at Harrogate as Surfex 86. The whole organisation and management was by a small committee chaired by Fred Morpeth of Manchester Section and assisted by other members from West Riding and Newcastle Sections, with Priory House staff providing the necessary back up and support services. Surfex 86 was an outstanding success and indications at present suggest Surfex 88 will be even better. I trust that all members will reward the Exhibition Committee for their efforts by giving their full support to Surfex 88 and, if at all possible, attend personally.

Our Conference Officer, Tony Jolly, has plans for our next conference at Chester in 1989 well in hand. He is ably supported by our Technical Officer, John Taylor, and his committee, who are arranging the technical content. Chris Pacey-Day and his staff will be providing the administration and co-ordination of the various activities. To ensure its success, the Chester conference requires much better support from OCCA members than that experienced at Eastbourne in 1987. The organisers are endeavouring to provide an attractive conference package, progress reports are now appearing regularly in JOCCA, please give them your full support.

We offer our sincere congratulations to the London Section on achieving their 50th anniversary. I was fortunate to attend an excellent dinner last month to celebrate the occasion. Officers of the London Section, past and present, were invited. The after dinner reminiscences of past history and success by some of the older members were very entertaining. I understand that the section are receiving much better support at meetings since they recently changed to a new venue. We hope they will continue to be successful for many years to come.

I wish all members a very happy new year and hope that you will all participate to ensure another successful year for OCCA.

John Bourne



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This Award was instituted in 1969 and recognises outstanding and long service to the Association, particularly at section level.

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Kalon Chairman to retire

Leslie Silver, OBE, Chairman of the Kalon Group PLC (President of OCCA 1973-75), has announced his impending retirement. Mr Silver, a major shareholder in the company, stated that there will be an orderly realisation of his holdings.

BIP expansion of speciality resins

Additional production capacity for "Beetle" alkylated resins is being provided at the Oldbury, West Midlands, works of BIP Chemicals Ltd. De-bottlenecking has increased output of the butylated resins plant by 20% in August of this year and a major plant expansion, now nearing completion, will increase capacity for BIP's methylated resins by 50%. Under an agreement which is being concluded with Monsanto Chemical Company, USA, BIP will have the use of their latest commercial, alkylated melamine resin technology and will launch new products during 1988.

Solvent Exxon

Exxon Chemical will be spending \$6 millions on a further expansion of its Antwerp hydrocarbon solvents plant, from 550,000 tonnes per year to 600,000 tonnes by increasing both its fractionation and dearomatisation capabilities. The work on this expansion will be completed by mid-1988. The plant is the largest of its type in the world and is currently producing half a million tonnes a year. Mr Malcolm Law, Exxon Chemical International Director of Performance Fluids, said: "Though the European hydrocarbon solvents market in several fields is stagnant or even declining, we believe there is considerable potential for us and we plan to continue to make the necessary investments to develop our production capability. The expansion of our solvents plant at Trecate in Northern Italy from 90,000 tonnes to 120,000 tonnes was completed in November."

Cuno building new R & D laboratory

Cuno Europe, a manufacturer of filtration and separation technologies has built a new research and development laboratory at their Meriden location. The expanded facility is more than double the size of the old Talcottville laboratory and will centralise all Cuno R & D.

The Perkin-Elmer LIMS 2000 and CLAS user group

The growing number of companies using Perkin-Elmer Laboratory Information Management and Chromatography Laboratory Automation systems (LIMS 200 and CLAS) within the United Kingdom, prompted Perkin-Elmer Ltd to initiate the formation of a User Group. The installations around the United

cont'd overleaf

Hoechst High Chem Strategy leads to sale of Berger

Hoechst UK Ltd has agreed to sell Berger, Jenson & Nicholson Ltd to Williams Holdings PLC. The consideration is £100 million plus the settlement of inter-company debt. The agreement was signed on Friday 4 December and is intended to become fully-effective on 6 January. The offer is subject to the approval of both Williams Holdings' shareholders and the Office of Fair Trading. In recent years, Hoechst's paints division has placed its emphasis on technical high added value industrial paints, automotive paints, vehicle refinishing and specialised industrial paints. Hoechst's acquisitions in 1986 of Renault Paints and of Ault & Wiborg PLC, a company operating in the vehicle refinishing sector, have strengthened its activities within Europe.

In order to sharpen and concentrate the industrial paints business in one group, Hoechst has consolidated these activities in Herberts GmbH in Wuppertal. In the case of Berger, Jenson & Nicholson in the UK, this process included the separation in mid-1987 of their industrial paints business from their decorative paints business. Their former industrial paints business (Berger Industrial Coatings) is now owned by Hoechst UK Ltd (75%) and Herberts

(25%). This company now operates as HPG Industrial Coatings Ltd. Consequently, the importance to Hoechst of Berger's decorative paints business, which historically has been concentrated in countries of the British Commonwealth, has diminished in the light of Hoechst's strategic decision to concentrate on its industrial paint activities.

Williams Holdings is a fast-growing multinational group operating mainly in the engineering field, vehicle distribution, D-I-Y and building products. They recently acquired Crown Paints and Polycell in the UK, thus achieving an important position in the British and international decorative paint and D-I-Y sectors.

Following the divestment of Berger, Jenson & Nicholson, Hoechst intends to increase its activities in the worldwide industrial paints business. Commenting on the sale, Arno Baltzer, the Chairman and Chief Executive of Hoechst UK, said: "The sale of Berger is very advantageous to Hoechst UK, as well as to Williams and to Berger itself. For Hoechst UK it will release resources which will provide an even stronger base for the development of our Hoechst High Chem strategy, with increased local production and the flexibility to make further acquisitions."

Cray Valley Products international sales and marketing conference



Delegates and wives of Cray Valley Products International Sales and Marketing Conference 1987, Tylney

The eleventh Cray Valley Products International Sales and Marketing Conference took place recently. Delegates from twenty eight countries met together at Tylney Hall Hampshire a magnificent Grade 2 listed mansion. The delegates and wives travelled to Tylney from W and E Europe, USA, Mid East, Far East, South East Asia, Australasia and Africa.

The working sessions took the form of Syndicate exercises in which delegates studied a number of the latest CVP resins, ranging from long life alkyds (Synolac 6000 series) to new high technology two component acrylics with novel curing mechanisms (Synolac 890 series). The Syndicates also worked on new ideas for customer relations, developing commercial service and the quality of technical service. The syndicate method was judged by all delegates as a superior method of Conference working compared to conventional "look & listen" lectures. The social programme included an evening cruise on the Thames with a barbeque, river boat jazz band and a tour of Windsor Castle. The conference concluded with a Gala Dinner & Dance, during which a specially commissioned Bronze and Brass Plaque was presented by the Agents to CVP to commemorate fifty successful years of resin developments and sales.

Stop Press: Coates Brothers have acquired Lorilex International (a major French based maker of printing inks) for £56.8 million from Societ Chemique des Charbonnees - CDF Chimie

Kingdom cover a wide variety of applications ranging through standard quality control procedures, contract analysis, research and development and metallurgical testing to the more familiar analytical laboratory requirements. The majority of the sites are members of the User Group. The main aim in setting up the User Group was to provide a forum for the exchange of ideas and expertise between System Managers of LIMS 2000 and CLAS. Outside speakers are brought in to cover broader topics such as system validation, when this is considered necessary. For further information contact Perking-Elmer Ltd, Post Office Lane, Beaconsfield, Bucks, HP9 1QA.

products

Coalescing solvent

Floridienne has available a new low odour coalescing solvent for latex paints, *Velate 262* isodecyl benzoate produced by the Velsicol Corporation. It is a clear light coloured liquid (Alpha 10-40) with a flash point of 174°C COC. In comparison with standard coalescing agents, *Velate 262* gave excellent low temperature coalescence and scrub resistance. The results showed that *Velate 262*, used as a direct replacement, had technical and commercial advantages. *Velate 262* will be supplied in drums and road tankers from UK bulk storage.

Reader Enquiry Service No. 40

Burlington hydraulic pumps

Burlington Engineers has available a new generation of Schwing heavy duty hydraulic pumps. The twin cylinder reciprocating piston design provides Schwing pumps with the ability to transport hitherto unpumpable material ranging from organic sludge with a 45% solid content, to highly abrasive and viscous silica slurry. Three model ranges are designed for 60, 90 and 150 bar delivery pressure respectively. Delivery capacities range from 0.1m³/h. to 115m³/h.

Reader Enquiry Service No. 41

Enhanced Labcal programme for paints and coatings formula design

Kirstol has introduced into the market place Applied Color Systems' (ACS) enhanced version of its *Labcal* programme package for paint and coatings formula design. Unlike "cut and try" formulation programme, *Labcal* allows chemists to design formula ladders from start to finish without relying on previous formula data. Target raw material costs can be specified by the formulator, who can also specify which parts of the formula can be adjusted to meet the specified costs. *Labcal* automatically calculates the required adjustments. Formula ingredients can be

Contd on the next page

Reed £2 million expansion for Paintainer



Reed all plastic lever lid Paintainers on six colour printing line.

Completion of a £2.33 million expansion programme at Reed Plastic Containers' plant in Oakham, Rutland, has doubled capacity for production of its Award-winning "Paintainer" range. These all-plastics lever lid cans, developed primarily for packaging water-based paints, are now generating growing demands from other sectors of the decorative and maintenance products market.

For example Paintainers—now available with capacities of 1-litre, 2.5-litres, a 3-litres promotional size and 5-litres – are proving increasingly popular for packaging adhesives and wood preservatives. A major reason for this is the excellence of the Paintainer closure. This can be levered off without it deforming and effectively resealed so that, if only part of the contents have been consumed, the remainder will be kept fresh for future use. The closure was also a key factor in the Paintainer winning a 1986 'World Star' Award from the World Packaging Organisation.

Commenting upon the doubling of Paintainer capacity Mike Rowley, Reed Plastic Containers' Managing Director says: "We foresee a continuing swing forward into plastic packaging for marketing water-based paints. In fact, our research indicates that the vast majority will be packaged in plastic containers by 1991."

The expansion programme, which created 21 new jobs at the Oakham factory, has involved installation of four new injection moulding machines and a second, highly sophisticated container finishing line. This line, which utilises a number of technological advances, incorporates a further six-colour offset printing machine and, like the first, an automated handling system. The increase in production capacity has also dictated need for substantial investment in

additional warehousing capacity.

Production of Paintainers begins with their bodies being manufactured from polypropylene on a battery of fast-cycling injection moulding machines. They are then conveyed by an automated handling system to the six-colour printer. This unit will maintain the highest standards in reproduction of even the most delicate and demanding multi colour graphics.

Printing on the containers is dried as they pass through a heat tunnel before they are conveyed to the rim welding station. Here, rim sections are ultrasonically welded onto the tops of the containers, providing the strength and rigidity which has proved so important in the success of the Paintainer concept. Then, depending upon their size, the containers move on to a unit where fold-down handles are welded or secured using a unique "cold indent" system.

The Paintainers, with lids being packed separately, are then palletised before being taken into the finished goods warehouse. The pallet loads are finally shrinkwrapped for storage and delivery to customers.

In comparison to metal cans, Paintainers offer a number of valuable advantages for marketing water-based paints and similar products. First and foremost, due to the raw material being chemically inert, there are no rusting problems. Paint manufacturers have identified significant downstream cost benefits with reduced levels of container returns.

There is also no denting, due to the excellent impact resistance of polypropylene. In addition, the design of the containers eliminates any worries about leakage through seams or rivet holes. Other benefits for Paintainer customers are faster filling speeds and much reduced in-plant noise levels.

entered into the programme in a variety of different ways, including: percent total solids by volume or weight; percent total pigment by volume or weight; PVC or P/B or percent vehicle solids by volume or weight. From information entered, Labcal quickly generates laboratory formulas with cost and physical data, plus QC and manufacturing information. Separate reports can also be produced for formula cost analysis, factory batch tickets and volatile organic compound analysis.

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meetings

OCCA New Zealand Convention

OCCA New Zealand Inc 26th Annual Convention will be held on 21-23 July 1988 at the Travel Lodge Hotel, Rotorua, New Zealand.

Seminar Topics include: Electron Beam Curing, Packaging Coating Trends, Colour Innovation, Paint & Adhesive Developments, Innovation in R & D Durene By-Products, Ceramic Coatings, Where Now and Technological Marketing Trends. For further information contact: OCCANZ, Convention '88, Box 5019, Wellington, New Zealand.

1988 SLF Congress

The 12th SLF Congress, organized by the Federation of Scandinavian Paint and Varnish Technologies to (Skandinaviska Lackteknikers Forbund) will be held in Helsinki on 9-11 May 1988. The address of the Congress bureau is: Tikkurila Oy SLF Congress, PO Box 53, 01301 Vantaa, Finland. OCCA members wishing to present a paper on behalf of OCCA should contact the General Secretary.

