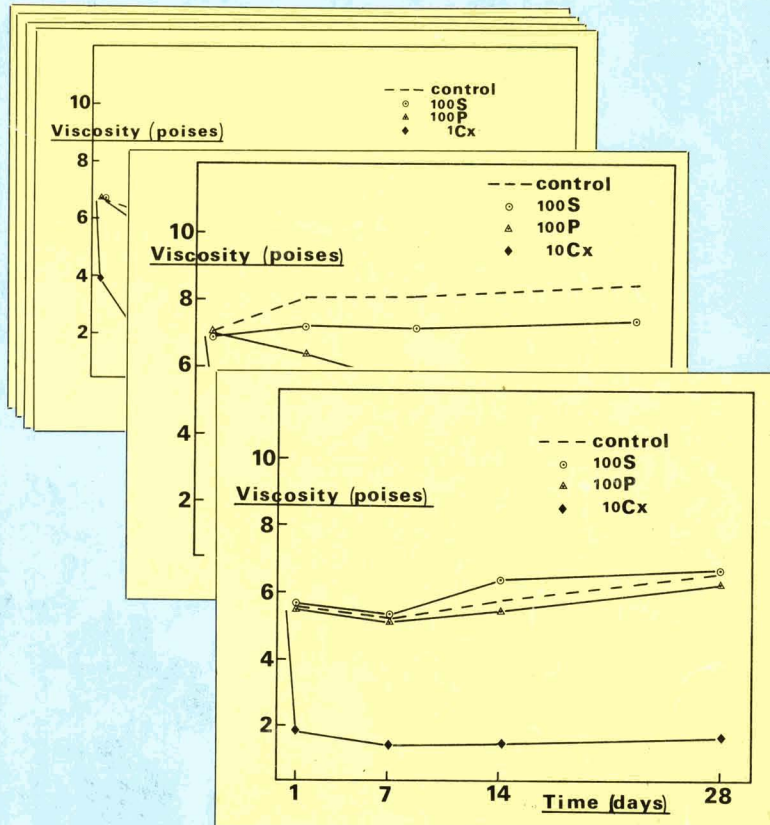




JOCCA

LIQUEFACTION OF CELLULOSIC PAINT THICKENERS

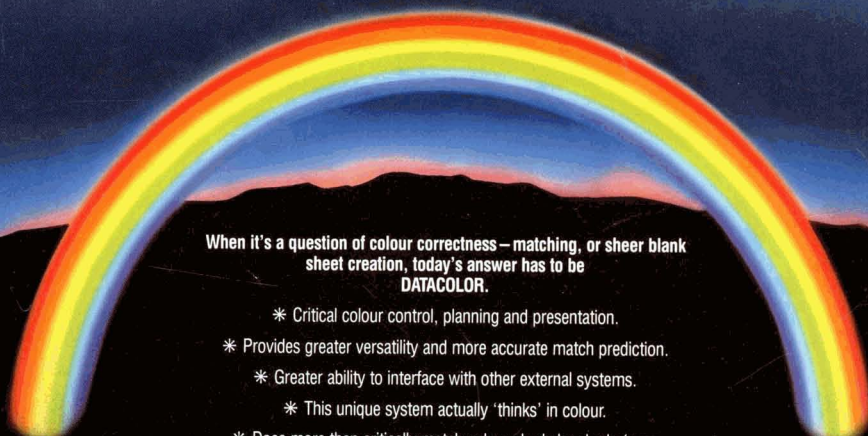


ALSO IN THIS ISSUE:

- Modifying epoxy resins using liquid polysulphides
- New propylene glycol ethers for waterborne coatings
- Anticorrosive primers free of lead and chromate
- Reviews

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OIL AND COLOUR CHEMIST'S ASSOCIATION

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

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FEBRUARY

i

Donald. S Newton, AMCT, CGIA, FICorrST, FTSC (1920-1988)

Mr R. H. Hamblin writes:

I was deeply saddened to learn of the death of Don Newton on 19 January. His contribution to the Association, both at Section level and as an Association Honorary Officer, was outstanding and had a considerable bearing on the prestige which the Association now enjoys, particularly in the world-wide recognition of the quality of its publications.

Not only did he serve as Hon Secretary of the Association from 1969 to 1974 but he was also twice Hon Editor (1962-65) and again from 1980 until his death; he was a Vice President from 1977 to 1979. A staunch supporter of the Bristol Section, he was twice elected its Chairman (1967-69) and again from the 1987 AGM until his death.

He will be greatly missed by his many friends, both in the Association and in the much wider circle of authors, who contributed under his guidance to the *Journal*, the monographs and other Association publications. He took immense trouble and justifiable pride in the *Journal*, with its wide circulation and the regard in which it is held in industrial and academic spheres. He encouraged many young authors with his comments and suggestions and was always ready to assist with publication matters not normally associated with the editorial function. His work in connection with Conference papers and help with the technical sessions, made many friends for the Association among the overseas lecturers and his keenness in pursuing authors who did not meet deadlines was an inspiration to the permanent staff. He was a good friend and mentor to several Assistant Editors of the Association, being a tower of strength and remaining unruffled in the problems which inevitably arise in publishing a monthly *Journal*.

He represented the Association on numerous bodies and, in so doing, advanced the Association's reputation. He had an unflinching interest in all matters relating to the Association and its well-being.

His contribution to the many Association Committees, on which he sat, were constantly valued by the Chairmen of those Committees and were always aimed at furthering the aims of the Association.

To his widow, Joyce, and his family the Association extends its condolences and thanks for their support to Don, which enabled him to play such an important part in the development of the Association.

Mr C. Pacey-Day writes:

Don Newton will be particularly missed by all the staff at Priory House. Don, in his capacity as Honorary Editor, was a frequent visitor to the offices and these visits were always appreciated by the staff who worked closely with him in the production of the *Journal*.

The last time the staff met Don was on the occasion of the Christmas celebrations when he was pleased to join

with us for lunch, during which he was the lively centre of conversation and added very much to the occasion.

The Association has lost a good friend.

Dr P. J. Fyne writes:

I was deeply saddened to learn of the sudden death of Don Newton, our Hon. Editor.

For many years Don had meticulously checked and corrected authors' manuscripts enabling sound technical papers to be published. He was always available to answer any technical queries that arose and he helped to smoothly steer the *Journal* to completion each month.

He will be greatly missed by us all at Priory House.

Mr G. W. Fowkes writes:

It is unusual to be able to apply the word "individual" to a person in both meanings; however, such is the case with Don Newton.

Here was a man known to many, yet not so well known by many. The only son of teacher parents, Don could adopt the role of philosophical academic without fear, bringing conversation to life or a drifting discussion back to relevant facts. Quick to see humour in many situations and occurrences but never flippant.

His activities with OCCA are detailed elsewhere and he will be sadly missed by the Bristol Section.

Residence in this area was brought about by his joining ISC Alloys to work on corrosion generally and zinc dust in particular, a time spent in the fruitful development of his family (five children) and his career (internationally known on Zinc Dust).

Choosing to remain in this area, he took early retirement on the ISC move to Bloxwich, in the Midlands, where he had completed his education and gained industry experience. Here, too, he met and married Joyce and is also fondly remembered by past colleagues and friends.

Don Newton had one other great interest – trains; using them for all his business travel throughout the world and modelling them in such time as was left.

Here again, his individualism was seen in devotion to his childhood G.N. & L.N.E.R. in the very heart of Brunel and G.W.R. land.

Last year, Don started his second term as Section Chairman as it was assumed that he would last almost for ever, enjoying good health until six months ago.

The immediate loss of Don Newton is known and his memorial will be the void at those sometimes quiet times when an individual and, perhaps, different point of view will be missing.

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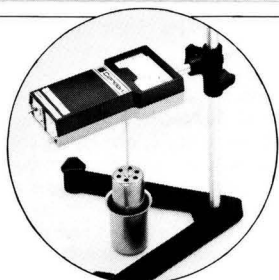
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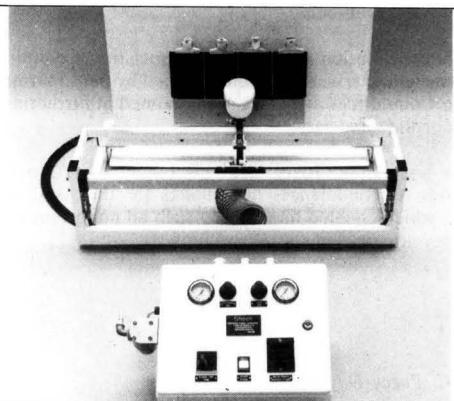
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Further US acquisition for Burmah

Burmah Speciality Chemicals Ltd has acquired the assets of Paragon Wax Refining Co of San Francisco. Paragon is the largest independent blender and merchant of petroleum wax on the West Coast of the USA. Dr Brian Ridgewell, chief executive of the Coatings Division of Burmah Speciality Chemicals said, "Paragon Wax is an important addition to our US wax business, which brings us new technology and good manufacturing and warehousing facilities in California. It will be operated as a division of our highly successful National Wax Company located in Chicago, with Fred Yaley Jnr, the former President of Paragon, retained as a consultant". Apart from National Wax, the Division consists of Yates Manufacturing in Chicago, Dussek Campbell companies based worldwide and Indtech in India.

Engelhard speciality chemicals acquisition

Engelhard Corporation and KaiserTech Ltd today jointly announced an agreement in principle for Engelhard to purchase the speciality chemical businesses of the Harshaw/Filtrol Partnership from KaiserTech Ltd and its subsidiary, Kaiser Aluminium & Chemical Corporation for \$264 million in cash.

The Harshaw/Filtrol Partnership consists of a number of speciality businesses, including catalysts, speciality minerals; colour pigments; compounds and other chemical for metal finishing; and crystal and electronic products.

Manders streamlines liquid industrial paints

The latest phase in the rationalization of production by Manders Industrial Division has now been completed with the concentration of all liquid coatings manufacture at its factory at Ruabon, North Wales.

This follows the concentration of all powder coatings production at the group's factory at Tyseley, Birmingham, and has involved the transfer of Tyseley's liquid coatings manufacture to Ruabon, freeing Tyseley for a big powder coatings modernisation programme.