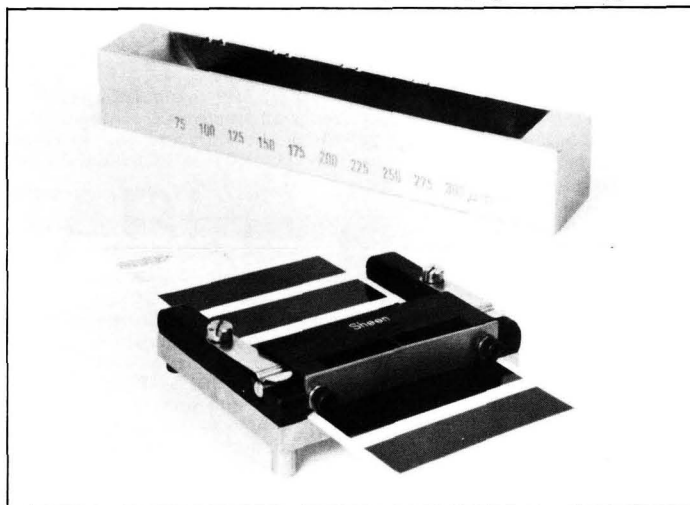
Spechem China '88

The 1988 Chinese International Speciality Chemicals & Engineering Exhibition will be held on 17-21 October 1988 at Beijing National Agricultural Exhibition Centre, China. For further information contact: Sino Trade Promotions (operated by Sinostar Ltd) 15A Wing Cheong Comm. Bldg, 19-25 Jervois St Central, Hong Kong.

Hazpak courses for dangerous substance drivers

Drivers of vehicles carrying packaged dangerous substances have four months to complete the training needed under new regulations coming into force next year. Regulation 7 of the Road Traffic (Carriage of dangerous substances in packages etc) Regulations 1986, which relates to driver training, will be in operation from 6 April 1988.

The duty to ensure that drivers receive the relevant training rests with the



New Sheen paint test applicators. Top: levelling/sagging tester. Bottom: Biddle duplex applicator.

Sheen Instruments has added two new applicators to its range of paint testing instruments. The *levelling/sagging tester* is a simple comparison test of these two properties of paints in the period between application and drying. A stainless steel frame allows both tests to be carried out. One edge contains five pairs of gaps for the levelling test and the other edge has ten steps for the sagging test. The second new instrument is the *Biddle duplex applicator*, which is used for the application of two parallel films on a hiding power chart for comparison of glass, opacity, colour etc. The unit is supplied with 100 Ref. 301L hiding power charts.

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operator of the vehicle and guidance on how to comply with this requirement is already available as an Approved Code of Practice on the Regulations. The level of training needed by a driver depends on how frequently he or she is involved in the carrying of dangerous substances.

The 'Hazpak' course, which lasts for two days, is recommended for someone who is regularly involved in carrying significant quantities of dangerous substances and is run by a number of centres all over the country. They are all approved by a consortium of trade associations, including the Road Transport Industry Training Board, which acts as the administrative centre. For further information contact: HSE, Baynards House, 1 Chepstow Place, Westbourne Grove, London W2 4TF. Tel: 01-229 3456.

Safety & Toxicity in the chemical laboratory and chemical industry

The Royal Society of Chemistry will hold a 1 day symposium on "Safety and Toxicity in the Chemical Laboratory & Industry" at the University of Aston, Birmingham on 1 March 1988. Amongst the several speakers will be Dr O'Neill of the PRA who will talk on "Aspects in the Paint Industry" and Mr B. G. Wallace of Shell UK who will talk on "Safety in the Chemical Industry with

special reference to epoxides." For further information contact: Dr H. Warson, Conference Organiser, 284 Warwick Road, Solihull, West Midlands B92 7AF, UK.

literature

Bayer Polyurethanes in Building

Bayer UK have produced an 8 page colour leaflet entitled "Polyurethanes in Building." This leaflet describes the contribution polyurethanes have made to wall and roof cladding, as integral insulation in building blocks, window frames - or spray on systems for roofs, factory-made or in-situ pipe and tank insulation. The leaflet is available from: Dept H, Polyurethane Business House, Strawberry Hill, Newbury, Berkshire RG13 1JA.

New Hays Coating Literature

A new brochure on coating techniques for Magnetic media, barrier coating, printed circuits has been produced by Hays Technology Systems. Copies are available from Hays Technology Systems, Dixon House, Works Road, Letchworth, Herts.

Radiation Curing Silicones

The latest brochure in the Th. Goldschmidt AG series, "Goldschmidt Informiert . . ." deals with its new product group of radiation curing silicones. The 36-page publication contains five papers covering both the chemistry of radiation curing silicone coating products as well as the electron beam and UV equipment required for curing. Copies of this "Goldschmidt Informiert" brochure are available from A. L. Smith, Th. Goldschmidt Ltd, Tego House, Victoria Road, Ruislip, Middx. HA4 0YL.

Wailles Technical Data Sheets

A complete set of technical data sheets encompassing the range of industrial protective coatings is available from Wailles Dove Bitumastic plc. For the data sheets contact Neil Ormrod at Wailles Dove Bitumastic plc, Hedgeley Road, Hebburn, Tyne & Wear, NE31 1EY.

Deadly Maintenance in the Chemical Industry

Maintenance work in the chemical industry causes 175 accidents a year yet most of these incidents could be prevented, says a report published today by the Health and Safety Executive (HSE). The report, by the HSE's Accident Prevention Advisory Unit (APAU), is based on a study of all accidents and dangerous occurrences in the chemical industry notified to HM Factory Inspectorate between 1982 and 1985.

David Eves, Chief Inspector of Factories, said that in the majority of cases the errors that led to the incidents could have been avoided by a little forethought and consideration. He also stated that many chemical companies make a determined effort to manage health and safety effectively but the study shows that without proper consideration of maintenance work incidents can still occur

at apparently well run sites. The report "Dangerous Maintenance," highlights what is going wrong and why, and most importantly offers practical advice on how to stop it occurring again.

The report "Dangerous Maintenance - A study of maintenance accidents in the chemical industry and how to prevent them" ISBN 0 11 883957 8 is available from HMSO Price £7.50.

people

Board changes at Crown Paints

Following the acquisition of Reed International's Paint & D-I-Y Group by Williams Holdings last July Crown Paints has now been formally designated the Paint Division of Williams Holdings PLC, with Paul Lever appointed as the Managing Director.

A new Board structure will be implemented from 1 January 1988. The Directors are: Paul Lever - Divisional Managing Director, Brian Baker, Managing Director - Technical Services, John Asher, Managing Director - Industrial Paints & Inks, Aiden Brophy, Managing Director - Overseas Companies & Export, Geoff Christiansen, Administration Director, Ken Gill, Manufacturing & Distribution Director, Terry Hudson, Marketing Director, Ron Hughes, Financial Director and Eric Thurston, Sales Director - UK & Europe.

Three directors are retiring from the company in December. They are: Tom Graham - Technical Director, Peter Cocks Commercial Director and Tom Gould - Company Secretary.

Their service to the company totals 85 years and the Board has thanked them for their contribution and expressed good wishes for their retirement. Tom Graham

and Peter Cocks will be retained as consultants by Crown to complete specific projects during 1988.

Colourgen expands

Following its successful first year Colourgen Ltd the European subsidiary of Colorgen Inc the specialist colour management company which manufactures colour matching and prediction systems is expanding both office space and staff at its Warrington HQ. Paul Webster joins the company to work as a programmer under the direction of the Technical Director Dr Jim Nobbs who is also Lecturer in Colour Chemistry at Leeds University. The company aims to broaden its existing software and add this to existing facilities in the UK and USA. Paul has considerable experience in various computer languages and this will enable him to contribute to the continuing effort to improve the speed and accuracy of the company's colour match prediction software and to utilise the latest co-processor technology. The company has also expanded its office space to provide more comfortable customer demonstration facilities.

New man takes over at Heraeus

Heraeus Equipment, leading supplier of laboratory, electronics and environmental simulation equipment, has a new managing director - Trevor Dixon. Mr Dixon has more than 20 years' experience in the scientific and industrial instrument field, having previously been managing director of Beckman, which he joined from Pye Unicam where he was marketing services manager. Mr Dixon (51) a qualified chemist is also a freeman of the City of London and a liveryman of the Worshipful Company of Scientific Instrument Makers.

bsi news

British Standards

The following new and revised publications are available:

BSI publications are obtainable from: BSI Sales Department, Linford Wood, Milton Keynes MK14 6LE (orders by post only).

The following new and revised publications are available:

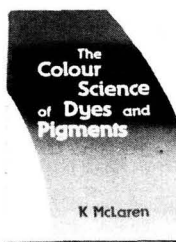
BS 6900: Specification for raw, refined and boiled linseed oils for paints and varnishes.

BS 4725: Specification for linseed stand oil for paints and varnishes.

PD 6518: Prefabricated temporary road marking materials for use at roadworks.

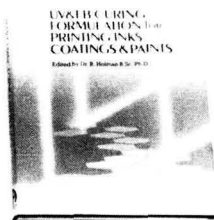
OCCA ties at £4.25 each are now available from the Association's offices, with a single Association Insignia on either a blue or maroon background.

PUBLICATIONS AVAILABLE THROUGH THE ASSOCIATION



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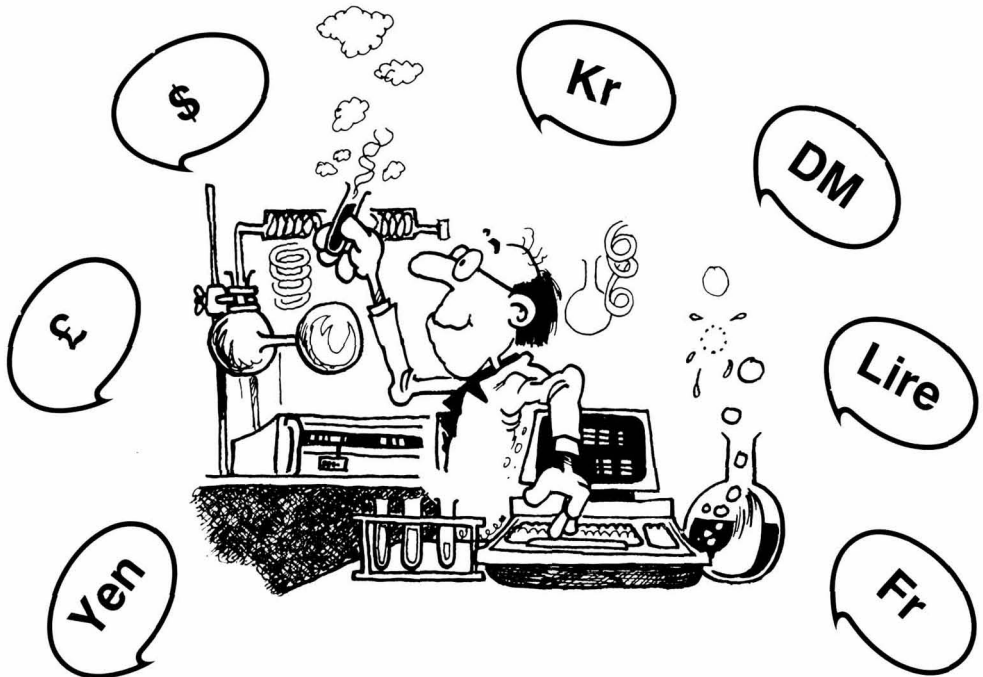
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21 - 24 JUNE AT THE GROSVENOR HOTEL

PROFITABLE **R&D?**



CALL FOR PAPERS

“Profitable Research & Development” has been chosen as the main theme for the Chester 89 International OCCA Conference. For the first time, the conference has been broadened to encompass the important commercial aspects of product research and development. Papers will be presented on the commercial and technological development of new products, instruments and machinery for the Surface Coatings Sector.

An added feature with the conference will be facilities for a Surface Coatings mini-exhibition, where companies will be showing the latest products, instruments and machinery on the market.

Interested **Authors** and **Exhibitors** should fill in the **Call for Papers Leaflet** enclosed with this issue.

Paint and Surface Coatings

Theory and Practice



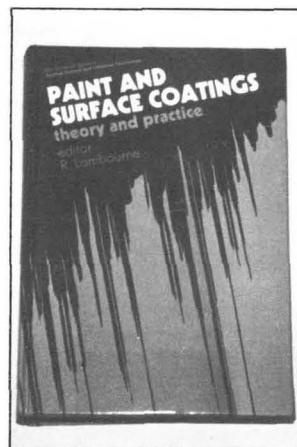
Edited by R. LAMBOURNE,
Technical Manager,
INDCOLLAG (Industrial Colloid
Advisory Group), Department of
Physical Chemistry, University of
Bristol.

696 pages; Published April 1987; £79.50/\$152.75
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CONTRIBUTORS:

- D. A. Ansdell, Technical Manager, Automotive Products*.
- J. Bentley, Senior Scientist, Research Department*.
- T. R. Bullett, Retired. Formerly Research Director, Paint Research Association, Teddington, Middx.
- A. Doroszkowski, Research Department*.
- F. K. Farkas, Consultant, FKF & Associates.
- J. A. Graystone, Technical Manager, Research Department*.
- R. A. Jeffs, Retired. Formerly with Research Department*.
- W. Jones, Research Department*.
- R. Lambourne, University of Bristol.
- A. H. Mawby, Director, Refinish Marketing, PPG Industries (UK) Ltd, Ladywood, Birmingham.
- Miss J. F. Rolinson, European Technical Manager for Products and Materials*.
- T. A. Strivens, Senior Scientist, Research Department*.
- G. P. A. Turner, Technical Manager, Industrial Coatings Research*.

* ICI Paints Division, Slough.



CONTENTS: Paint Composition and Applications—A General Introduction; Organic Film Formers; Pigments for Paints; Solvents, Thinners and Diluents; Additives for Paint; The Physical Chemistry of Dispersions; Particle Size and Size Measurement; The Industrial Paint Making Process; Coatings for Buildings; Automotive Paints; Automotive Refinish Paints; General Industrial Paints; An Introduction to Rheology; The Rheology of Paints; Mechanical Properties of Paints and Coatings; Appearance Qualities of Paint—Basic Concepts; Specification and Control of Appearance; Durability Testing.

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Control of organic pigment dispersion properties

R. B. McKay

Ciba-Geigy Pigments, Hawkhead Rd, Paisley, Renfrewshire PA2 7BG, UK

Summary

Organic pigment powders (or granules) are finely divided, molecular crystalline solids. The minor dimension of the crystals is usually in the size range from 0.02 to 0.05 micron. The crystals are assembled into an aggregate structure. The effectiveness of a given pigment in imparting colour to application systems, such as printing inks and paints, is dependent upon how finely it can be dispersed in the application medium. Certain physical characteristics of the aggregate structure determine the level of dispersion attainable and how readily it can be achieved. These are examined here in the context of copper phthalocyanine and calcium 4B toner pigments for viscous printing inks (low polarity).

It is shown that even in a highly compact aggregate structure the crystals are not irreversibly bound together. Good dispersibility is achievable by minimising the strength of attraction between crystals. The requirements are reduction in the area of contact between crystals and lowering of the polarity of the crystal surfaces. These requirements can be met by controlled crystal growth to produce an open aggregate structure with minimal face-to-face packing of crystals, and by treatment of the crystal surfaces with certain additives. The structural aspects are more important than the magnitude of the interaction of dispersion medium with the crystal surfaces, certainly in the case of calcium 4B toners.

Introduction

Organic pigments consist of finely-divided molecular crystals that are essentially insoluble in application media, such as inks and paints¹. They have to be dispersed therein by mechanical means. The primary function of an organic pigment is to impart colour. Its effectiveness in this respect is determined largely by how finely divided it is in the final ink or paint film. Colour strength (intensity per unit weight of pigment) increases markedly with decrease in particle size in the submicron region. Optimum colour strength from a given pigment in practice requires a mean particle size of the order of 0.1 micron or less.

The pigment crystals are of various shapes, commonly brick, rod or plate, with the minor dimension usually in the range 0.02 to 0.05 micron. Ideally for high colour strength the particles in the final ink or paint film should be individual crystals. Where particles cannot be approximated to spheres or cubes (most cases), the minor dimension is the optically effective size². For example, calcium 4B toners consist of rectangular plate-shaped crystals. It has been shown that in viscous printing inks it is the plate thickness that relates to colour strength³. In this case the crystals are substantially individually dispersed.

The onus on the pigment manufacturer is to produce pigments that can be dispersed readily and substantially as individual crystals in the application media. This requires control of crystal size and aggregate structure of pigment powders and granules. The present paper examines and identifies what factors influence the separation of crystals on dispersion in a viscous printing ink medium in the context of attaining optimum colour strength. Little has been published on this topic, which may conveniently be referred to as dispersibility.

Two types of pigment are featured, namely copper phthalocyanine (C.I. Pigment Blue 15)⁴ and calcium 4B toner (C.I. Pigment Red 57.1). These are respectively the most important blue type for general use and the most important red type for printing inks.

Weak flocculation profoundly affects the rheological behaviour of organic pigment dispersions. This topic has not been considered here. In the present viscous ink systems, which are printed under high shear conditions as very thin films, the effect of weak flocculation on colour strength is minimal. Weak flocculation in fluid, low polarity dispersions of copper phthalocyanine pigments has recently been discussed⁵.

Experimental

The pigments used here were free from additives, unless specifically otherwise stated. The copper phthalocyanine pigments consisted of brick-shaped or rod-shaped crystals with approximately square cross-section. Crystal dimensions were determined manually from transmission electron micrographs at 60K total magnification. Five electron micrographs were used per sample and a minimum of 30 crystals sized per micrograph. The geometric specific surface area S was then calculated on the basis of total area divided by total weight.

The calcium 4B toner pigments consisted of rectangular plates. The mean plate thickness t was evaluated from X-ray diffraction line broadening; the main face area A was determined from transmission electron micrographs. These methods have been described elsewhere³. The geometric specific surface area S was then calculated from these mean dimensions.

The BET specific surface areas were determined from nitrogen gas adsorption data obtained at -196°C and $P/P_0 < 0.3$ using a Carlo-Erba Sorptomatic 1800 instrument. Pigment samples were outgassed overnight at room temperature prior to use. The cross-sectional area of the nitrogen molecule was taken to be 0.162 nm^2 . Heats of immersion of outgassed pigments in viscous printing ink medium and pure liquids were measured using a Calvet ambient microcalorimeter operated at 25°C . This method was based on that of Ref 6. Individual results quoted are the average of at least three determinations.

The ink medium consisted of 50% (w/w) phenolic resin, 32% alkyd resin, 18% high boiling aliphatic hydrocarbon. It had a viscosity of approximately 8.5 Pa.S. at room temperature. Dispersibility assessments were made using standard ink testing procedures. Pigments were dispersed in the ink medium by cavitation stirring followed by 3-roll milling. Quality and ease of dispersion (collectively referred to as dispersibility) were assessed by optical microscopy and colour strength development methods, respectively, using conventional ink technology procedures. Further details of test methods are given in Ref 3.

Results and Discussion

From previous work on copper phthalocyanine⁷ and calcium 4B toner pigments³ it was found that nitrogen molecules do not penetrate between the crystals in face-to-face contact under the conditions used in the determination of the BET specific surface area. S_{BET} is therefore a measure of the external surface (cf. Ref 8) of the aggregate structure. The ratio S_{BET}/S is therefore an indication of the degree of face-to-face packing of crystals – the higher the degree then the lower the ratio.

β -Copper phthalocyanine pigments with good dispersibility in viscous printing ink media are commonly prepared by the following route. Non-pigmentary (large crystal size) copper phthalocyanine is dry milled to produce pigmentary α, β -form mixture, which is then converted to β -form by heating in an organic liquid under optimised conditions. The α, β -form mixture has extremely poor dispersibility, so much so that it cannot be meaningfully assessed. α, β -CuPc-I is an example. It consists of small brick-shaped crystals ($S = 130 \text{ m}^2\text{g}^{-1}$) in a compactly aggregated state ($S_{\text{BET}} \approx 2\text{m}^2\text{g}^{-1}$; $S_{\text{BET}}/S \approx 0.02$). Face-to-face packing of crystals is dominant. Treatment with organic liquid grows the crystals (reduction in S) by a recrystallisation process involving $\alpha \rightarrow \beta$ -form conversion⁹. S_{BET} increases markedly and $S_{\text{BET}}/S \rightarrow 1$. Figure 1 illustrates this effect with data obtained for β -form pigments prepared by stirring α, β -CuPc-I with a variety of liquids for 24 hours at 60°C. Mean crystal volume has been used to indicate extent of crystal growth. So the crystal growth has resulted in an open aggregate structure in the pigment powders with edge-to-edge and/or edge-to-face packing of crystals. Face-to-face packing has been virtually eliminated. The good dispersibility of pigments thus produced is exemplified with a variety of commercial β -form pigments in Table 1. The highest dispersibility ratings (arbitrary units) are given by the pigments with large crystal size.

Now consider how openness of aggregate structure leads to enhanced dispersibility. Table 2 compares α, β -CuPc-I with an optimally grown β -CuPc-III ($S = 94 \text{ m}^2\text{g}^{-1}$, $S_{\text{BET}} = 89 \text{ m}^2\text{g}^{-1}$). Heat of immersion values ($\Delta_w H$) are given for a number of liquids chosen such that there was no $\alpha \rightarrow \beta$ -form conversion with α, β -CuPc-I under the conditions used in the calorimetry. The heats of immersion per unit of the BET surface area ($\Delta_w H/S_{\text{BET}}$) are in the range 91 to 98 mJm^{-2} for β -CuPc-III; the corresponding heats for α, β -CuPc-I are almost two orders of magnitude higher. Large differences of this type have been encountered in previous work and the cause established¹⁰. The effect is due to the liquid molecules (at 25°C) penetrating between crystals in face-to-face contact in α, β -CuPc-I and thus gaining access to crystal surfaces in the interior of the aggregate structure that are inaccessible to nitrogen molecules in the BET area determination (at -196°C).

Consideration of the heats of immersion per unit area of the crystal surfaces ($\Delta_w H/S$) now provides information on the extent of penetration of the liquid molecules into the aggregate structure of α, β -CuPc-I. The $\Delta_w H/S$ values for α, β -CuPc-I are comparable to those for the open-structured β -CuPc-III. This indicates that the liquid molecules gain access to virtually all the crystal surfaces in α, β -CuPc-I. So penetration between crystals in face-to-face contact is virtually complete. The face-to-face contacts are therefore not of an irreversible type (i.e. not sintered nor cemented).

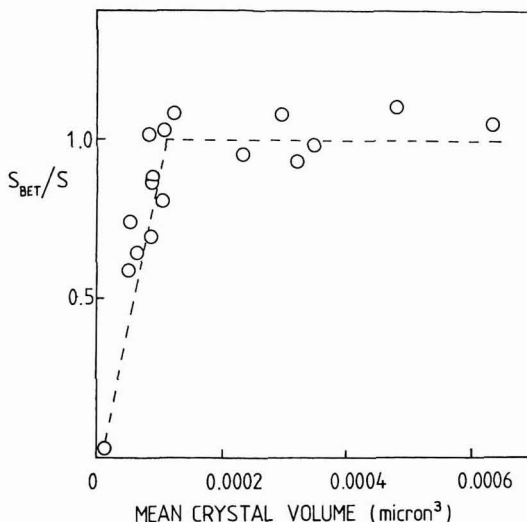


Figure 1. Effect of crystal growth on the aggregate structure of β -CuPc pigments prepared from α, β -CuPc-I.

Table 1

Effect of crystal size (hence openness of aggregate structure) on dispersibility of β -copper phthalocyanine pigments.

β -CuPc Pigment	Mean crystal volume, (micron^3)	Dispersibility*
1	0.000189	6
2	0.000175	6
3	0.000146	6
4	0.000141	6
5	0.000138	6
6	0.000058	5
7	0.000050	4
8	0.000079	3
9	0.000074	3
10	0.000027	3

* arbitrary units (the higher the better)

Table 2

Heat of immersion data for copper phthalocyanine pigments.

Liquid	α, β -CuPc-I			β -CuPc-III		
	$\Delta_w H$ (Jg^{-1})	$\frac{\Delta_w H}{S_{\text{BET}}}$ (mJm^{-2})	$\frac{\Delta_w H}{S}$ (mJm^{-2})	$\Delta_w H$ (Jg^{-1})	$\frac{\Delta_w H}{S_{\text{BET}}}$ (mJm^{-2})	$\frac{\Delta_w H}{S}$ (mJm^{-2})
Ethanol	12.9	6400	99	8.1	91	86
n-Butanol	12.3	6100	95	8.2	92	87
n-Hexanol	12.3	6100	95	8.2	98	87
n-Heptane	11.9	5900	92	8.1	91	86
n-Dodecane	11.8	5900	91	7.9	89	84

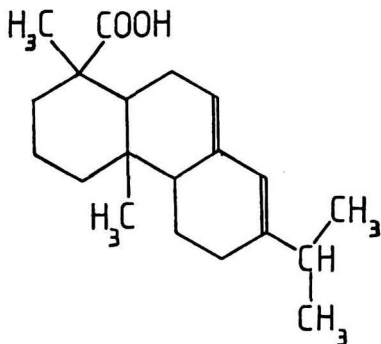


Figure 2. Chemical constitution of abietic acid.