New investment at Ruabon has significantly boosted capacity including the installation of a bank of eight 2,000-litre mixers and the setting up of a wood finishes facility with a high-speed mixer serving four pots feeding two sand mills, which in turn feed four 4,000-litre mixers. Other new equipment includes a metering system throughout the plant, pan-washing machinery and extensions to warehousing capacity, the tank farm and the barrel bund. An IBM 36 has replaced the previous office computer and the factory has a computer colour-matching system.

The management team at Ruabon has been strengthened by the transfer of two senior staff from Tyseley. Geoff Berry



Graham Walker, Site Director, Hoechst UK Ltd, Stainland, explaining proposed plans for the development of the Stainland site.

In 1972 Hoechst UK bought the Brookroyd Mills site and when in 1976 the John Shaw Company ceased operation, a long term plan was conceived to gradually replace the old textile buildings with new modern and more efficient manufacturing plant landscaped into the countryside. The first new plants for surfactants and pigment dispersions were installed in 1978 and in 1981 new purpose built offices and laboratories were opened. Over the last four years 10,000 ft² of additional covered warehousing has been constructed and a new Remafin plastics coloration plant installed.

Since the John Shaw closure Hoechst UK has invested almost £4.5 million in the Stainland site and employment has increased from almost nil to just over 200. In order to further expand and progress Hoechst UK now propose to replace the remaining part of the old mill with additional modern warehouses and production units at a further cost in excess of £3 million. Planning applications have recently been submitted to Calderdale MBC.

The plans for the next three years also include additional commercial offices, expansion of facilities within the surfactants plant, processing units for products for the metallurgical industry and a new site roadway system giving easier access to the new warehousing. Further expansion plans beyond 1990 are also envisaged as and when the business climate and production logic are able to support them.

becomes technical co-ordinator and SPC facilitator for the Division, and John Carter becomes liquid paint production manager.

The production team at Ruabon is led by a newly appointed production director, Alan Playford, whose qualifications and experience over 20 years will further strengthen management at the Ruabon site. At Tyseley, the recruitment of David Ankkret as director powder coatings provides specialist experience and qualification in the field of powder coatings, supported by Peter Williams as works manager, with David Jones as technical manager.

Reg Davis, managing director of Manders Industrial Division, says: "The rationalization of our manufacturing facility for liquid paint at Ruabon is now completed and new production equipment for our powder unit at Tyseley is being installed. Management at all levels has been strengthened by recruitment, as well

as in-house development, and we are now well set to provide improved standard of quality and service to our customers into the next decade and beyond".

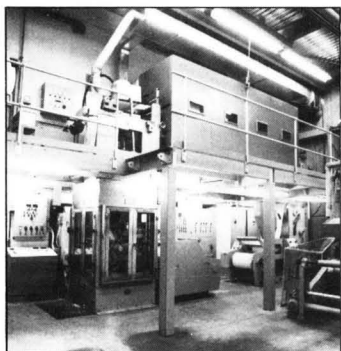
New pilot coating facility

A new pilot coating service is available to prospective customers by Hays Technology Systems (previously known as T. H. Dixon).

The facility is offered in conjunction with Spooner Industries Ltd and is located at the Spooner Ilkley test centre in West Yorkshire. The range of equipment offered for trial purposes is thought to be the most comprehensive pilot facility at a machinery manufacturer anywhere in Europe.

R & D Line No 7 was initially installed in 1983. This year it has been upgraded, with the most important addition being a Hays/Dixon 1020 coater. Together with a Spooner-TEC air floatation dryer and necessary ancillary equipment it provides a

products and equipment



Hays pilot coating facility.

complete coating/lamination line handling web widths up to 0.5 metres. Customers can evaluate products or processes using their own stock under various coating application methods and drying conditions.

The line is capable of three roll reverse, two roll reverse, gravure, reverse and direct, offset gravure coating processes and it can also be used for direct slot coating. Test speeds, depending on the process, are up to 1,000 metres/minute.

DSM starts negotiations on take-over of Freeman Chemicals

The chemical group DSM (Heerlen, the Netherlands) and the building construction group H. H. Robertsen (Pittsburgh, USA) have begun talks aimed at the signing of a letter of intent formalizing an agreement to enter into negotiations on the take-over Freeman Chemicals Ltd by DSM.

Freeman Chemicals Ltd operates in the field of resins, sheet moulding compounds and bulk moulding compounds, as well as distribution. The company is based at Ellesmere, UK, and has production facilities in various places, including Eastwood and Deeside. Freeman Chemicals Ltd has annual sales of approximately 70 million Dutch guilders and employs 230 people.

If the negotiations result in take-over, Freeman Chemicals Ltd will be taken up into DSM's resins division, DSM Resins. DSM is interested in Freeman Chemicals Ltd because in terms of products and marketing the company links up well with the activities and the strategy of DSM Resins.

Longer life guaranteed for Valcote paints

Valcote polyester liquid paint, from the Industrial Coatings Division of BASF Coatings + Inks, is now available with a ten-year guarantee when it is used as an architectural finish on aluminium. The guarantee applies to gloss, semi-gloss and matt finishes. BASF Coatings + Inks believes it is the first manufacturer to issue such a guarantee for polyester liquid coatings. Subject to Valcote being applied and stoved by an approved coater in accordance with the product's recommended procedures, BASF guarantees that the product will meet the specifications laid down by BS 4842:1984 'Liquid Organic Coatings for Application to Aluminium Extrusions and Preformed Sections for External Architectural Purposes', and will continue to do so for a period of ten years.

Reader Enquiry Service No. 20

New extender

Ultracarb is a new extender from Microfine Minerals based on a unique mineral deposit of partially hydrated magnesium calcium carbonate. The material occurs as ultrafine platy particles that are exceptionally white and pure. After processing, Ultracarb has many properties normally associated with synthetic or modified extenders such as precipitated calcium carbonate, alumina tri-hydrate, light magnesium carbonate and calcined china clay.

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New colloidal particle size and zeta potential system from Malvern



ZetaSizer 3

Malvern Instruments are to introduce a completely new laser system for characterizing colloidal particles. The ZetaSizer 3 will be the first instrument to combine full particle size distribution analysis with zeta potential determination and automatic sample handling. Expected to be available in Spring 1988, the ZetaSizer 3 will be shown at the 1988 Pittsburgh Conference in New Orleans. Zeta potential – the electrical potential close to a particle's surface – and particle size are important parameters in determining behaviour and properties of colloidal materials. Among the main features of the new Malvern ZetaSizer 3 are: Modularity – the basic system will be for particle sizing in the range 0.003 to 3 µm; to this can be added zeta potential and automatic sample handling modules. Variable angle particle sizing – PCS (photon correlation spectroscopy) light scattering measurements at angles from zero to 145° give full, model independent size distribution determinations. Completely new software programmes have been developed. Other features include: electrophoretic mobility, automatic sample handling, extended data presentation and automatic cell positioning.

Reader Enquiry Service No. 21

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SYMPOSIUM

As an associated event the Paint Research Association will be holding a symposium on "Towards better Industrial Finishing" on 13-14 June, 1988, Cairn Hotel, Harrogate. For further information contact
Dip Dasgupta at PRA on 01-977 4427,
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Surface treated grades of Ultracarb are available to modify dispersion, rheology, reinforcement, flow properties and wettability. Stearate coated grades are most widely used but a variety of other surface treatments give enhanced benefit in particular applications.

Reader Enquiry Service No. 22

Unusual wood finish

An unusual finish for use on medium density fibre board, or any solid timber, has been introduced by *Sonneborn & Rieck*. *Jaxalac VTI 93:479* is a two pack finish which provides the visual effect and feel of suede or velour. It is formulated on a two pack polyurethane using an aliphatic polyisocyanate as a curing agent. Tested by FIRA, the finish has excellent durability and resistance to marking. It complies with BS 3962 and is available in a wide range of colours. Application is by either conventional or automatic spray and is expected to be of special interest to furniture manufacturers.

Reader Enquiry Service No. 23

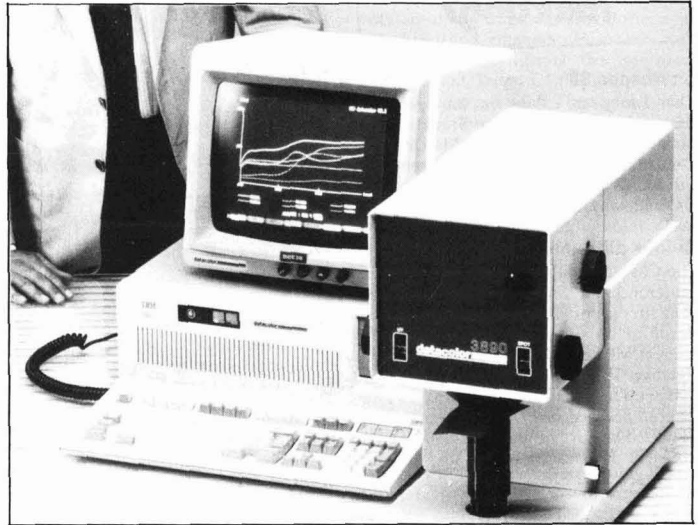
TV back panel coatings developed by HMG

One of the latest applications for *H. Marcel Guest's* specialist paint technology was for a component that is commonly found in the home, yet is scarcely noticed, despite forming part of a highly popular household appliance. The substrate in question was the back panel of a television set – or rather thousands of TV backs – for which the company has developed a two-part coating system.

Despite the synthetic appearance, these panels are actually fabricated from compressed and formed fibreboard – a superior relation of everyday cardboard – which possesses the appropriate technical properties and cost characteristics. In its natural state, however, this material has a tremendous propensity to absorb moisture, allowing it to soften, swell and deform. HMG's paint system was formulated to arrest that tendency and impart an appealing semi-sheen finish.

The system was designed by HMG's R & D team around certain production restrictions: it had to be applied easily and to dry rapidly, although the panels could

Industrial Colour Matching with Yorkshire Computer Services



The terminal which controls Yorkshire Computer Services colour matching system.

Yorkshire Computer Services Ltd has been appointed UK and Eire Distributors for *Datacolor* of Switzerland. *Datacolor* is one of the leading companies in the world for the manufacture of a wide range of Industrial Colour Matching Systems, their latest machines having improved efficiency in the colour match prediction areas, as well as providing much greater ability to interface with other external systems. A prime advantage of this system, over similar equipment currently available, is its ability to differentiate between the darker shades of black, dark navy and full blue. Paint and lacquer matches are produced instantly at the lowest possible cost. *Datacolor's* most recent customers include the paint manufacturers *Glasurit* and car manufacturers *BMW* and *Daimler Benz*.

In the automotive industry visual colour matching of metallic finish surfaces is considerably more difficult than normal paintwork. The very nature of metallic paintwork with its complexities of light refraction and reflection demands specialist, multi angle measuring technology as does the tendency to coat parts of the bodywork (inside and out) from plastic, in the same colour as the rest of the car. The *MMK III* Measuring Head from *Datacolor*, measures at three different angles – simultaneously. The *MMK III* is complemented by another hand held unit ideal for scanning hitherto inaccessible areas such as trim detail.

Both measuring heads are compatible with the colorimeter – a purpose designed multi-channel spectrophotometer (*MCS Unit*). *Datacolor* utilizes the *IBM PC/AT 03* and also in future different models of the *IBM PS 2*. A model 3890 Spectrophotometer machine using the *IBM PC/AT 03* is available for demonstration in *Yorkshire Computer Services Showrooms* at their *Greengates, Bradford* offices.

Yorkshire Computer Services Ltd has over 20 years experience in the design and implementation of computer systems with particular emphasis and strengths in the colour match prediction environment. *Y.C.S.* will give computer software and paint & pigment calibration services which will allow customers to get maximum payback quickly. *Y.C.S.* have the experienced management team to back these services. *Derek Harrison*, Director in charge of *Datacolor Services*; *Paul Emsley*, Technical Director; *Peter Cussons*, Marketing Director; *John Warden*, Colour Consultant and *John R. Spode*, Paint & Pigments.

Reader Enquiry Service No. 24

not be dipped, because of fibreboard's absorbent qualities, or subjected to intense curing temperatures, due to the material's intrinsic nature.

To overcome the absorbency problem, HMG developed a cross-linked, epoxy modified, sealing primer that provided a moisture resistant barrier coat; being

formulated for extremely rapid air drying, it could be spray applied without soaking excessively into the fibreboard. The top coat took the form of an acrylic modified lacquer, which imparts an attractive durable finish, with abrasion and fire resistant properties.

Reader Enquiry Service No. 25

meetings

Chemspec 88

This European exhibition for speciality chemicals will be held at Frankfurt on Main, W. Germany on 23-24 March 1988. For further information contact: Chemspec Europe 88 Exhibition, Queensway House, 2 Queensway, Redhill, Surrey, England.

Colour difference

The Colour Group (GB) will be holding a conference on Small Colour Difference Measurement at the University of Bradford on 6-7 April 1988. Of special interest will be the talk by Prof B. Rigg on Relative Tolerances in the CMC Colour difference formula in paints. The Conference fee to OCCA members is £36.00. For further information contact: Ms M. B. Halstead, 5 Farmlands, Enfield, Middlesex EN2 8JE.

The basics of colour

A short course of lectures and demonstrations on the basic principles of colour physics and measurements will be held at UMIST on 13-15 April 1988. Accommodation has been reserved at a nearby hotel. The course fee is £170.00 inclusive of lunches. For further details contact: Helen Cane, Continuing Education Office, Registrars Department, UMIST, PO Box 88, Manchester M60 1QD.

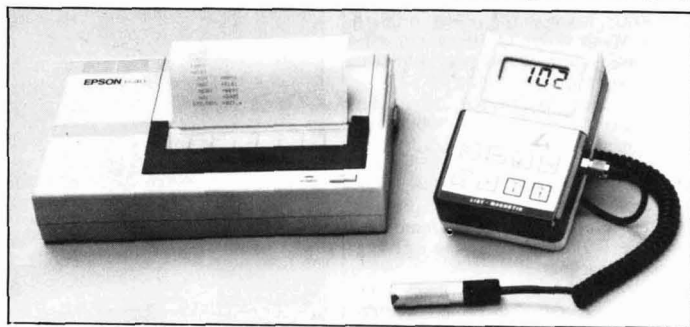
Particle Size Analysis 1988

The 6th International Conference on Particle Size and Surface Area Analysis will be held at The University of Surrey, Guildford on 19-20 April 1988. For further details contact: PSA 88, Department of Chemical & Process Engineering, University of Surrey, Guildford GU2 5XH.

people

Croda board appointment

Mr A. R. Fenney FCMA is appointed a director of Croda International plc with effect from 1 January 1988. Roy Fenney (45) has been with Croda since 1970 and held various senior financial positions in the Group, in the UK and overseas, until his appointment in 1985 as Managing Director of the Colloids Division which covers Croda's operations in gelatin, speciality proteins and food acidulants. In his new position he will be responsible for the Industrial Chemicals and Food business sectors, which as well as the Colloids Division includes Divisions engaged in technical oils.



Wymark's Duo-Check

The Duo-Check is the latest in the range of non-destructive, portable thickness gauges manufactured by List-Magnetik of West Germany and distributed in Britain by Wymark. The Duo-Check is a highly accurate and sophisticated instrument that incorporates the latest microprocessor technology and is capable of measuring by both magnetic-induction and eddy-current principles. It can be used with any of a range of probes to measure the thickness of paints, plastics, rubber, enamel, electroplating and so on, up to 2000 microns (= 2mm) on ferrous or non-ferrous substrates. The Duo-Check is a hand-held, battery-powered instrument with digital display, that can store in its memory up to 130 measurements. It can analyse these measurements for statistical purposes, and can display at any time such data as the number of readings, the maximum, minimum and mean values and the standard deviation.

New chief chemist at S&R

John Shea (32) has been appointed Chief Chemist of industrial paint and finishes manufacturer Sonneborn & Rieck Ltd, the company has its headquarters and laboratories at Hainault, Essex. Mr Shea joined the company in 1972 as a trainee laboratory technician. For the last seven years he has been section leader in charge of the company's wood finishes development team.

BP Chemicals appoints new works general manager at Grangemouth

Mr E. L. Ferguson, OBE, has been appointed Works General Manager of BP Chemicals Grangemouth factory, with effect from 1 December 1987. Ted Ferguson was previously Project Director (North West Europe), BP Petroleum Development. He received his OBE in 1982 for contributions to the construction industry, in particular Sullom Voe.

Smart coating thickness gauge

What makes the Duo-Check particularly useful for practical quality control purposes is its ability to print-out readings and statistics. A small printer is available as an optional extra, or the Duo-Check can be connected for this purpose to any computer with a suitable interface. Although it is feasible to print-out measurements as they are taken, a more attractive possibility is to take only the hand-held Duo-Check instrument into the factory for the purpose of taking measurements and then bring it back to the office whenever convenient to print out all the data for the quality control records. From the management point of view, this has the important advantage that coating thicknesses are always recorded.

Reader Enquiry Service No. 26

New manual weighing system

Darenth Ltd has launched a new microprocessor-based dual display electronic indicator, the model 1100, for manual weighing systems. It combines the advantages of an analogue display with the accuracy of a digital readout, both using light-emitting diodes. The indicator can be used in new installations and as a conversion for existing mechanical analogue indicators. It has the flexibility necessary to cater for high tare loads.

Reader Enquiry Service No. 27

New Chairman of Crosfield Group

Richard J. Duggan has been appointed Chairman of Crosfield, the Unilever speciality chemicals subsidiary. Mr Duggan has had a variety of technical roles within Unilever. He is currently Head of Unilever's Research Laboratory at Port Sunlight, one of the largest industrial research establishments in Europe. He joined Unilever in 1960 on graduating from Liverpool University with First Class Honours in Chemistry and the Leverhulme Prize.

Liquefaction of cellulosic paint thickeners. Part 1: Degradative effects of redox and enzyme contamination

W. R. Springle

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Summary

An investigation involving the addition of redox chemicals to a range of cellulose-ether solutions showed the latter could be thinned by as little as 10 ppm potassium persulphate and sodium metabisulphite. However, subsequent work using 12 emulsion paints thickened with various cellulose-ethers showed the paints were generally not substantially damaged in terms of either low shear or high shear viscosity by addition of 100 ppm of the redox chemicals (equivalent to an average contamination level in the latex emulsion of 383 ppm redox chemical). By contrast, addition of 1-10 ppm of a commercial cellulase enzyme caused substantial viscosity loss in the majority of test paints. Finally, a limited study is described, aimed at distinguishing between enzyme and redox contamination in emulsion paints, when it was observed that a "pattern" in viscosity response appeared to provide useful information.

Introduction: Refs. 1-8, 10-12, 14-20.

This paper and Part 2 to follow derive from a 1981-85 series of projects which were undertaken with three main objectives in mind, namely, (1) to determine the relative practical importance to the UK/European paint industry of redox and enzymatic thinning processes, (2) to investigate differentiation between these two types of contaminants in emulsion paints, and (3) to quantify in practical terms enzymatic thinning of emulsion paints. Items (1) and (2) are covered in Part 1 of the paper, item (3) in Part 2.

Water soluble cellulose-ethers are widely used by the surface coatings industry as thickening agents to control viscosity and gel structure of emulsion paints, with "degree of polymerisation" (DP) and "degree of substitution" (DS) being relevant parameters. An unintentional reduction in DP in an emulsion paint generally results in loss of structure and viscosity, possibly to the extent of gross deterioration involving phase separation, sedimentation of paint solids, and noticeable watery "sloppiness" in the container (usually termed thinning or liquefaction). The postulated "traditional" route to emulsion paint thinning assumes the presence in cellulose-ether solution, aqueous raw material, or emulsion paint of microbial cellulase

enzymes (viz. specific organic catalysts generated by microorganisms) which accelerate hydrolysis of the cellulosic polymer chain and produce molecules small enough to be absorbed and metabolised. Deriving from this assumption are the attitudes and basic precautionary/preventative measures which the industry takes to try to avoid and prevent such problems.