The markedly inferior dispersibility of α,β -CuPc-I can now be attributed simply to the much larger area of contact of the crystals in the aggregate structure. This gives a more cohesive structure in which it is more difficult to separate the constituent crystals than is the case with the open structure of β -CuPc-III.

Dispersibility of open structured pigments can be improved further by treatment of the pigment surfaces with additives of the abietic acid type (referred to here as resin). Figure 2 shows the chemical constitution of abietic acid. Calcium 4B toners for viscous printing inks provide a good example. These pigments are prepared by an aqueous precipitation process and contain up to 20% resin by weight. An excess of resin is present during crystal formation and growth stages, and serves to control crystal size. The amount of resin retained in the final pigment is inversely related to crystal size. It is therefore adsorbed on the crystal surfaces.

In calculating the specific surface area of the crystals allowance has to be made for the resin content, which is not detected by the crystal sizing methods. This is done by considering the resin to be distributed in a uniform adsorbed layer of thickness x on the crystal surfaces. The geometric specific surface area calculated by the following equation³

$$S_c = \frac{2(A^{1/2} + 6x + 2t)}{(t + 2x)(A^{1/2} + 2x)\rho}$$

relates to the external surface of this layer. ρ = density of the pigment (1.58 g.cm^{-3} by method of Ref.11).

S_{BET} values are a large fraction of the S_c values, so the aggregate structure is of a predominantly open type with edge-to-edge and/or edge-to-face contact of the (rectangular plate-shaped) crystals predominating³. Dispersibility in the viscous printing ink medium increases with increase in the amount of resin per unit area of crystal surface. This is exemplified in Figure 3 and can be explained as follows:

An unresinated calcium 4B toner has a polar surface and extremely poor dispersibility in the viscous ink medium. This polarity is shown by immersion calorimetry. Heats of immersion results are given in Table 3 for the unresinated pigment 4B-21. Heats of immersion ($\Delta_w H$, Jg^{-1}) in polar liquids are much greater than in non-polar liquids, such as

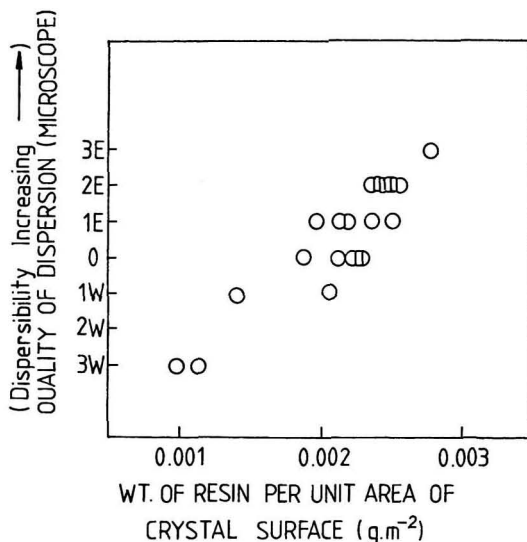


Figure 3. Dependence of the quality of dispersion of calcium 4B toners in ink medium on surface concentration of abietyl resin (optical microscopy assessments on comparative basis relative to a standard dispersion = O, E = better, W = worse).

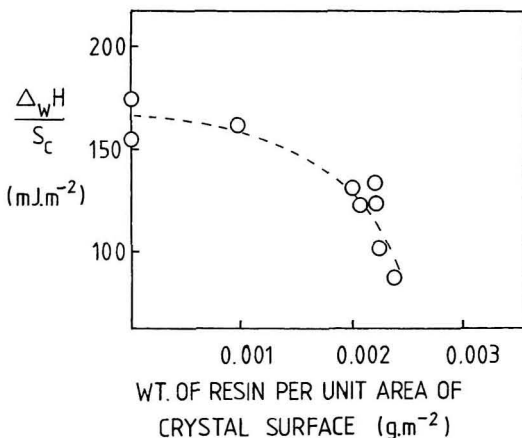


Figure 4. Effect of surface concentration of resin on heat of immersion (per unit area of resin-coated crystal surface) of calcium 4B toners in ink medium.

Table 3

Heats of immersion of resin-free 4B toner and β -copper phthalocyanine pigments in various liquids.

Liquid	$\Delta_w H$ (Jg^{-1})	
	4B-21	β -CuPc-III
n-Heptane	5.5	8.1
n-Dodecane	6.0	7.9
Ink Medium	10.9	—
n-Propanol	18.7	7.7
2-Ethyl Hexanoic Acid	22.5	9.9

aliphatic hydrocarbons. The spread of values is much wider than that obtained with the relatively non-polar β -CuPc-III. The high value with 2-ethyl hexanoic acid is attributable to the specific interaction of the carboxyl group with polar sites in the calcium 4B surface. The ink medium interacts much less strongly than 2-ethyl hexanoic acid, which is a reasonable model for abietic acid. On this basis the ink medium is unlikely to displace abietic acid from the pigment surface.

Treatment of the calcium 4B toner surface with abietyl resin reduces the heat of interaction with the ink medium. This is shown in Figure 4 where $\Delta_w H/S_c$ values decrease as the surface concentration of resin increases. The effect can be attributed to the resin molecules being adsorbed to the surface with the carboxyl group in contact with the surface and the hydrocarbon residue orientated outwards. This gives effectively a hydrocarbon surface and lowers the surface polarity.

The improved dispersibility conferred on the calcium 4B toners by abietyl resin is attributable to the lower surface polarity and hence weaker interaction between crystals at areas of contact in the aggregate structure. In this way the crystals become more readily separable in the dispersion process in the viscous ink medium.

Note that the properties of the aggregate structure of the pigment powder appear to be more important than the heat of interaction of the dispersion medium with the crystal surfaces. Dispersibility of the calcium 4B toners increases as the heat of immersion decreases.

Conclusions

Good dispersibility in the present context is dependent primarily upon the low cohesive strength of the aggregate structure of the pigment powder. The strength of interaction between the constituent crystals has to be minimised. This requires a substantially open aggregate structure with predominant edge-to-edge or edge-to-face contact of crystals, so that area of contact is minimised. Ideally face-to-face packing should be eliminated. Further benefit can be gained by lowering the surface polarity of the pigment crystals and thereby reducing the strength of interaction at the areas of contact.

Acknowledgement

The author wishes to thank Mrs A. MacLennan, Mr I. McCormack and Dr R. R. Mather for most of the experimental work.

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The effect of water-soluble contaminants at the steel/paint interface on the durability of the paint coating.

M. Morcillo, S. Feliu, J. C. Galván and J. M. Bastidas

Centro Nacional de Investigaciones Metalúrgicas, Ciudad Universitaria, 28040 Madrid, Spain

Abstract

Painting on steel which has been corroded in polluted atmosphere can noticeably shorten the planned life of the paint system. Sulphur dioxide and sodium chloride polluting agents in the atmospheres bring on the formation of water-soluble sulphates and chlorides in the rust, with these substances being responsible for the premature deterioration of the paint coating.

In this study, the effect that variable levels of these corrosion-stimulating substances at the steel/paint interface have on the behaviour of typical paint systems is analyzed. The experiments were carried out in different atmospheres in Spain and in climatic and salt fog chambers.

It has been found that even levels of 100 mg/m² of NaCl and 1000 mg/m² of FeSO₄ can be sufficient to significantly shorten the life of many paint systems in common use. On the other hand, this phenomenon is not observed in a "zinc-rich" silicate-type coating.

Introduction

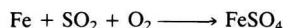
Frequently paint is applied after the steel structure has been erected, meaning that it has been exposed to the corrosive action of the atmosphere for a certain length of time. Polluting agents in the atmosphere, normally sulphur dioxide, resulting from the combustion of different fuel products, and sodium chloride from the sea can play a decisive role during this period, as they result in the formation of water-soluble sulphates and chlorides that promote the formation of rust. Premature deterioration in paint coatings applied on steel contaminated with these corrosion-stimulating substances have been reported repeatedly by different users of paint coatings (electric companies, shipyards, etc.).

This problem is presently a source of concern for the international scientific and technical community. For this reason attempts are being made, for example, within the ISO TC/35 SC/12, for standardizing methods of detection and measurement of these residual polluting agents. However, parallel studies for the purpose of clearing up certain basic questions are missing. For example: What chemical substances or combinations of them are capable of interacting with the underlying steel and of producing these premature deteriorations in the paint coating? Are there critical concentration levels for these corrosion-stimulating substances that, once exceeded, produce a significant deteriorating effect on the coating? How do different paint systems behave in these situations? What roles do the thickness of the coating and the exposure environment play? etc. In this study, an attempt is made to answer some of these questions.

Background

This topic was brought to light and attracted the curiosity of scientists when, in 1959, J. E. O. Mayne¹ published an

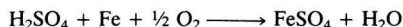
interesting article on painting rusty steel that had been rusted in an atmosphere polluted with SO₂. He demonstrated the existence of seasonal variations on the properties of rust in terms of its FeSO₄ content. A harmful effect on the stability of different paint systems applied on rusted steel was attributed to this agent whenever its concentration reached a certain level. The presence of FeSO₄ at the steel/paint coating interface promotes an accelerated formation of rust, which rapidly leads to blistering and to the destruction of the coating. Following a first stage, in which the SO₂ is adsorbed by the rust (FeOOH), there is a formation of FeSO₄ through the direct action of the SO₂ and the oxygen in the atmosphere



Then, more rust is formed from the FeSO₄ in the presence of moisture and oxygen, with the liberation of sulphuric acid



which, in turn, reacts with the iron to form more FeSO₄



According to these reactions, in which the sulphuric acid regenerates, there is a continuous formation of rust as long as the supply of oxygen and moisture to the surface of the metal is ensured. In Figure 1, the process of the formation of rust caused by FeSO₄ is shown². Similar processes have been suggested for other ions (Cl⁻, S⁼)^{3,4}. In the literature, these chemical substances are referred to as corrosion-promoting, stimulating or accelerating agents.

In his article, Mayne¹ also demonstrated that a large percentage of the FeSO₄ was found in the rust firmly adhered to the steel, and that it was not possible to eliminate it by mechanical wire-brushing. Later, Tanner⁵ and Schwartz⁶ found FeSO₄.4H₂O in the shallow pits under the layer of rust by using X-ray diffraction. Evans⁷ comments on the fact that those areas form the anodic points of differential aeration microcells and that the SO₄⁼ ions, through the electrochemical process, move toward them to give rise to the formation of FeSO₄ as an anodic product (Figure 2). Harrison and Tickle⁸ also verified the preferential accumulation of Cl⁻ ions at the metal oxide interface. It was Schwartz who first began to refer to the "nests" of FeSO₄. Ross and Callaghan⁹ demonstrated, by using an electron probe microanalyzer, that those nests represent the ultimate stage in the distribution of the FeSO₄ in the rust.

Recently, Calabrese and Allen¹⁰ have detected the existence of corrosion-simulating substances trapped between the superficial folds of the steel produced by the blasting operation, which cause the well-known

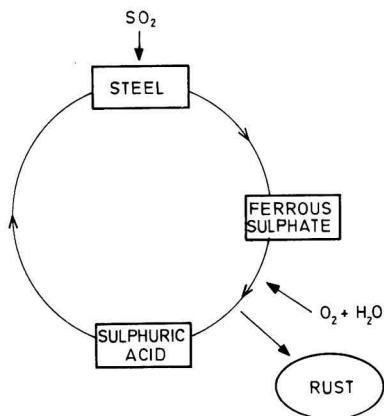


Figure 1. Cyclic mechanism of the formation of rust on steel as caused by ferrous sulphate².

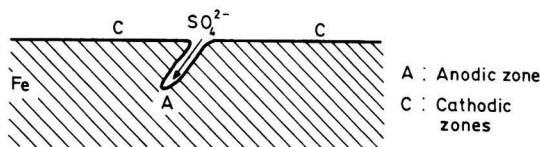


Figure 2. Schematic diagram according to Evans⁷, of the electrochemical process by which the sulphate ions are situated at the bottom of the superficial pits on the metal.

phenomenon of flash rusting. In these capillary areas, low relative humidities can be sufficient cause for giving rise to the condensation of moisture, and to foster the electrochemical corrosion process, accentuated by the presence of those substances.

Experimentation

Mild steel plates, 3 mm thick, hot rolled and having their millscale intact (grade A of the Swedish standard SIS 055900-1967) was used for preparing the specimens of 12.5 × 25 cm. These were shot blasted (S-280) until the ASa 3 grade of this Swedish standard was achieved.

The presence of water-soluble contaminants at the steel/paint interface was simulated by placing onto the specimens variable quantities of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and NaCl, in amounts equivalent to the contamination levels shown in Table 1. These salts had previously been dissolved in a mixture of distilled water and methanol. The latter of these two compounds was added to help solvent evaporation. To decrease as far as possible the formation of rust on the underlying steel, the contaminated specimen was introduced into a stove to hasten the evaporation process. Then, different paint systems, with different dry film thickness, were applied by air spraying (Table 2).