During 1976-80 a number of American papers proposed an alternative, non-enzymatic mechanism by which it was claimed that US latex paints would be more likely to thin in practice. The mechanism depended upon the degradative action on cellulose-ether molecules of redox chemicals utilised in the latex emulsion polymerisation process, and three related statements were made which potentially had far-reaching implications, namely; "... there is sufficient data to suggest that the presence of oxidants/reductants in raw materials may be the dominant cause of viscosity decrease in latex paints, not the presence of cellulase"; "it now seems evident that the interaction between oxidising and reducing agents is a major cause of viscosity decrease in latex paints"; and "The ASTM sub committee DO1.28 on biodeterioration just recently concluded that viscosity loss in latex paints was often chemical rather than biological".

In order to try to judge the practical significance of the above hypotheses to the UK/European paint industry, Paint RA was commissioned to undertake firstly a liquefaction study involving cellulose-ether solutions, and then a similar study involving European type emulsion paints. This work is described in Part 1 of the paper, together with a relevant though limited study on attempting to differentiate between oxidant and enzyme contaminants in emulsion paint by some simple technique.

Experimental: Refs. 6, 8, 9, 13, 15, 16, 20.

Cellulose-ether study

Based on advice from several major emulsion/emulsion paint manufacturers tests were carried out with 2% w/v solutions of Natrosol 250MR (medium viscosity hydroxy ethyl cellulose),

Methocel J5MS (hydroxy propyl methyl cellulose), and Cellosize QP52,000H (high molecular weight hydroxy ethyl cellulose). Solutions of the three cellulose-ethers were prepared such that pH conditions of 7 and 8.5 were obtained (the latter with both potassium carbonate/hydrochloric acid adjustment and ammonia adjustment), and series with and without the non-ionic wetting agent Triton CF10 (used at 0.1% w/v) were also prepared. The various series were inoculated with 10,50 and 100 ppm of the oxidants benzoyl peroxide and potassium persulphate and the reductants diethylene triamine and sodium metabisulphite, and with 25 ppm + 25 ppm mixtures of each oxidant/reductant. Test samples were incubated at either 40°C for 12 weeks or at 60°C for three weeks.

An aseptic technique was used throughout to try to ensure the absence of cellulolytic microorganisms/enzymes, solutions being filter sterilised or autoclaved at 110°C where appropriate. In order to monitor viscosity changes without exposing test samples to opportunist microbial invasion, duplicate comparison solutions were prepared containing 0.5%, 1.0%, 1.5% and 2.0% w/v concentrations for each cellulose-ether test series. These calibration solutions (representing each of the test variables) were incubated with their appropriate test series, each test sample being compared visually at the end of incubation in order to select a cellulose-ether concentration having an equivalent viscosity. The "substrate loss" reported in Tables 1 and 2 has been obtained by subtracting this final "equivalent concentration" from the known starting concentration of 2g/100ml.

Emulsion paint study

A range of 14 emulsion paints were used in the study, intended to represent the different types of interest at the time to the UK/European paint industry (see table 3). These comprised five products formulated and submitted by paint companies (viz. VV2, VV3, VV5, VEV2, and SA1 in Table 3), the remainder being prepared at Paint RA from commercial raw materials using a Morehouse Cowles International Dissolver or a Chemcol-Mischer High Speed Mixer (the latter for batches under 2kg).

For this work the significant experimental criterion was taken to be emulsion paint viscosity as it would be measured by a paint manufacturer. From various possible methods, the two instruments chosen for use were a low shear rate ICI Rotothinner Viscometer (spindle speed 562 rpm) able to distinguish relatively small changes in "gel" viscosity, and a high shear rate Cone and Plate Viscometer (shear rate 10 K.sec⁻¹) to determine base viscosity and reflect the "application" characteristics of an emulsion paint. Both instruments could be easily cleaned and alcohol sterilised between each measurement.

All paint test samples were distributed at

Table 1

Liquefaction of 2% w/v Natrosol 250 MR by redox chemicals

Redox Agent	Equivalent Substrate Loss (g/100 ml)*		
	pH7	pH8.5	+0.1% Triton CF10, pH8.5
Benzoyl Peroxide (B)			
10 ppm	0	0	0-0.5
50 ppm	0-0.5	0-0.5	0-0.5
100 ppm	0-0.5	0.5	0.5
Potassium Persulphate (P)			
10 ppm	0.5	0.5	0.5
50 ppm	1.5	1.5	1.5
100 ppm	1.5	1.5	1.5
Diethylene Triamine (D)			
10 ppm	0	0	0-0.5
50 ppm	0	0-0.5	0.5-1
100 ppm	0.5	not tested	not tested
Sodium Metabisulphite (S)			
10 ppm	0.5	0.5	0-0.5
50 ppm	0.5	0.5-1	0.5
100 ppm	0.5-1	1	0.5-1
Couples			
25 ppm (B) + 25 ppm (S)	0-0.5	0.5	not tested
25 ppm (P) + 25 ppm (S)	1	1	not tested

* Estimated by comparison of each test sample with its appropriate calibration series.

Table 2

Liquefaction of 2% w/v Methocel J5MS by redox chemicals

Redox Agent	Equivalent Substrate Loss (g/100 ml)*		
	pH7	pH8.5	+0.1% Triton CF10, pH8.5
Benzoyl Peroxide (B)			
10 ppm	0	0	0-0.5
50 ppm	0-0.5	0.5	0-0.5
100 ppm	0.5	0.5	0-0.5
Potassium Persulphate (P)			
10 ppm	0.5-1	0.5-1	0-0.5
50 ppm	1-1.5	1	1
100 ppm	1.5	1.5	1.5
Diethylene Triamine (D)			
10 ppm	0	0-0.5	0-0.5
50 ppm	0-0.5	0-0.5	0-0.5
100 ppm	0.5	0-0.5	0-0.5
Sodium Metabisulphite (S)			
10 ppm	0.5	0.5-1	0-0.5
50 ppm	1	0.5-1	0.5
100 ppm	1.5	1	0.5
Couples			
25 ppm (B) + 25 ppm (S)	0.5-1	0.5-1	not tested
25 ppm (P) + 25 ppm (S)	1	1	not tested

* Estimated by comparison of each test sample with its appropriate calibration series.

a constant volume of 150 ml into pre-sterilised 250 ml lacquered cans. Based on data from the earlier work potassium persulphate and sodium metabisulphite were chosen as oxidant and reductant for study, while a commercial cellulase (Cx) derived from the fungus *Trichoderma viride* was used as a source of enzyme (activity rated as 0.19 Enzyme Units/mg). A previous survey of European manufacturers had established that 50 ppm excess initiator was considered the maximum thought likely to be introduced into an emulsion paint, therefore, it was considered appropriate to carry out the work with redox contamination levels of 10, 50 and 100 ppm, although levels of 200 and 300 ppm were introduced into certain systems for comparison. It being relevant to consider what these levels represent in terms of latex emulsion contamination by redox chemicals, Table 3 gives appropriate data which indicate that 10, 50 and 100 ppm in paint require average emulsion contamination levels of 38.4, 191.6 and 383 ppm respectively for the paints used.

For the majority of paint test systems the oxidant, reductant or redox couple was pipetted into the completed paint as 2 ml filter sterilised solution. This enabled representative blank controls to be prepared by substituting sterile distilled water for the redox solution, and allowed for comparison between paints prepared in-house and those submitted ready formulated. However, to simulate the likely route of entry in practice, certain paint series were also prepared in small batches from redox contaminated latex emulsions of varying age. All paint samples were thoroughly stirred initially and then stored at 25°C and 65% rh, with viscosity being measured after one day, seven days, 14 days and 28 days. In addition, some systems were also incubated at 40°C and/or stored for a longer period with viscosity being measured after 56 and 84 days. The cellulase enzyme was added to test paints in a similar manner but at concentrations from 0.1 ppm (1.9×10^{-5} EU/g paint) to 100 ppm (1.9×10^{-2} EU/g paint), with viscosity measurements being made as before.

Oxidant/enzyme differentiation

Papers concerned with detection of oxidant (eg. potassium persulphate) in emulsion paint have been published, and reference has been made to detection of cellulase and of products from enzymatic hydrolysis of cellulose-ethers in emulsion paints. The work described here was concerned with differentiation in a fairly simple and practical manner between emulsion paint which was contaminated with either potassium persulphate or cellulase enzyme. Various approaches were examined, that which gave the most interesting results being a viscometric comparison of samples modified by addition of selected chemical agents. A series of experiments were carried out with

paints VV2, VV3 and VEV1, (see Table 3) to which had been added 50, 200 and 400 ppm of potassium persulphate or 10^{-5} EU/ml of cellulase enzyme. Separate addition was made to these paints of the enzyme inhibitors phenyl mercury acetate and zirconium chloride (to give 50, 500 and 1000 ppm metal) and of the reducing agents diethylene triamine and ascorbic acid (at 125, 200 and 500 ppm), with low shear viscosities being measured on an ICI Rotoviscometer (spindle speed 562 rpm) after 24 and 48 hours. Reference paints and water controls were similarly measured to give viscosity comparisons.

Results and Discussion: Refs. 8, 15, 16.

Cellulose-ether study

Representative results on Natrosol 250MR and Methocel J5MS are presented in Tables 1 and 2, the "substrate loss" having been estimated by comparison of each test sample with its appropriate calibration series as described earlier. Very similar results were obtained with Cellosize QP 52,000H.

The data showed that the cellulose-ether solutions could be thinned by all four redox chemicals under some conditions. However, liquefaction was generally much greater with potassium persulphate followed by sodium metabisulphite. Variation in experimental parameters such as pH, presence of ammonia, presence of a wetting agent, and sample incubation temperature, did not produce dramatically different results, while combinations of oxidising/reducing agents tended to give liquefaction levels intermediate between those of the individual components. In practical terms, it appeared that levels of potassium persulphate and sodium metabisulphite as low as 10 ppm could cause cellulose-ether solutions to degrade and lose viscosity—the question was then whether the same effect would occur when these redox chemicals were introduced into formulated emulsion paints.

Emulsion paint study

Representative results of changes in low shear viscosity with time for paints VEV2, A1, SA1, and VV5 are shown graphically in Figures 1-4 (the last having an extended incubation time). The reductions (as % of control paint viscosity) in both low shear and high shear viscosities which had been achieved by all paints containing 100 ppm redox agents after 28 days are shown in Table 4, together with similar results on enzyme contaminated paints (1 ppm or 10 ppm level). Equivalent data obtained on paints VV4, A1 and SA2 in which the redox agents had been added to the latex emulsions prior to paint manufacture are given in Table 5.

Considering 28 day effects on low shear "gel" viscosity of paints contaminated with

Table 3

Paint types studied and redox contamination levels calculated for emulsions

Paint Code*	Paint Type*	Emulsion Contamination Level (ppm)		
		for 10 ppm in paint	for 50 ppm in paint	for 100 ppm in paint
VV1	va/veova:HEC	44	220	440
VV2	va/veova:HEC	29	144	287
VV3	va/veova:HEC	29	144	287
VV4	va/veova:EHEC	44	220	440
VV5	va/veova:HPMC	formulation not fully disclosed		
VV6	va/veova:SCMC	44	220	440
VEV1	va/ethylene/vc:EHEC	41	204	408
VEV2	va/ethylene/vc:HPMC	formulation not fully disclosed		
VAC1	pva copolymer:MC	32	158	317
A1	acrylic:HEC	27	137	275
A2	acrylic:HEC	24	123	245
A3	acrylic:polyacrylate	23	114	227
SA1	styrene acrylate:HEC	formulation not fully disclosed		
SA2	styrene acrylate:HPMC	85	424	847
Average Emulsion Contaminated Level		38.4 ppm	191.6 ppm	383 ppm

* Formulations given in PRA Technical Report TR/3/83, HEC = hydroxy ethyl cellulose, EHEC = ethyl hydroxy ethyl cellulose, HPMC = hydroxy propyl methyl cellulose, SCMC = sodium carboxy methyl cellulose, MC = methyl cellulose.

Table 4

Percent reduction in low shear (LS) and high shear (HS) test paint viscosity after 28 days

Paint Code	Potassium Persulphate*		Sodium Metabisulphite*		P + S Couple*		Cellulase Enzyme*	
	LS(%)	HS(%)	LS(%)	HS(%)	LS(%)	HS(%)	LS(%)	HS(%)
	VV1	14	0	0	0	17	0	82
VV2	0	—	0	—	—	—	16	—
VV3	0	0	0	0	—	—	85	73
VV4	18	0	3	0	19	—	83	63
VV5	13	0	2	0	11	0	80	86
VV6	0	0	0	0	0	0	47	57
VEV1	9	0	2	0	11	0	58	50
VEV2	11	—	0	—	10	—	75	—
VAC1	6	12	6	12	6	0	65	50
A1	36	14	14	7	—	—	71	72
A2	0	0	0	0	0	0	11	29
A3	2	0	0	0	0	0	0	0
SA1	6	—	0	—	—	—	80	—
SA2	0	0	0	0	0	0	18	19

* Redox agents at 100 ppm, Cellulase enzyme mainly at 10 ppm (1 ppm in VV6).

Table 5

Prior addition of redox agents to emulsion: percent reduction in low shear test paint viscosity after 28 days

Paint System	Potassium Persulphate* (%)	Sodium Metabisulphite* (%)	P + S Couple* (%)
VV4			
Fresh emulsion	7	0	4
7 day emulsion	3	0	1
14 day emulsion	10	0	0
A1			
Fresh emulsion	30	4	18
7 day emulsion	20	8	3
14 day emulsion	7	0	2
SA2			
Fresh emulsion	0	0	0
7 day emulsion	0	0	0
14 day emulsion	0	0	0

* Redox concentration 100 ppm in completed paint

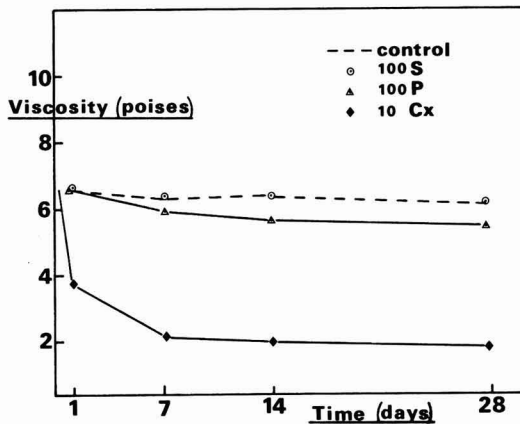


Figure 1. Low shear viscosity (poises) vs time (days) for Paint VEV2 contaminated with 100 ppm potassium persulphate (P), 100 ppm sodium metabisulphite (S), and 10 ppm cellulase enzyme (Cx).

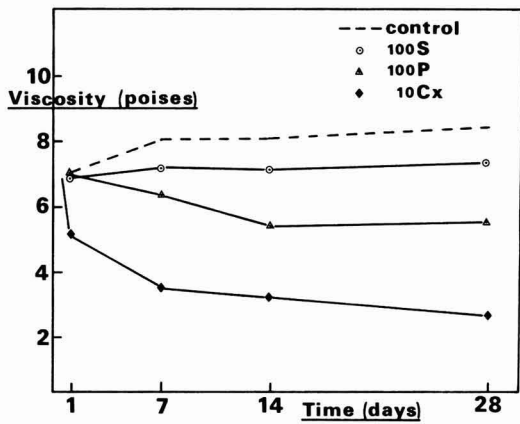


Figure 2. Low shear viscosity (poises) vs time (days) for Paint A1 contaminated with 100 ppm potassium persulphate (P), 100 ppm sodium metabisulphite (S), and 10 ppm cellulase enzyme (Cx).

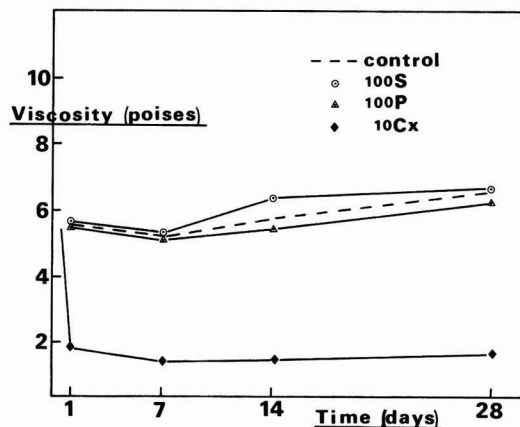


Figure 3. Low shear viscosity (poises) vs time (days) for Paint SA1 contaminated with 100 ppm potassium persulphate (P), 100 ppm sodium metabisulphite (S), and 10 ppm cellulase enzyme (Cx).

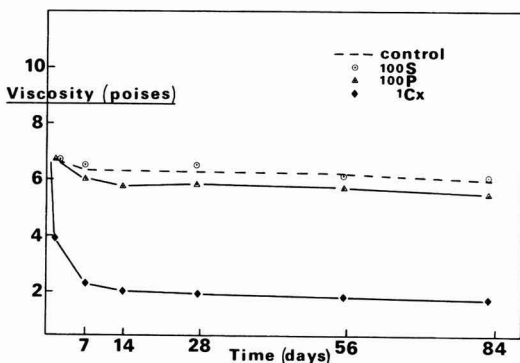


Figure 4. Low shear viscosity (poises) vs time (days) for Paint VV5 contaminated with 100 ppm potassium persulphate (P), 100 ppm sodium metabisulphite (S), and 10 ppm cellulase enzyme (Cx).

100 ppm redox chemicals (NB. this would require an average latex emulsion contamination level of 383 ppm, see Table 3), sodium metabisulphite appeared to show its only real action on paint A1 but the 14% "viscosity reduction" recorded was mainly due to the blank control having thickened slightly over the test period (see Figure 2). Potassium persulphate and the redox couple behaved somewhat similarly to each other, with a number of paints showing "viscosity reductions" of 2-19%, and paint A1 showing an apparent reduction of 36% with the former chemical (see Table 4). However, in most cases the actual viscosity reduction was less than one poise, and sometimes the % magnitude resulted more from an increase in control paint viscosity (with which each sample was compared) than from a loss of sample

viscosity. For example, although paint A1 with 100 ppm potassium persulphate addition was 3.1 poises below its control, the test paint was actually only 1.6 poises below the low shear viscosity at which it started (see Figure 2).

By comparison, 10 of the 14 paints contaminated with the commercial cellulase enzyme (Cx) showed apparent viscosity reductions of 47-85% with three others showing 11-18% reductions (see Table 4). Only paint A3 (which contained a polyacrylate rather than cellulosic thickener) was unaffected by the enzyme. The % reductions given represent low shear viscosities ranging from 1.25-6.8 poises below blank control values. Data are also presented in Table 4 on reductions in high shear "application" viscosity, which

show the generally minor impact of the redox chemical addition compared to the cellulase enzyme addition. In reality, while all the redox contaminated paints retained high shear viscosity between the acceptable commercial limits of 0.7-1.3 poises, nine of the cellulase contaminated paints fell below the 0.7 poise minimum with a number showing "gross deterioration" in the container (eg. phase separation, sloppiness, etc). Of other experimental parameters examined, extending the sample incubation period or increasing redox concentration to 300 ppm did not produce substantial changes in low shear viscosity (eg. see Figure 4), an incubation temperature of 40°C rather than 25°C appeared to mainly influence reaction rate rather than magnitude, and introducing redox chemicals into the latex emulsion

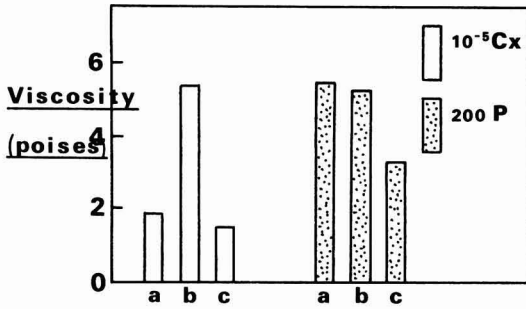


Figure 5. Viscosity response pattern (at 24 hours) of Paint VV2 contaminated with 10^{-5} Enzyme units/ml cellulase (Cx) or 200 ppm potassium persulphate (P), followed by addition of (a) water, (b) 0.1% ww Hg, or (c) 0.02% ww ascorbic acid.