Table 1

Saline contamination levels tested in mg/m^2

NaCl	FeSO_4
20	150
100	250
500	500

Once the coatings had been cured, the specimens were subjected to the following tests:

1. Natural weathering tests involving these environments (rural, urban and industrial) of different corrosivity (Table 3). At present, only results for four years of exposure are available.
2. Salt Spray (fog) test (ASTM B 117), with a testing period of 3200 hours.
3. Artificial weathering tests in climatic chambers, in accordance with the following specifications:
 - 3.1. DEF-1053 Method No. 26. Alternative running of carbon arcs in 6-hour periods, with a chamber temperature of about 40°C. In between carbon arc operational periods, deionized water spraying with a resistivity over 300,000 $\Omega \cdot \text{cm}^2$. Testing period: 3500 hours.
 - 3.2. ASTM G53. Condensation (44°C) and cooling (65°C) cycles were used of 4-hour duration each, with a 0.5 hour in between. An ultraviolet radiation was continuously produced. Testing period: 2300 hours.

Results and discussion

Regular inspections of rusting and blistering grades shown on painted surfaces were carried out. Failure was evaluated according to ASTM D 610 and ASTM D 714 standards, respectively. Serving as a control test for the influence of salt contamination at the steel/paint interface, steel panels similar to those used previously, but on which no contaminant agent whatsoever was deposited, were tested simultaneously.

The deterioration of the paint coating initially manifests itself through a fine blistering (8-9 MD-D of the ASTM D714 standard) on the whole surface of the specimen (Figure 3). In some systems, such as in the case of the alkyd and epoxy-polyurethane resins, the deterioration has already appeared within the first six months of exposure to the atmosphere. In other systems, the deterioration takes longer to appear (1-2 years). The type of atmosphere and the thickness of the coat also play a role in the period of time needed for the phenomenon to appear. However, in 3-4 years of exposure to the atmosphere, the deterioration becomes manifest in all the atmospheres in which the study was carried out and in practically all of the systems and coating thicknesses taken into consideration. An exception to this behaviour is found in those systems consisting of a primer base of zinc silicate, in which blistering did not take place, at least during the 4 years of exposure to the atmosphere. This tolerance on the part of zinc-rich coating regarding saline contamination at the steel/paint interface is in accordance with what Evans^{7,11} initially stated, on noting that paint with a high zinc content would be one of the solutions to this problem.

Later, depending upon the type of paint system, the blisters either stabilize or progress very slowly (which is the case of the polyurethane and epoxy/polyurethane systems), or they may increase, even bursting open, due to the accumulation of oxide on their interior (Figure 4). The chlorinated rubber and vinyl systems show this effect quite clearly.

Atmospheric exposure

After 4 years of exposure to the three natural environments taken into consideration in the study (Table 3), no blistering was apparent on the control panels. However, this phenomenon occurred whenever the level of NaCl

Table 2
Characteristics of paint systems

Paint System	Primer	Undercoat	Topcoat	Total thicknesses (µm)
Oil/Alkyd	INTA* 164103	—	INTA 164218	1: 90, 125, 175, 250 2: 65, 100, 130
Alkyd	INTA 164201	—	INTA 164218	65, 95, 125
Chlorinated Rubber	INTA 164705	INTA 164701 A	INTA 164704 A	75, 95, 130
Vinyl	INTA 164604	INTA 164602 A	INTA 164603 A	60, 90, 120, 150
Polyurethane		RENFE 03.323.125		120, 150
Epoxy/Polyurethane	MIL C-82407	RENFE 03.323.125		100, 150, 190
Zinc Silicate	INTA 164408	—	—	60, 95
Zinc Silicate/ Chlorinated Rubber	INTA 164408	Tie Coat	INTA 164704 A	80, 110, 140
Zinc Silicate/ Vinyl	INTA 164408	Tie Coat	INTA 164603 A	100, 120, 150

* INTA: Instituto Nacional de Técnica Aeroespacial Standards.

Figure 3. Fine initial blistering of the coating of paint due to the presence of saline contaminant agents at the metal/paint interface.



THICKNESS: 75 µm



THICKNESS: 95/130 µm

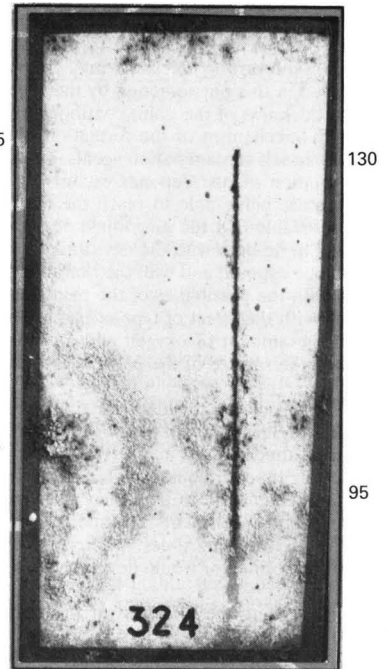


Figure 4. Appearance of a chlorinated rubber coating with consequence of the existence of saline contaminant agents at the steel/paint interface. The blisters have burst open due to the accumulation of rust on their interior.

pollution at the interface was of 500 mg/m². When the NaCl level was of 100 mg/m², the deterioration of the coating was only detected in certain situations: this is the case of the polyurethane system in the rural atmosphere or the chlorinated rubber system in the other two atmospheres (Table 4).

As it seemed strange that the phenomenon did not appear when the contaminant was FeSO₄, precisely the contaminant mentioned the most in the literature, it was considered that possibly the concentration of FeSO₄ deposited at the interface (150-500 mg/m²) was not sufficient to bring on the phenomenon. Thus, new specimens were prepared, with FeSO₄ contamination levels of 500, 1000 and 2500 mg/m², on which new coatings were applied, and they were exposed to two marine atmospheres included in another test program carried out in coastal locations. After two years of exposure, it was observed that three of the four paint systems tested (Table 5) showed blistering induced by the presence of FeSO₄ when this contaminant has a level of 2500 mg/m². Once again, the chlorinated rubber system shows itself to be more sensitive to contamination, since the level of 1000 mg/m² was sufficient in this case to produce blistering. These results agree with those found by Mayne¹, who showed, at Cambridge, that the critical level of FeSO₄ in rust is that of 0.6 mg/sq. in. (937.5 mg/m²).

In Table 6, the results obtained in the different atmospheres are shown for the 500 mg/m² level of NaCl at the steel/paint interface. The blistering, as has been mentioned previously, was generally small-sized (6-8 on the ASTM standard) and affected a large percentage of the test surface (MD-D of the ASTM standard). Regarding the rust grade, two coatings, that of the chlorinated rubber type and that of the vinyl type, were characterized by their low resistance to the development and accumulation of oxide inside the blisters. The polyurethane and epoxy/polyurethane systems do not show rust on the paint surface (rust grade No. 10).

An interesting question may be: What are the roles played in this phenomenon by the type of atmosphere and the thickness of the paint coating? On commenting on the cyclic mechanism of the formation of rust in presence of soluble salt contamination agents, we noticed that repeated formation of rust depends exclusively on the oxygen and moisture being able to reach the metal. It would thus be foreseeable that the magnitude of the phenomenon would have to do both with the environment to which the coating is to be exposed and with the characteristics of coating; that is, with the possibilities of the painted surface to be wetted and with the effect of type of paint, coating thickness etc., on the amount of oxygen and moisture to be transferred from the surface of the paint to the metal.

On observing the data for the three locations given in Table 6, it can be said that the deterioration arises in all the three atmospheres in practically the same manner; the wetting times are quite similar and high enough to allow all the phenomenon to take place. It would be interesting to carry out experiments in atmospheres with very low and very high wetting times, where phenomenon either could not be verified or would develop very rapidly, respectively.

Regarding the thickness of the coat of paint, its effect does not seem of a great significance either, at least in the range of thickness (60-150 μm) taken into consideration;

Table 3

Corrosivity characteristics of the different atmospheres

Test Station	Corrosivity		Classification of the Atmosphere
	Bare Steel (μm/year)	Bare Zinc (μm/year)	
El Pardo, Madrid	14.7	0.94	Rural
Ciudad Universitaria, Madrid	27.9	0.78	Urban
San Adrián del Besós, Barcelona	46.4	1.59	Industrial

Table 4

Contaminant levels (mg/m²) of NaCl at which a significant deterioration of the paint system is observed in the different atmospheres in this study.

Paint System	Atmosphere†		
	Rural	Urban	Industrial
Oil/Alkyd	500	500	500
Alkyd	500	500	500
Chlorinated rubber		100	100
		500	500
Vinyl		500	500
		100	
Polyurethane	500	500	500
Epoxy/Polyurethane	500	500	500
Zinc Silicate (Z.S.)	—	—	—
Z.S./Chlor. Rubber	—	—	—
Z.S./Vinyl	—	—	—

† — = No effect observed until 500 mg/m² of NaCl.

Table 5

Tests in marine atmospheres. Results after two years of exposure.

Paint System	Total Thickness μm	Levels† of FeSO ₄ for which the blistering phenomenon appears induced by the presence of the saline contamination at the interface
Alkyd	180	—
Chlorinated Rubber	170	1000 and 2500 mg/m ²
Chlorinated Rubber/Acrylic	150	2500 mg/m ²
Vinyl/Alkyd	180	2500 mg/m ²

† — No effect observed until 2500 mg/m² of FeSO₄.

blistering occurs, in general, regardless of the thickness of the coats of paint taken into consideration in the study, although in some cases (Figure 4) the phenomenon seems to decrease in intensity the thicker the coat of paint.

Salt fog test

This is a very aggressive environment. The effects of the

Table 6

Blister and rust grades after 4 years of exposure to the steel atmospheres for the different paint systems applied on a steel surface contaminated with 500 mg/m² of NaCl

Paint System	Thickness (µm)	Rural Atmosphere		Urban Atmosphere		Industrial Atmosphere	
		Blister Grade	Rust Grade	Blister Grade	Rust Grade	Blister Grade	Rust Grade
Oil1/Alkyd	90	—	10	—	10	6M	10
	125	—	10	6M	10	6F	10
	175	8MD	10	8F	10	6M	10
	250	6D	10	8MD	10	6M	10
Oil2/Alkyd	65	8F	10	6MD	8	6M	10
	100	8-10D	10	4-6MD	9	6M-MD	8
	130	8D	10	4MD	10	6MD	8
Alkyd	65	6D	10	6MD	10	6MD	9
	95	6D	10	8M	10	6MD	9
	125	6D	10	—	10	—	10
Chlorinated Rubber	75	6-8D	0	6D	6	6D	3
	95	8D	1	4-6D	6-7	6D	4
	130	8D	1	8MD	7	6D	6
Vinyl	60	Lifting	8	—	10	4MD	9
	90	Lifting	8	6-8D	10	6D	5
	120	8-9MD	10	—	10	4D	10
	150	2MD	2	4D	10	4D	10
Polyurethane	120	8D	10	4D	10	4D	10
	150	8D	10	4D	10	4D	10
Epoxy/Polyurethane	100	8D	10	6D	10	4D	10
	150	8D	10	4-6D	10	6D	10
	190	8D	10	4D	10	6D	10
Zinc Silicate (Z.S.)	60	—	10	—	10	—	10
	95	—	10	—	10	—	10
Z.S./Chlorinated Rubber	80	—	10	—	10	—	10
	110	—	10	—	10	—	10
	140	—	10	—	10	—	10
Z.S./Vinyl	100	—	10	—	10	—	10
	120	—	10	—	10	—	10
	150	—	10	—	10	—	10

— = No effect observed.

saline contamination agent at the interface and of the exposure to the salt fog are both superimposed. This makes it difficult to distinguish between both effects in a separate way. Thus, in the case of the oil/alkyd and alkyd systems, having a lower chemical resistance, deterioration (blistering and oxidation) of the coating by the salt fog is so intense that it masks the possible effect due to the saline contamination at the interface (Table 7). In the other paint systems, the appearance of fine blistering over the entire surface of the test piece has been attributed to the effect of the contaminant agent. In a way similar to the tests conducted in the atmosphere, this blistering is not observed when dealing with low levels of FeSO₄ contaminant agent (150-500 mg/m²), nor in the case of using a zinc silicate primer either.

In Table 7 the results obtained for the different levels of the NaCl contaminant agent are given. From among these results, it is of interest to point out that the blistering appeared quite rapidly (400 hours) in the case of the polyurethane and epoxy/polyurethane systems when the

NaCl contamination was of 500 mg/m². Also, it can be noted the significant effect of the contaminant agent even at low concentrations (20 and 100 mg/m²).

Artificial weathering tests

Accelerated weathering tests were performed in special test chambers, all of which were similar in that a coated specimen was exposed in a controlled environment containing a high intensity ultraviolet light source and a facility for spraying water onto the test surfaces.