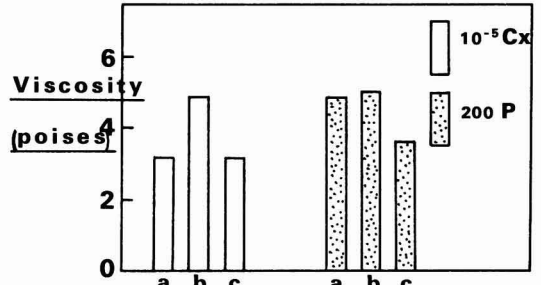


Figure 6. Viscosity response pattern (at 24 hours) of Paint VEV1 contaminated with 10^{-5} Enzyme units/ml cellulase (Cx) or 200 ppm potassium persulphate (P), followed by addition of (a) water, (b) 0.1% ww Hg, or (c) 0.02% ww ascorbic acid.

prior to paint manufacture did not increase effects (see Table 5).

Oxidant/enzyme differentiation

Figures 5 and 6 show the type of response "patterns" obtained when oxidant and cellulase enzyme contaminated paints were modified by addition of 0.1% ww mercury (as solubilised phenyl mercury acetate) or 0.02% ww ascorbic acid, and then low shear viscosity measured after 24 hours. The paint industry has "traditionally" used mercury compounds to combine wet-state antimicrobial action with enzyme inhibitory action and, from this starting point, was conceived the rather arbitrary idea of retarding or accelerating viscosity changes of a paint according to whether enzyme or oxidant contamination was present. The practical procedure evolved was to split a test paint into three equal samples of 150 ml, add respectively (1) water, (2) 0.1% ww Hg solution to the same volume, (3) 0.02% ww ascorbic acid solution to the same volume, and measure low shear viscosity after 24 and 48 hours. The "pattern" which tended to emerge (see Figures 5 and 6) was as follows:

uncontaminated paint – all three viscosities remained similar,

enzyme contaminated paint – the mercury treated sample gave a relatively higher viscosity,

oxidant contaminated paint – the ascorbic acid treated sample gave a relatively lower viscosity.

Obviously, these results were obtained with laboratory contaminated samples, and the usefulness of such an approach in practice remains to be verified. An essential prerequisite for interpretation of the type of data obtained would be that a paint manufacturer build up a library of viscosity response "patterns" for his normal products in order to distinguish the "anomalous" paint!

Conclusions

At a level as low as 10 ppm the redox

chemicals potassium persulphate and sodium metabisulphite were shown to cause degradation leading to viscosity loss in cellulose-ether solutions of the type used as paint thickeners.

In a study using 14 emulsion paints of UK/European type, it was shown that paints contaminated with 100 ppm of the same redox chemicals (equivalent to an average latex contamination of 383 ppm) were *not* substantially reduced in terms of low shear "gel" viscosity (except perhaps for an acrylic paint coded A1). Also, *none* of the paints fell below a commercially acceptable high shear "application" viscosity.

By comparison, the majority of paints were substantially thinned in terms of both low shear and high shear viscosity by the presence of 1-10 ppm commercial cellulase enzyme (an exception being a polyacrylate thickened paint), and only enzyme contaminated paints showed "gross deterioration" in the container.

A simple method was demonstrated, involving addition of chemical agents to paint, which gave a generally consistent viscosity response "pattern" according to whether enzyme or oxidant contamination was present. The tests were carried out on laboratory produced samples, and it is stressed that practical usefulness remains to be verified.

Acknowledgements

The author's thanks go to Paint RA for permission to publish this paper, and to Mrs A. L. Smith for her contribution to the early work on cellulose-ether solutions.

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The modern approach to modifying epoxy resins using liquid polysulphides: Part 1

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Abstract

This paper deals with some of the recent advances in polysulphide-epoxy technology which has overcome previous limitations and has led to a resurgence in the popularity of these systems. The performance benefits that can be obtained from the improved LP modified resins are discussed with particular reference to the coating and adhesive industries.

Liquid polysulphide polymers are usually associated with the manufacture of high performance sealants for the building, construction, insulated glass and transportation industries. Within these application areas the adhesion, flexibility, durability and chemical resistance of the polysulphides are well understood and documented over a period of 40 years' service. What is not so well known is that these beneficial properties can be supplied to other resins by combination with the polysulphide polymers. This paper will deal with one such combination, namely the polysulphide-epoxy resins and the recent technical developments which makes these systems attractive to the coating and adhesive industries.

Epoxy resin based compounds have many excellent properties including rapid curing at normal temperatures, good adhesion to most surfaces, toughness and chemical resistance to many dilute acids, alkalis and solvents. The incorporation of a polysulphide component into an epoxy resin leads to improvements in certain properties without adversely affecting the existing performance capabilities of the epoxy system. Polysulphide modification enhances both the uncured and cured properties of an epoxy resin. To understand these benefits and how they are obtained requires an examination of the

chemical and physical structure of the polysulphide polymer itself (see Figure 1). Chemically, the liquid polysulphides are polymers of bis-(ethylene oxy)methane containing disulphide linkages. The formal or ether linkages in the polymer backbone provide considerable mobility and therefore flexibility to the polymer. The liquid polysulphides are classified as elastomers and have glass transition temperatures in the range -55°C to -65°C . The saturated nature and the very high sulphur content of the polymer provides chemical resistance to a wide variety of organic solvents, dilute acids, alkalis and water. The absence of unsaturation and carbonyl structures also endows the polymer with good outdoor weathering and resistance to both ultra violet light and ozone. The polymer molecule is terminated with mercaptan groups which are capable of reacting with a variety of chemical species including the epoxide groups of an epoxy resin. The value of the molecular repeat unit "N" governs the molecular weight of the liquid polysulphide polymer.

Table 1 shows that the value of "N" can be controlled at the manufacturing stage to produce liquid polymers whose molecular weights range from 8,000 to 1,000. The higher molecular weight LP-31, LP-2 and LP-32 polymers are used primarily for the production of sealants. The lower molecular weight LP-3, LP-33 and the newly developed ZL-1400C polymer are used as elastomeric modifiers, particularly for epoxy coatings and adhesives. The choice of LP-3, LP-33 and ZL-1400C as modifiers for epoxy resins has been based on their molecular weight, viscosity, mercaptan

content, and functionality. Due to their low molecular weights LP-3, LP-33 and ZL-1400C have the lowest viscosities of the commercially available LPs. Viscosities of less than 2 Pascal seconds at 25°C make the polymers extremely easy to handle and incorporate into an epoxy resin. This low viscosity is particularly advantageous during cold weather conditions. Even at 4°C the polymers have viscosities which are considerably lower than those associated with standard unmodified bisphenol A (DGEBA) epoxy resins at 25°C . The excellent low temperature handling characteristics of LP-3, LP-33 and ZL-1400C are reflected in their Pour Point values which lie in the -23°C to -26°C range.

Table 1 illustrates two more important parameters, namely mercaptan content and branching. The liquid polysulphides with the highest mercaptan content are LP-3, ZL-1400C and LP-33. This makes them the most reactive polymers for the manufacture of a polysulphide modified epoxy resin. Branching is introduced into the polysulphide molecule by adding the trifunctional monomer, trichloropropane at the manufacturing stage. The production of LP-3 for example, requires the addition of two mole per cent of trichloropropane whereas LP-33 requires half a mole per cent. None of the liquid polysulphides are highly branched and with the exception of ZL-1400C have a functionality of just over two. ZL-1400C is a linear polymer with exactly two mercaptan groups per molecule. LP-3 and LP-33 have a minimum amount of molecular branching and contain 2.12 and 2.03 mercaptan groups per mole respectively. The linearity of ZL-1400C and the near linearity of LP-3 and LP-33 ensures that during co-reaction with an epoxy resin chain extension rather than cross-linking reactions predominate. This ensures maximum flexibility for the cured product.

Figure 2 illustrates the chemistry of the manufacture of an LP modified epoxy resin. Two moles of an epoxy resin co-react with one mole of the liquid polysulphide to form the cured LP-epoxy resin. The

Figure 1 Polysulphide Polymer

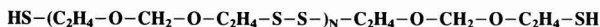


Table 1

Morton Thiokol Liquid Polysulphide Polymers

	LP-31	LP-2	LP-32	LP-3	LP-33	ZL-1400C
Average Molecular Weight	8000	4000	4000	1000	1000	1000
Repeat Unit "N" Value	42	23	23	6	6	6
Average Viscosity at 25°C (Pa.sec.)	110	47	47	1.2	1.7	1.2
Average Viscosity at 4°C (Pa.sec.)	740	380	380	9	16.5	9
Pour Point ($^{\circ}\text{C}$)	10	7	7	-26	-23	-26
Mercaptan Content (%)	1.0-1.5	1.5-2.0	1.5-2.0	5.9-7.7	5.0-6.5	5.9-7.7
Trichloropropane (Mole %)	0.5	2.0	0.5	2.0	0.5	0
Mercaptan Groups per mole	2.24	2.48	2.12	2.12	2.03	2.0