In the accelerated weathering tests, some of the paint systems showed a certain degradation (changes in colour and /or brightness, chalking, etc.), which was surface deterioration mainly^{12,13}. For this reason, although these tests are, perhaps, the most suitable for indicating the behaviour of the coating itself, they do not always offer an accurate indication of the corrosion protection provided by the coating. As their effects may not be able to penetrate the coating, these tests do not seem to be specially valid for

Table 7

Salt Fog Test. Test time, in hours, for fine blistering on the whole surface of the specimen (6-8 MD-D of the ASTM D714). Results corresponding to the NaCl contaminant.

Paint System	NaCl contamination level (mg/m ²)	Paint film thicknesses (μm)	Salt Fog (hours)	Paint film thicknesses (μm)	Salt Fog (hours)
Oil1/Alkyd	20	120, 165	*	120, 175	*
	100	105, 115	*	220, 270	—
	500	110, 160	*	180, 250	—
Oil2/Alkyd	20	90, 110	—	100, 145	—
	100	70, 100	—	125, 165	—
	500	60, 90	1150	115, 170	1150
Alkyd	20	85, 115	—	135, 230	—
	100	75, 120	—	95, 160	—
	500	90, 140	—	95, 160	—
Chlorinated Rubber	20	60	1800	90, 135	—
	100	65	1800	90, 130	—
	500	65	1000	90, 125	1000
Vinyl	20	60, 90	—	110, 155	—
	100	55, 80	2400	110, 160	—
	500	50, 100	1400	160, 205	—
Polyurethane	20	90, 120	1400	115, 140	1400
	100	90, 115	700	130, 145	700
	500	90, 110	400	110, 130	400
Epoxy/Polyurethane	20	100, 145	1400	185, 205	1400
	100	70, 130	550	185, 200	550
	500	105, 145	400	165, 180	150
Zinc Silicate (Z.S.)	20	70	—	95	—
	100	70	—	90	—
	500	60, 65	—	85, 95	—
Z.S./Chlorinated Rubber	20	80, 110	—	120, 135	—
	100	80, 120	—	115, 135	—
	500	85, 130	—	125, 140	—
Z.S./Vinyl	20	100, 110	—	130, 145	—
	100	95, 105	—	140, 150	—
	500	110, 120	—	120, 150	—

— = No effect observed * = Masked possible effect

studying the influence of saline contamination at the metal/coating interface. In fact, only in some cases blistering has been detected after over 300 hours of testing (Table 8).

Conclusions

It has been found that levels of 100 mg/m² of NaCl or 1000 mg/m² of FeSO₄ at the steel/paint interface can be sufficient to significantly shorten the life of many paint systems in common use for exposure to the atmosphere. A "zinc-rich" coating (of the silicate type) seems to tolerate the existence of saline contamination at the interface. The deterioration of the coating (blistering and metal oxidation) practically does not depend upon the thickness of the dry coat of paint, at least regarding the range of thicknesses taken into consideration (60-150 μm).

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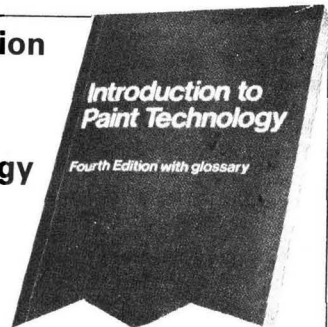
Table 8

Results corresponding to the accelerated weathering tests conducted in climatic chambers

Paint System	ASTM G-53	DEF-1053 Method No 26
Oil1/Alkyd	—	NaCl, 500 mg/m ² (*) NaCl, 100 mg/m ² (*)
Oil2/Alkyd	NaCl, 500 mg/m ² (*)	NaCl, 500 mg/m ² (*)
Alkyd	—	NaCl, 500 mg/m ² (*)
Chlorinated Rubber	NaCl, 500 mg/m ² (*)	—
Vinyl	—	—
Polyurethane	—	—
Epoxy/ Polyurethane	—	NaCl, 500 mg/m ² (*)
Zinc Silicate Z.S.	—	—
Z.S./Chlorinated Rubber	—	—
Z.S. Vinyl	—	—

* = Blistering — = No effect observed

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Development of novel driers

N. Usman

Paint Research Association, Teddington, Middlesex TW11 8LD, UK

Summary

This paper describes the investigation carried out by the Paint RA into possible alternatives to the conventional cobalt/lead/calcium systems used in alkyd based paints.

Introduction

Most solvent borne decorative paints dry by reaction with atmospheric oxygen. Until recently cobalt plus auxiliary driers such as lead and calcium, have been the most commercially available drier systems known to give effective drying under the low temperature, high humidity conditions which characterise most of the UK painting season.

The use of lead has been under increasing attack by environmentalists. The Ninth Report of the Royal Commission on Environmental Pollution was presented to Parliament in April 1983. This dealt with all aspects of lead in the environment and one of the recommendations (Chapter IX, item 12) stated:

“The maximum permitted concentrations of lead in paint for household use should be progressively reduced to a level comparable to that applied in the USA and in the meantime all paints containing more lead level than this should carry a warning label”.

The Royal Commission also recommended that the lead level should be reduced to the current USA limit of 0.06%. Such reduction would mean that lead driers could no longer be used which would result in paints of inferior performance unless a satisfactory substitute for lead driers can be found.

In response to the Royal Commission Report the Paintmakers Association in February 1985, voluntarily agreed that “lead driers will cease to be used in gloss paints and their related systems, for whites and near whites by 1 January, 1986, and for colours by 1 July, 1987”.

Background and experimental work

Over the past nine years the Paint RA has been involved in three major projects concerned with developing safe, effective and economical replacements for lead or cobalt as used in a conventional cobalt/lead/calcium drier system.

From 1978-1979 a research programme was carried out for the Paintmakers Association to identify possible alternative paint driers to those based on cobalt and lead. At that time cobalt supplies were seriously threatened by disorders in Central Africa and the use of lead was under attack by environmentalists especially in the United States.

A first phase programme^{1, 2} consisting of a literature and

Table 1
Selected alkyd resins

Alkyd type	%NV	Acid value	% Solids 2 Poise	% Phthalic Anhydride	% Oil Oil Type	Polyol
Ennelkyd 1143	100	12	—	16	73 Soya	Pentaerythritol
Synolac 28W	65	10	48	26	65 Soya	Pentaerythritol
Synolac 50W	75	9	51	22	70 Linseed	Pentaerythritol
Synolac 76W	60	8	48	20	73 Tall	Pentaerythritol
Alftalat VAS 3312	46	10	35	28	46 Soya	Pentaerythritol
Alftalat AS 483	50	14	30	38	48 Soya	Glycerol
Synolac 60W	70	10	54	24	68 Soya	Pentaerythritol

information survey followed by a short screening programme on candidate materials resulting from the survey was agreed in July 1979. This was then followed by a second phase programme³ which involved a more detailed study of promising materials including complexes of manganese with 2,2' dipyridyl and 1,10 phenanthroline, the novel manganese complexes supplied by Dr McAuliffe, and copper naphthenate complexed with 2,9 dimethyl 1,10 phenanthroline. These materials were evaluated in Paint RA formulated gloss paint and undercoat, and several commercial formulations which were supplied by the manufacturer, with and without driers. The testing programme involved:

A manual touch dry test at half hour intervals

A hard dry test to BS 3900: Part C3, 1971 (mechanical thumb)

A recoatability test carried out 24 hours after spreading. This involved 20 seconds vigorous rubbing with a white spirit soaked tissue.

A colorimetric determination of whiteness (expressed according to the whiteness scale in ASTM E313-73).

This investigation indicated that a combination of manganese driers with 2,2' dipyridyl (0.02% manganese with a 1:3 molar ratio of 2,2' dipyridyl) could be an effective and economically acceptable alternative to the usual cobalt/lead driers in air drying paints.

The above research programme was then followed in 1984 by a full technological evaluation of manganese/2,2' dipyridyl⁴ in over 30 primer, undercoat and topcoat formulations supplied by Paint RA member firms. The tests were carried out on the drier-free commercial paints after addition of the novel driers. The one-pack drier system was incorporated in the drier free paints to give 0.02% manganese based on resin non-volatile. The fully formulated commercial paints served as the controls. The testing schedule involved measuring drying performance at ambient and at low temperature as well as determining colour, storage stability and resistance to natural and artificial weathering.

The results of the tests for room temperature drying, storage stability and gloss and chalking after artificial weathering showed that the novel drier paints were fully comparable with the controls. Tests for low temperature drying and colour showed that approximately half the paints tested were better than or comparable with the controls. Results from checking and cracking tests on wooden panels after artificial weathering indicated that by

and large the novel paints were either comparable or better than the controls. The natural weathering tests will extend towards the end of 1987. By January 1987 the test panels had been exposed for approximately two years and there were no significant differences between the durability behaviour of controls and novel paints.

In 1985, five classes of materials were evaluated as possible replacements for the lead driers in a conventional cobalt/lead calcium drier system⁵. These materials were metal driers, metal amines, oxygen carriers, one electron oxidising agent and enzymes.

The first stage of this work was to select test paints i.e. paints which dry in the presence of lead and fail in its absence at low temperature and high humidity. Seven alkyd resins were selected to formulate seven topcoats and seven undercoats at Paint RA. Each alkyd resin was chosen for its unique oil length, oil type and polyol type and they are listed in Table 1.

For each paint system four drier/paint combinations were prepared as given in Table 2.

Table 2

	% Drier metal on non-volatile resin		
	Cobalt	Calcium	Lead
1	0.05	-	-
2	0.05	0.2	-
3	0.05	-	0.5
4	0.05	0.2	0.5

All the 56 combinations were tested for their drying performance at low temperature high humidity. (Fison's environmental chamber operating at $5 \pm 2^\circ\text{C}/90\%$ RH). The paint films were left in the chamber for 24 hours then tested immediately for recoatability and hard dry as described before for the earlier evaluation programme. Four of the 14 paints tested needed lead in order to dry effectively. These paints were:

73% Tall Oil Undercoat (Synolac 76W)
65% Soya Undercoat (Synolac 28W)
73% Soya Undercoat (Ennelkyd 1143)
73% Soya Topcoat (Ennelkyd 1143)

Three of these paints, 73% Soya topcoat, 65% Soya undercoat and 73% tall oil undercoat were selected for evaluation of metal driers. Results obtained from these evaluations showed that undercoats were more difficult to

dry than the topcoats. Metal driers like zirconium and cerium could dry the topcoat but none of the metal driers (Zr, Ce, Nd, Ba and Bi) tested were found suitable for drying the undercoat.

This study was further extended to selected commercially available air-drying gloss and undercoat formulations supplied by members of Paint RA both as fully formulated products and as drier-free paints. The five classes of materials being evaluated as alternative drier systems were incorporated into the drier-free product and the performance at low temperature high humidity compared with the corresponding fully formulated paints. The commercial formulations consisted of both white and coloured paints. Colour measurements were also carried out on all white paints evaluated.

Promising drier systems in commercial white paints

A wide range of alternative drier systems were found suitable for drying the commercial topcoat as listed below:

- 0.1 Co, 0.2 Ca
 - 0.13 Co, 0.2 Ca, 0.2 Zr (Cozirc 69)
 - 0.05 Co, 0.2 Ca, 0.2 Zr, 2,2' dipyridyl (Zr:Amine 1:2)
 - 0.05 Co, 0.2 Ca, 0.02 Mn, 2,2' dipyridyl (Mn:Amine 1:3)
 - 0.05 Co, 0.2 Ca, 1:10 Phenanthroline (Co:Amine 1:3)
 - 0.05 Co, 0.2 Ca, 0.01 Copper naphthenate
 - 0.05 Co, 0.2 Ca, 0.1 Benzoyl peroxide
 - 0.05 Co, 0.2 Ca, 0.01 D-amino acid oxidase
 - 0.05 Co, 0.2 Ca, 0.01 Peroxidase
- } Commercial samples
supplied in solid form

N.B. All drier concentrations were based on resin non-volatile. Fully formulated commercial paints and conventional combination of driers (0.05 Co, 0.2 Ca, 0.5 Pb) added to the drier free paint served as the controls.

For the undercoat, only a limited number of drier combinations were satisfactory, listed as follows:

- 0.1 Co, 0.2 Ca
- 0.13 Co, 0.2 Ca, 0.2 Zr (Cozirc 69)
- 0.05 Co, 0.4 Ca, 0.2 Zr
- 0.05 Co, 0.2 Ca, 0.01 Copper naphthenate
- 0.05 Co, 0.2 Ca, 0.02 Benzoyl peroxide
- 0.05 Co, 0.2 Ca, 0.1 D-amino acid oxidase

It was not possible to carry out extensive study on these drier systems in terms of storage stability and reproducibility. Some repeat tests on copper naphthenate and enzyme systems indicated loss of drying which could be due to the adsorption of the materials onto the pigment surface or in the case of enzyme to the unstable nature of the compounds.