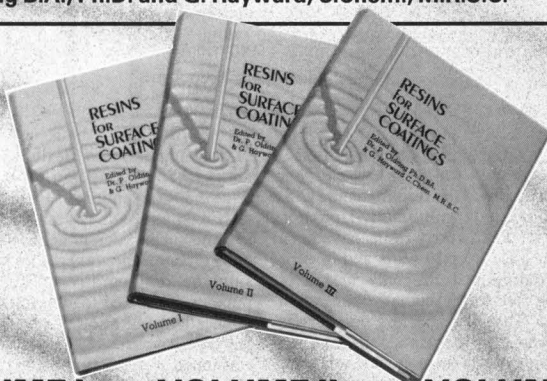
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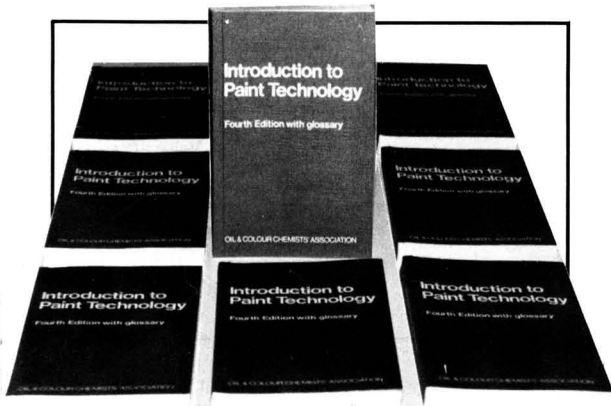
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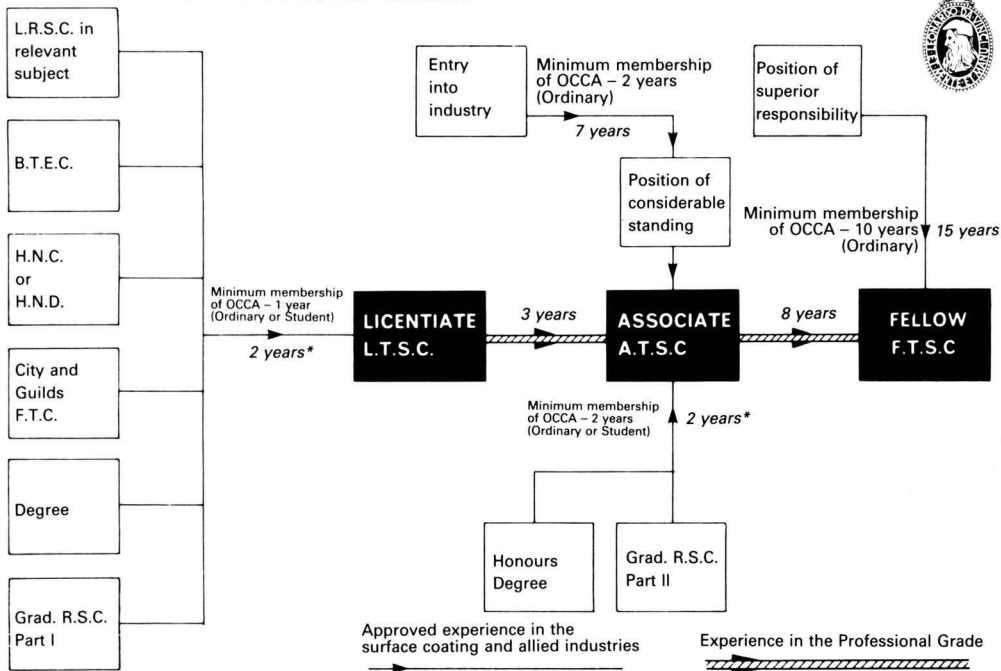


Table 2

Adhesion to steel: BS-3900 Part E6 cross cut test

Coating	Cross cut classification	
	Initial Value	Value after 500 hours Salt Spray
Conventional LP-Epoxy	0	0
Liquid Epoxy-LP Prepolymer	0	0
Liquid Epoxy-Polyamide	1	5
Liquid Epoxy-Polyamine	0	5
Solid Epoxy-LP Prepolymer	0	0
Solid Epoxy-Polyamide	0	0

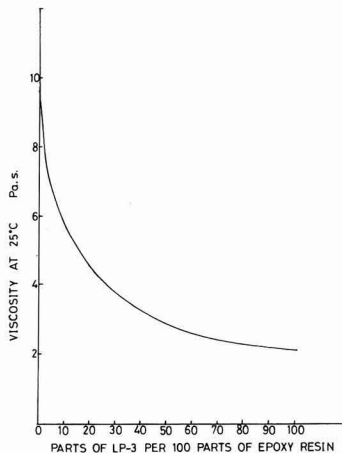


Figure 4. The addition of LP-3 to a DGEBA Epoxy Resin. Viscosity at 25°C.

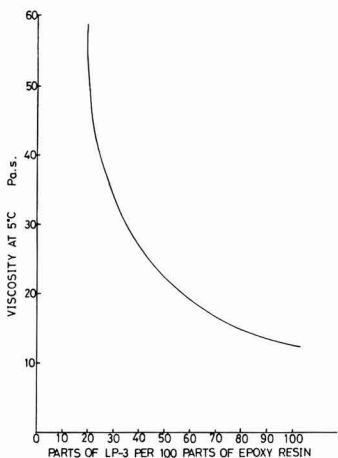


Figure 5. The addition of LP-3 to a DGEBA Epoxy Resin. Viscosity at 5°C.

of LP-3 at levels in excess of 25 parts to 100 parts of epoxy resin allows the mixing and pouring of LP-epoxy systems to be readily undertaken at this temperature. Even under these adverse conditions high solids or solventless systems can be readily applied.

Control of Cure Characteristics

LP addition can, depending on the level of LP used and on the reactivity of the curative, accelerate or retard the rate of cure of an epoxy-amine system.

Figure 6 shows that the incorporation of LP-3 into a DGEBA - Ancamine 1608 system leads to a shortening of the usable pot life of the mix. This effect is particularly evident for additions of up to

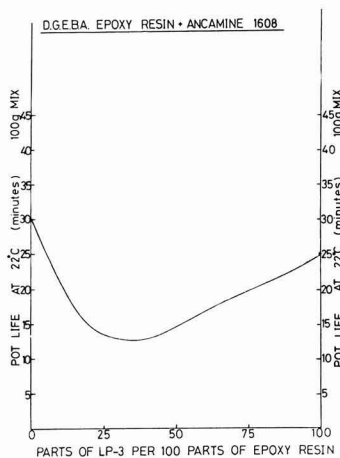


Figure 6. The effect of LP-3 level on Epoxy Resin Pot Life. (Acceleration of cure).

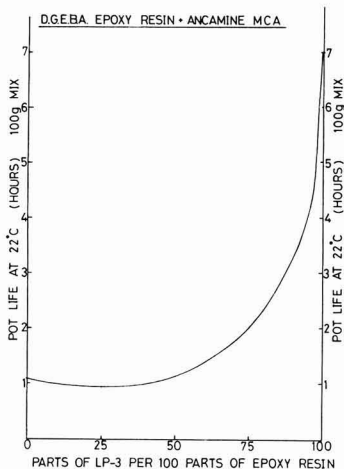


Figure 7. The effect of LP-3 level on Epoxy Resin Pot Life. (Retardation of cure).

50 pph of LP-3. Ancamine 1608 is a highly reactive aliphatic amine used in solventless and high solids coatings. Figure 7 shows that the less reactive DGEBA - Ancamine

MCA system's pot life is not affected by LP-3 incorporation until the level of addition exceeds 50 pph of epoxy resin. Above this level the pot life is extended dramatically. Ancamine MCA is a cycloaliphatic amine used in industrial flooring and wall coatings and in adhesives for bonding concrete, applications where an extended pot life is desirable. The rapid low temperature cure of epoxy resins can be facilitated by incorporation of a polysulphide in combination with a reactive primary amine. For example a DGEBA epoxy resin containing 50 pph of LP-3 and cured with Ancamine 1608 has a usable pot life of 20 minutes at 0°C. This is 10 minutes shorter than the 22°C pot life value of the unmodified epoxy resin. This acceleration of cure is produced without the use of a tertiary amine catalyst or an expensive polymercaptan curative, thereby ensuring no loss of flexibility in the cured product.

The cured properties benefited by LP modifications are:

Adhesion

Polysulphide modified epoxy resin systems not only retain, but through improved wetting properties, enhance the adhesive characteristics of epoxy coatings and adhesives.

There are numerous methods of assessing the adhesion of an organic coating to a substrate, the simplest being the cross cut test BS-3900 Part E6. Table 2 illustrates the cross cut adhesion values of a number of laboratory prepared epoxy coatings both before and after cold salt spray exposure. The coatings were formulated as 80% solids systems containing titanium dioxide at the Critical Pigment Volume Concentration as the only filler. The coatings were spray applied onto mild steel plates to a nominal dry film thickness of 150-200 microns. The plates had been shot blasted and left for 7 days at 21°C and 50% RH prior to coating. The steel plates were in poor surface condition at the time of coating. The conventional LP-epoxy coating is modified with 30 parts of LP-3 per hundred parts of liquid epoxy resin whereas the liquid epoxy-LP prepolymer composition contains 50 phr or LP-33. The liquid epoxy-polyamide and polyamine coatings contain respectively 80 phr and 60 phr of curative. The LP modified

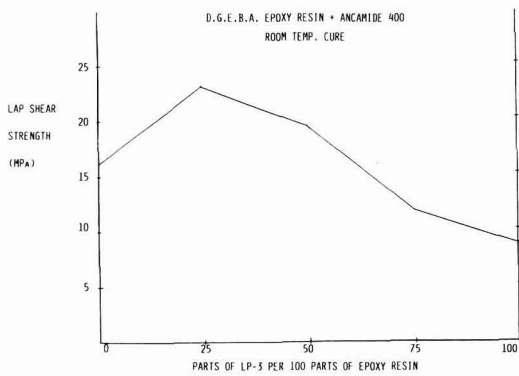


Figure 8. The effect of LP-3 level on the Lap Shear Strength of an Epoxy-Polyamide Resin.

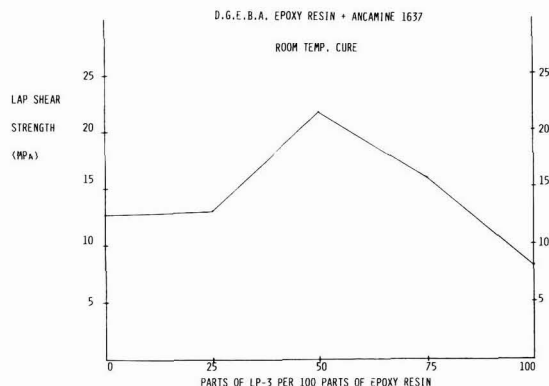


Figure 9. The effect of LP-3 level on the Lap Shear Strength of an Epoxy-amine system.

coatings exhibit excellent adhesion to the steel after salt spray exposure. The liquid epoxy-polyamide and liquid epoxy-polyamine coatings on the other hand were completely removed from the substrate when tested according to the cross cut method. The solid epoxy-polyamide and the solid epoxy-LP prepolymer coatings both adhere tenaciously to steel after salt spray exposure. The latter contains 25 parts of ZL-1400C per 100 parts of solid epoxy resin whereas the solid epoxy-polyamide contains 55 parts of the polyamide curative. Solid epoxy-polyamide coatings are widely used to protect structural steel work and their adhesion is well documented. When applied as high build coatings however, they lack flexibility and are unable to withstand movement such as vibration or flexing without cracking.