Conclusions

The earlier two phase research programme led to the conclusion that combination of manganese driers with 2,2' dipyridyl could be an effective and economically acceptable alternative to the conventional drier system. The follow up work on the full technological evaluation of manganese/2,2' dipyridyl indicated that although this system could not be regarded as a universal replacement for paint driers currently in use, there was a significant proportion of commercial paints where it could be utilised without detriment.

The initial screening of the Paint RA formulations demonstrated the importance of selecting the suitable paint system when evaluating drier combinations. For example, a linseed oil based alkyd was found to dry effectively in the absence of lead. Clearly, if this resin was included in the drier evaluation work, an inert substance used in conjunction with cobalt and calcium would have been reported as a lead drier replacement.

The results obtained from the evaluation of five classes of materials as alternative driers showed that in the absence of lead the top coats were easier to dry than the undercoats at low temperature high humidity. From the materials found effective for drying, benzoyl peroxide is unsuitable because of its toxicity and instability and high levels of cobalt are undesirable because they lead to problems of durability. Copper naphthenate must be the more attractive option from the commercial point of view as it is not only readily available but also used by the industry and general public as a wood preservative. It also has the additional advantage of being considerably cheaper. Although the material has an intense green colour its drier activity is such that it can be used at concentrations low enough to prevent discolouration even in brilliant white topcoats. The problem of loss-of-dry on storage remains to be solved but once this is overcome the technical, economic and safety advantages of copper naphthenate should ensure that the material is widely used as a lead drier replacement.

Drier mechanisms

Recently Paint RA has launched a three year research project on Drier Mechanisms. The purpose of this project is to define the essentials of the drying process and how this can be controlled by the choice and optimum application of driers. The ultimate aim is to guide the paint formulator in the selection of drier systems that perform well even under adverse weather conditions.

References

(1, 3, 4 and 5 available to Paint RA members)

1. Falla, N. A. R., Usman, N., Smith, G., Swampillai, G. J., Schweitzer, H. R. and Starkie, A. J., "Alternative Driers to Cobalt and Lead", Paint RA Research Contract Report 1979 No. Q9/C1/G(R).
2. Anderson, G. de W. and Falla, N. A. R., "Alternative Driers to Cobalt and Lead", *JOCCA*, 1983.
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4. Usman, N., Sharma, B. K. and Falla, N. A. R., "Assessment of a Novel Manganese Drier (Part 1)", Paint RA Research Contract Report 1985 No. TR/4/85.
5. Usman, N. and Mitchard, N., "Non-Lead Driers", Paint RA Research Contract Report 1986 No. TR/2/86.

Reliability of Durability Testing

Dear Sir,

In his letter (*JOCCA*, October 1987) Mr Bullett raises the possibility that loss of entrained solvent may be responsible for the loss of mass from thick paint films, and suggests an exposure with a heat source only to settle the question. Unpublished work by Simpson and McEwan at Tioxide showed no loss of mass for a pigmented acrylic/MF paint film after 2,000 hours of exposure in this manner. Identical paints exposed to carbon arc UV radiation for the same time lost 24 mg, from the same area (90cm²) as was used in Figure 7 of my paper. An Airdrying alkyd paint did lose 7 mg when exposed to the heat source, and this compares with 61 mg under carbon arc radiation. Thus it would seem that acrylic/MF paint mass loss results would not be complicated by loss of solvent, but airdrying alkyd paints do show significant mass loss not arising from direct film degradation. This may be due to continuing cross-linking, loss of adsorbed volatiles, loss of entrained solvent or a mixture of all three.

Tioxide Group PLC,
Stockton-on-Tees,
Cleveland TS18 2NQ
England

Yours faithfully

D. V. Moulton
12 November 1987

Colour pigment failure – street name plate signs

Dear Sir,

I would like to know if a reader could offer information on methods to be employed to prevent the colour reducing effects of ultra violet light on the coloured pigments used on the Council's street name plates.

Our street name plates have an aluminium back plate, a white vinyl is blended or painted with colour which is vacuum heat treated onto the plate, the letters are applied and again the plate is subjected to a vacuum heat treatment. The background colour is brown, the letters are white.

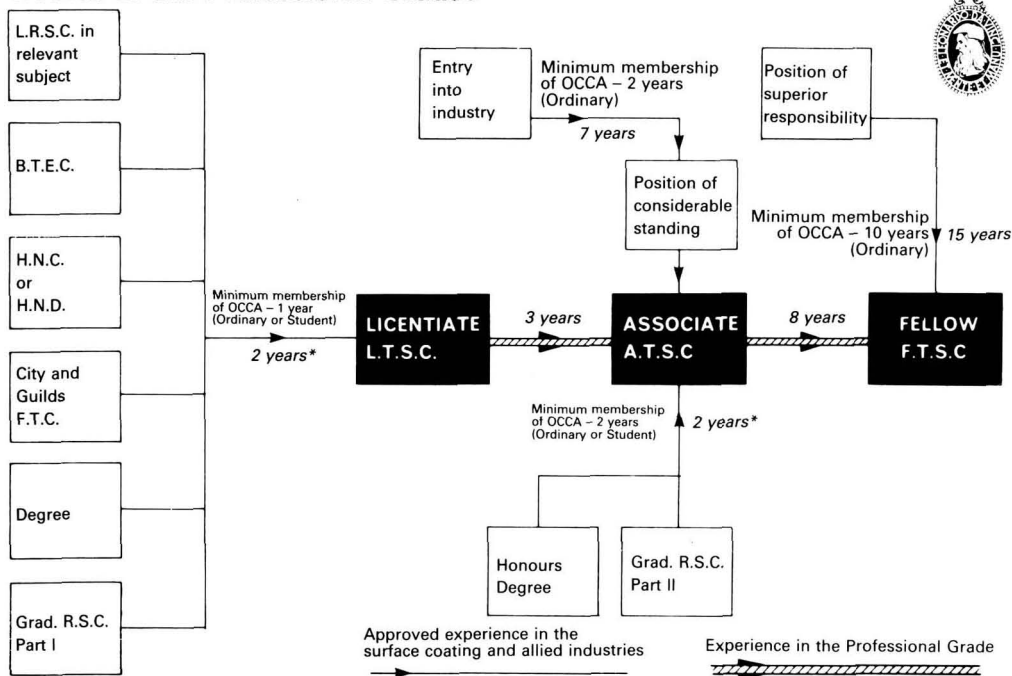
Over a period of say 6 years the brown colour fades to a pale grey and consequently makes reading the plate difficult. I would, therefore, be interested to learn of any recommendations for repainting existing plates as an interim measure and in the long term of any techniques for producing more permanent colour fixing on new plates, could we for example use a clear varnish or lacquer with an ultra violet mask?

The Building Control Surveyor,
City of Wakefield Metropolitan District Council,
West Yorkshire

Yours faithfully,
B. E. Lea
11 November 1987

Optional Professional Grade for Ordinary Members

Routes to the Professional Grades



Manchester Section.

Printing half tone flexo

The first ink lecture of the 1987/88 session was held at the Silver Birch, Birchwood Centre, Warrington, on Monday the 15 November 1987 and a paper entitled "Printing Half Tone Flexo" was presented to the 85 members and guests present, by Mr R. B. Kinkead of Coates Brothers Inks Ltd.

Half Tone Flexo is a pictorial representation, and quality depends on the team of Machinery, Photography, Printer, Ink Manufacturer and Plate Maker all working together. The advantages of this type of printing are that it can be used on flexible substrates, lower cost, and practical.

In the early days quality was not of a high standard for a number of reasons concerning the Stack Presses used, however improved presses, modified inks, improved plates, and better photographic techniques now give much enhanced quality.

The aim is to produce an accurate pictorial representation by a series of dots, using four colours, yellow, magenta, cyan and black. The ink plays a major factor in this, but cannot cure all the ills from imperfect machinery, however good quality cannot be reproduced without good inks.

To conclude, with up to date printing techniques, and inks, half tone flexo can now match litho and gravure printing for quality.

The lecture was followed by an extensive and lively question time, the vote of thanks was proposed by Tony Wright, and those present were then able to look at a min-exhibition demonstrating the quality of half tone flexo, before participating of a Buffet sponsored by Coates Brothers Inks Ltd.

M. G. Langdon

London Section

Colour difference measurements

The third technical meeting in the current programme of the London OCCA section took place on Thursday, 19 November 1987.

The venue of "The Naval Club" is continuing to be a success. The attendance of this joint meeting between OCCA and the Society of Dyers and Colourists, producing standing room only for latecomers.

The guest speaker for this joint meeting was Dr Bryan Rigg from Bradford University and he presented a paper entitled "Colour difference measurements for quality control in Paint and Textiles".

Dr Rigg opened his lecture by displaying the CIE colour difference formula, adopted some 10 years ago and proceeded to explain and discuss the formula, with its

strengths and weaknesses. He then outlined the development from the C lab formula and the experimental work carried out by E. McDonald of J. P. Coates Ltd and then continued with the work carried out at Bradford University by himself and his research students to further develop and improve the formula. He presented a number of formulae including JPC79, CMC and BFD. He expressed the view that these were major improvements over the C lab formula.

During his discussion of the CMC formula he indicated that this was in fact the formula most likely to be adopted by the BSI as its standard colour formula in the future.

Dr Rigg throughout his talk used numerous overheads and slides to illustrate the various formulae, chromaticity diagrams and data prepared during this research.

He concluded by expressing his view that big strides have been made since the introduction of "C" lab and that "CMC" represents such an improvement. However, if such research work were allowed to continue even these new formulae could be further improved.

A vote of thanks to Dr Rigg for this interesting talk was proposed by Dr Banfield.

P. C. Neal

Newcastle Section

Matted coatings

The second meeting of the 1987/8 session took place at St Mary's College, University of Durham, on 5 November 1987. The speaker on this occasion was Herr A. Elbrechter of Degussa who gave a talk on matting agents "Matted Coatings for many reasons".

He began by reviewing the historical use of matting agents, especially in the decoration of woodwork, walls and ceilings of stately homes, where a combination of waxes, matting agents, even sanding, was used to achieve the desired aesthetic effects. The wider modern use for decorative, technical, defence and safety reasons was illustrated with slides of office furniture, laboratory equipment, camouflage and automobile interior surfaces.

Herr Elbrechter defined the properties of an ideal matting agent as easy dispersability with resistance to over-dispersion, high efficiency, high transparency, good suspension, mar-resistance, versatility of use, durability and negligible thixotropic effect. These were illustrated in a number of ways:

First he showed the consistency of performance of an established coated grade when incorporated by Red Devil shaker (5-25 minutes), high speed disperser (5-70 minutes) or ball mill (2-8 hours): gloss values at 45°, 60° and 85° showed little change despite large differences in shear-rate and time.

Next, a comparison of four grades; two relatively coarse

(one coated), one finer coated grade and a very fine, high efficiency type, showed the two coated grades to give constant 60° gloss values, irrespective of shear and time, whereas the high efficiency grade gave gloss differences of 30%-40%: to overcome the latter effect a photomicrograph clearly showed the benefits of the master batch technique for incorporation of matting agent.

Diagrams and photographs were used to illustrate the physical process of matting being an effect produced by diffuse reflectance of incident light from a rough surface. It became clear that matting agents are most effective in solvent/water-thinned coatings, where loss of bulk volume during the drying process causes the film surface to shrink around the matting agent particles. With solvent-free or very high solids coatings matting is very difficult because shrinkage effects are much reduced.

Herr Elbrechter discussed the development of the Degussa range of matting agents from 1955 to 1975. The early coarse, low efficiency types gave way to others having, in turn, easier dispersability but poorer suspension, better suspension but some overcoatability problems, low moisture content for use in moisture sensitive systems, good dispersability/suspension and overcoatability and, finally, an extremely fine thermal silica having very high efficiency, the very highest transparency and extreme versatility.

He concluded with some graphs showing sources of gloss variation in the use of matting agents:

- (i) the effect of binder wetting efficiency, showing variable angle gloss differences with three different matting agents in either two-pack polyurethane or nitrocellulose systems: the latter showed uniform gloss changes with all three matting agents, whereas the poorer wetting DD system showed wide variations.
- (ii) the influence of film thickness was shown with a polyurethane varnish flatted with O.K.412, a coated grade, and measured at 45°, 60° and 85°: gloss values were significantly higher at greater thickness, such as one might obtain at overlap joints on walls.
- (iii) the influence of application method was shown with a low-gloss stoving enamel applied at 30 d.f.t. by either spray or drawdown: the latter showed 5% higher gloss.

Question time afterwards dwelt on topics as diverse as how to ensure uniformity of gloss, the nature of the coatings used on matting agent particles, shrinkage of high-solids UV-cured coatings and means of adding matting agents to a factory batch without excessive dust or loss of expensive raw material into the exhaust ventilation system. The Chairman, Mr S. Lynn, then thanked Herr Elbrechter for a most enjoyable lecture.

It must be noted that the lecture had been interrupted, as was a large assembly at dinner in the same building, by the fire bells ringing; at which point about two hundred people trooped outside into the Guy Fawkes Night air to await the arrival of the Fire Brigade. Surprisingly, on such a busy night for them, three appliances arrived in double quick time, to be informed that the cause of the alarm was merely a young lady's electric iron, which had burnt out and

alerted a smoke detector, thus triggering the alarm. Within five minutes everyone was allowed back inside to resume their activities. The interruption did not deter Herr Elbrechter who accepted it all with good humour and an amusing reference to Guy Fawkes himself.

J. Bravey

Midlands Section

DPP pigments

The second meeting of the Midland Section was held on the 19 November 1987 at the Clarendon Suite, Stirling Road, Edgbaston, Birmingham. Members and guests heard Dr A. Iqbal of Ciba-Geigy Ltd give a talk on "DPP Pigments - A new generation of heterocyclic pigments".

Dr Iqbal said he would divide his talk into two parts, firstly the background to the development and synthesis of DPP, then, secondly, he would describe its properties and uses. There are very many coloured compounds available; of these about 50% are Azo derivatives, 25% Phthalo and the remaining 25% all the other Chromofors. DPP (Di-Keto Pyrrolo-Pyrrole) is a new Chromofor - its structure has been known since 1974 but no route for commercial production had been found. Initially, about 2 kg of reactants were needed to produce 1 gm of the pigment. Majority of the work carried out was to find a better route for the production of the pigment and after many attempts, over quite a period of time, the current process was established. The pigment presently available is a red, but yellow to violet shades are possible by substitution in the Benzene Ring.

Going on to the second part of his talk, Dr Iqbal outlined the properties of DPP pigments. These have high heat stability, some up to 500°C, good opacity similar to that of Molybdate Chromes and good weathering properties, better than that of the Perylene Reds. The colour saturation was similar to that of the Azo pigments. Depending on the crystal size and structure, the pigments varied from transparent to opaque. They can be used in a wide variety of finishes from Automotive to heat resisting, with none of the toxicity problems associated with Lead or Cadmium pigments. The Automotive industry was already showing great interest in this range of pigments for use in the very near future as bright colours with excellent durability could be obtained.

After a lively question time the meeting closed with a vote of thanks proposed by Mr D Kimber.

B. E. Myatt



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ICCATCI Meeting at FSCT Paint Show



ICCATCI meeting at FSCT Paint Show, Dallas. Standing (l to r): Executive Secretary, Robert F. Ziegler (FSCT); Treasurer, James E. Geiger (FSCT); Past-President, William Mirick (FSCT); President-Elect Deryk R. Pawsey (FSCT); Past-President, A. Clarke Boyce (FSCT); Past-President, Joseph A. Bauer (FSCT); Graham Fowkes (FSCT Liaison Committee); Past-President, Terry F. Johnson (FSCT); Annik Chauvel, Editor of *Double Liaison* (FATIPEC); and Executive Vice-President, Frank J. Borrelle (FSCT). Seated: Past-President, Jacques Roire (FATIPEC); President, Yoshiaki Oyabu (JSCM); President, John Bourne (OCCA); President, Lothar Dulog (FATIPEC); President, Carlos E. Dorris (FSCT); Past-President, Ted Saultry (OCCA); Delegate, Anders Kjellin (FSPVT); and Past-President, Amleto Poluzzi (FATIPEC).

A luncheon meeting for visiting members of the International Committee to Coordinate Activities of Technical Groups in the Coatings Industry (ICCATCI) sponsored each year by the Federation of Societies for Coatings Technology (FSCT) was held at its Annual Meeting and Paint Industries' Show (October 87).

ICCATCI is composed of: FSCT; Federation of Associations of Technicians in the Paint, Varnish, Lacquer and Printing Ink Industries of Continental Europe (FATIPEC); Oil and Colour Chemists' Association (OCCA); Oil and Colour Chemists' Association Australia (OCCA); Japan Society of Colour Material (JSCM); and Federation of Scandinavian Paint and Varnish Technicians (FSPVT).

The next meeting of ICCATCI will be held during the FATIPEC Congress in Aachen, Germany, September 18-24, 1988.



“Profitable Research & Development”

The preparations for the 1989 Conference at Chester (Roman name Deva) are progressing well. We already have some firm promises of papers – the subject we have chosen seems to be a popular one. Let us hope that this will prove to be the case with prospective delegates. John Taylor will be issuing widely a leaflet “Call for Papers” shortly and this will include further details (look in this Journal also for this).

Where is Chester?

Some people do not know the answer to the above question. In fact Chester is



Eastgate Street, Chester

readily accessible from all parts of the UK. The following facts may, therefore, be of some encouragement.

Chester is

1. on the UK motorway complex
2. 35 minutes (by motorway M56) from Manchester airport

Travelling times are approximately as follows:

“T” by Train, “R” by Road

London	3 hrs (T)	3½ hrs (R)
Leeds	2 hrs 10 mins (T)	1½ hrs (R)
Glasgow	4¼ hrs (T)*	2¾ hrs (R)
Bristol	3¾ hrs (T)*	2¼ hrs (R)

*includes changing trains

A. C. Jolly

London Section's 50th Anniversary Celebrations

As part of its 50th Anniversary celebrations, the London Section hosted an Anniversary Dinner for its former Committee members from 1970 and other distinguished guests. Nineteen guests attended the Dinner at the Naval Club on 13 November and enjoyed an excellent meal and the companionship of former colleagues. Current Chairman, Barry Canterford, invited guests to reminisce on the five decades of the London Section to which David Roe, Herbert C. Worsdall, Robert Hamblin and Derek Eddowes were happy to respond.

The following current and former Committee members were present:

Former Committee Members

Mr P. J. Barnes
Mr D. Cordery
Mr D. Eddowes
Dr H. Hamburg
Mr A. Healey
Mr A. Hopwood
Mr R. Munn
Mr A. Newbould
Mr D. Roe
Mr A. Soane
Mr H. C. Worsdall

Current Committee Members

Mr K. Arbuckle
Dr T. Banfield
Mr D. Bannington
Mr B. Canterford
Mr B. F. Gilliam
Mr R. Gough
Mr J. Hurst
Mr L. Jennings
Mr J. Locke
Mr P. Neal
Mr P. Thukral
Mr J. Tooth



Herbert Worsdall



L to R: Ron Gough, Tom Banfield and Ken Arbuckle.

OCCA welcomes Frank Craik

Frank Craik has recently been appointed Advertisement Manager of JOCCA. Frank joins OCCA with 35 years' experience in technical advertising sales and administration. He has 17 years with commercial publishing houses which finally saw him as Business Manager of the processing journals group of Morgan-Grampian. He started an independent consultancy and representation in 1971 and has been employed by the Institution of Chemical Engineers and the Institution of Plant Engineers. Until September 1987, he was advertisement manager of Chartered Institute of Public Finance and Accountancy. He is a Fellow of the Institute of Sales and Marketing Management and Honorary Companion Member of the Institution of Chemical Engineers.



David Roe



L to R: Derek Eddowes and Herman Hamburg.



FROM THE GENERAL SECRETARY

JOCCA into the nineties

Wednesday 9 December was the occasion for an historic meeting at Priory House when, after an interlude of over 10 years, the Association's Publications Committee met to discuss the future of the Journal.

The meeting, under the Chairmanship of Don Newton, considered proposals to redesign and relaunch the Journal with the objectives to improve the presentation of industrial news and events, to provide a better service to OCCA members and to attract more advertisers. All these objectives were to be attained without any

reduction in the quality of technical articles for which JOCCA has an international reputation.

The redesigned Journal will have a crisper image – with more colour, including the front cover, and a more attractive layout for the technical articles. The overall impression will be a modern presentation of quality technical articles, coupled with up to date information on the industry.

The meeting also considered and approved a change in the management of the Journal with the creation of a small Management group to oversee the day to day production of the Journal and to work in close liaison with Priory House staff. The role of the Publications Committee would continue to be the selection and commission of technical articles, the life blood of the Journal, and Sections are urged to submit more papers for publication.

The meeting also learnt of proposals, considered by the Finance Committee, to use the services of an Advertising and Marketing Consultant to promote the Journal and seek additional advertising. Frank Dixon Craik will be spearheading

this initiative and will soon become a familiar name as he promotes the Journal on the Association's behalf. Frank will succeed, given the support of members and their companies – if you are in a position to place advertisements or to influence colleagues, Frank Craik will be wanting to hear from you.

Some members may question why it is necessary to adopt such a commercial attitude towards the activities of a learned society. The answer is simple – the Association needs a sound financial footing in order to provide the range of services expected by members. By improving the financial viability of the Association through commercial activities these services can be provided without additional recourse to members' subscriptions. Surely it's worth supporting?

formation of what is now the Zimbabwean Section.

Dirk obtained his BSc degree at the University College of the Orange Free State and his MSc at Rhodes University. He joined Buffalo Paints in Durban as Chief Chemist in 1947 and later became Technical Director. In 1969 he moved to the Transvaal and joined the South African Bureau of Standards in 1970. He became Head of the Paints and Sealants Division in 1973 and was appointed Director of Chemical Technology in 1979, a position he held until his retirement in 1985. He took an active interest in the International Organisation for Standardisation and served on ISO Technical Committees – ISO/TC35 – Paints and Varnishes.

Dirk Pienaar was a champion golfer, both at university and since. On his 60th birthday he was playing to a handicap of four. He was a reader for 'Tape Aids for the Blind' and his voice will be heard on such recordings for some years to come.

Dirk was most enthusiastic in anything he tackled and he strove for perfection. At the same time he had a very genuine interest in his fellow man. Kurt Engelbert, who was Secretary/Treasurer of the South African Section Committee at the time that Dirk was Chairman, writes: "I found him extremely approachable, human and courteous which did not stop him from insisting on correct and precise procedures, an experience which I found very valuable in my professional and personal life, in sport and association administration".

We convey our deepest sympathy to his widow, Betty, and to his daughter, son, daughter-in-law, son-in-law and grandchildren by whom he is survived. We also assure them of the best wishes of OCCA in facing the future.

Manchester Section

Quiz Evening

A very successful and enjoyable evening was held at the Silver Birch, Birchwood Centre, Nr. Warrington, on the 21 September 1987.

This informal event, which is sure to become annual, was attended by 58 people forming teams of four and five from companies including Crown Paints, Johnstones Paints, Usher-Walker, Coates, Q.C. Colours, I.C.I., Ciba Geigy and Foscolour. A strong challenge was also provided from the West Riding Section.

Following two rounds of questions, with a buffet provided at the mid point, a tie breaker was necessary between Usher-Walker, Ciba Geigy and the Coates 'B' team with Coates 'B' becoming the recipients of the Quiz Evening trophy and the first prize of bottles of wine kindly donated by Steely Minerals.

T. Wright

OCCA ties at £4.25 each are now available from the Association's offices, with a single Association Insignia on either a blue or maroon background.

Obituary

D. J. Pienaar

P. A. J. Gate writes:

Following a heart attack, Dirk Pienaar died in Pretoria on 9 October 1987 at the age of 64. This was announced at a meeting of the Transvaal Section held on 20 October 1987. As a mark of respect to him, the announcement was followed by a short period of silence.

Dirk was an ardent supporter of OCCA and one of the founder members of the South African Section. He attended the founding meeting in Durban on 21 January 1949. In 1956 he became Chairman of the South African Section, and was Chairman of the Transvaal Section in 1974-76. He was one of the first members to become a Fellow in the Professional Grades and served on the South African Professional Grades Committee for many years. He actively promoted the professional grade qualifications within the paint industry and related government organisations. He served on the Transvaal Section Committee and was the South African Vice President on Council from 1976 to 1978. In 1976 he headed a team in organising the first OCCA symposium to be held in Port Elizabeth in an effort to start a branch in that city. This was successfully accomplished. He also, whilst Vice President, took an active part in the

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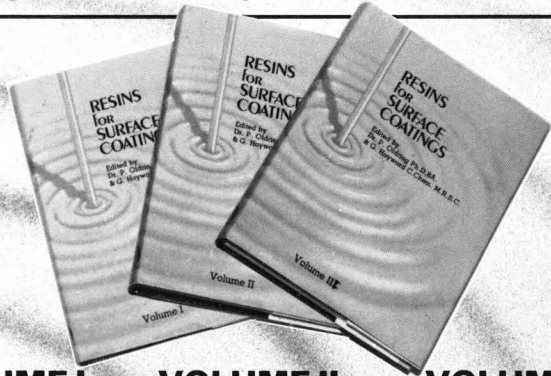
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