The improvements in adhesion afforded by LP modification are further demonstrated in Figures 8 and 9, where the Lap Shear strengths of two unfilled, room temperature cured, epoxy adhesives are plotted against LP-3 content.

Lap shear strengths were evaluated according to ASTM Method D1002 on degreased mild steel specimens using a 0.2 mm thick glue line over a joint overlap area of 25 mm by 15 mm. Figure 8 shows that for the epoxy-polyamide system (DGEBA + Ancamide 400), LP additions of up to 50 phr lead to an increase in the lap shear strength of the adhesive. Maximum adhesion is obtained when 25 phr of LP-3 is incorporated. Figure 9 illustrates that LP-3 additions of 25 to 70 phr are capable of increasing the adhesive strength of the epoxy-aliphatic amine system (DGEBA + Ancamine 1637). In this case a level of 50 phr of LP-3 provides maximum adhesion.

It should be noted that in both the above examples, relatively small additions of liquid polysulphide are required to produce significant improvements in the lap shear strength of the system.

Flexibility

LP modified epoxy resins exhibit outstanding flexibility and elasticity. LP modified epoxy coatings evaluated according to ASTM Method D-522, the Conical Mandrel test, can provide elongation figures which are near to the limit for the test. Table 3 illustrates the Conical Mandrel elongation figures for some of the coatings previously described in this paper.

Flexibility is maintained even after extended salt spray exposure (see Table 4) and demonstrates the advantage of formulating coatings using a flexibiliser which is chemically bound to the epoxy molecule and is therefore non-extractable.

Impact Resistance

The incorporation of a rubbery component into an epoxy resin also gives improved impact resistance. The effect of polysulphide modification is to change the epoxy resin from a brittle to a ductile material. Figure 10 illustrates the

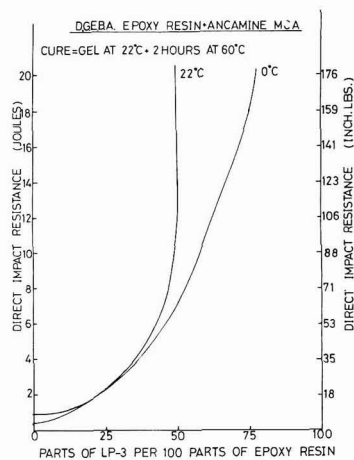


Figure 10. The effect of LP-3 level on the impact resistance of an Epoxy-amine system.

Table 3

Flexibility: ASTM Method D-522, Conical Mandrel Test

Coating	Mandrel Elongation (%)	Coating Thickness (Microns)
Solid Epoxy-Polyamide	5	300
Liquid Epoxy-LP Prepolymer	15	200
Solid Epoxy-LP Prepolymer	30	250
Conventional LP-Epoxy	30	200

Table 4

Flexibility: ASTM Method D522, Conical Mandrel Test

	Conical Mandrel Elongation	
	Initial Value	Value after 1000 Hours Cold Salt Spray
Conventional LP-Epoxy Coating	30	28

beneficial effect of LP-3 addition on the direct impact resistance of 3 mm thick castings of a DGEBA epoxy resin cured with the cycloaliphatic amine, Ancamine MCA. Impact tests were carried out on castings maintained at 22 and 0°C. Improvements in impact strength are particularly beneficial for coatings or adhesives intended for low temperature use.

As an example the LP-epoxy coating in Table 5 was spray applied onto smooth finish, type QD, cold rolled steel Q-Panels to a dry film thickness of 150-200 microns and then left for 7 days at 22°C/50% relative humidity prior to testing. At the end of the 7 day period the samples were placed at various set temperatures for 30 minutes and then reverse impact tested by the falling weight method. The results are given in Table 5. At 0°C the coating has an impact resistance greater than 18 Joules. At -40°C the impact resistance is 18 Joules. The coating ultimately becomes brittle at temperatures between -55 and -60°C. At these temperatures the coating has a reverse impact resistance of less than 0.45 Joules (<4 inch lbs.). Test panels painted with this coating can be bent through 180 degrees without cracking of the coating until the test temperature reaches -55°C. The Pencil Hardness of the coating as measured by ASTM Method D3363 is 5H and serves to illustrate that impact resistance and flexibility can be supplied to a coating without compromising surface hardness or mar resistance.

Thermal Shock Resistance

Another benefit of converting a brittle epoxy resin into a ductile product is the development of thermal shock resistance. The incorporation of flexible polysulphide polymer molecules into the more rigid epoxy chain ensures that the cured coating or adhesive can withstand, without fracturing, the expansion and contraction movements encountered during thermal cycling. Correctly formulated polysulphide-epoxy systems can withstand the effects of thermal cycling over a wide temperature range, from -55 to +85°C.

Chemical and Corrosion Resistance

A prime virtue of polysulphide-epoxy resins is their outstanding chemical and corrosion resistance. Unmodified epoxy resins have excellent resistance to dilute acids, alkalis and certain solvents. The incorporation of an LP component into the epoxy resin extends this resistance to include a wide variety of oils, aromatic and aliphatic hydrocarbons, esters and ketones. Coatings and adhesives based on polysulphide-epoxy resins exhibit reduced swell and maintenance of flexibility and adhesion when immersed in these fluids. LP-modified epoxy resins are particularly suitable for use as coatings or adhesives which come into contact with oils, fuels or water. These systems have excellent imper-

Table 5

Reverse impact resistance of conventional LP-Epoxy coating

CONVENTIONAL LP-EPOXY COATING					
Part A	pbw	Part B		pbw	
LP-3	60	Liquid Epoxy Resin (EEW=190)		120	
Titanium Dioxide	94	Solid Epoxy Resin (EEW=500)		80	
Ancamine 1768	76	Flow Promoter		10	
Xylene	40	MEK		20	
Toluene	40	MIBK		92	
		Xylene		79	
		Butyl Cellosolve		13	
	310			414	
Weight Mix Ratio Part A: Part B = 310:414					
Reverse Impact Resistance (Joules)					
Conventional LP-Epoxy Coating	22°C	0°C	-30°C	-40°C	-60°C
	>18	>18	18	18	Failed

meability to solvent or fuel vapours, moisture vapour and gases. Given this "all round" chemical resistance it is not surprising that LP modified epoxy resins are used on oil rigs, fuel storage installations and chemical plant.

Resistance to water and water vapour stems not only from the impermeability of the LP-epoxy resin but also from the fact that, unlike certain other polymeric flexibilisers, the LP component is not hydrolysable.

Impermeability and resistance to hydrolysis is no advantage to a coating or adhesive if the resin does not adhere to the substrate in the first place. The superior wetting characteristics of LP modified epoxy systems provides improved adhesion to the substrate. The better the adhesion to the substrate the less likely any fluid can reach the resin - substrate interface and attack the substrate itself. This effect was noted in the coatings evaluated for cold salt spray resistance (see Table 2). In this test the LP modified resins not only maintained their adhesion to the mild steel plates but also prevented the ingress or spread of corrosion. The liquid epoxy-polyamide and epoxy-polyamine based coatings on the other hand could not prevent corrosion spread and became de-bonded from the badly corroded steel substrate.

As a result of their adhesion and chemical/corrosion resistance LP modified epoxy resins are often formulated into primer coatings for concrete, steel and aluminium substrates.

Weathering Resistance

The absence of unsaturation or carbonyl structures in the polysulphide molecule make the liquid polymers less susceptible to UV or ozone attack. The inclusion of these polymers into an epoxy resin will not

prevent the yellowing associated with epoxy systems that have been subjected to continuous bright sunlight - although in some cases it can reduce this yellowing. However, the liquid polysulphide does significantly reduce the level of embrittlement that occurs when epoxy based resins are subjected to the combined effects of ultra violet light and elevated temperatures.

As an example, the three coatings discussed in Table 6 were exposed to UV (type A) light and temperatures of 70°C in a Q-UV Weatherometer prior to being reverse impact tested. Table 7 compares the resulting impact resistance of these coatings. All three coatings have excellent unexposed impact resistance, but this is reduced as the systems become embrittled. This effect is most noticeable for the epoxy-polyamide coating which, by the end of the exposure period has negligible impact resistance. The LP modified coatings on the other hand perform in a superior manner and have residual impact resistance on completion of this demanding test. Such accelerated weathering test results correlate well with findings obtained from outdoor exposure trials conducted in the UK and India where the LP modified coatings demonstrate their insensitivity to moisture and bright sunlight.

Conclusion

This paper has illustrated the wide range of beneficial properties that are associated with polysulphide modified epoxy resins. Coatings and adhesives based on these resins are used whenever their excellent adhesion, flexibility, impact resistance and chemical and corrosion resistance make them the most cost effective choice. Some typical application areas for these products are tested overleaf.

Table 6

Coatings Subjected to Heat and UV Light

	Solid Epoxy LP-33 Prepolymer Coating	Solid Epoxy ZL-1400C Prepolymer Coating	Solid Epoxy-Polyamide Coating
Solid Epoxy LP-33 Prepolymer (1)	100	—	—
Solid Epoxy ZL-1400C Prepolymer (1)	—	100	—
Solid Epoxy Resin (1)	—	—	100
Polyamide (2)	—	—	55
Titanium Dioxide	25	25	25
Flow Promoter	20	20	20
Ancamine 1608	4	4	—
Solvent mixture	40(3)	40(3)	53(4)

(1) Solid epoxy resin of E.E.W. = 500

(2) Polyamide resin of amine value = 210-220 mgKOH/g

(3) Solvent mixture = 2:1 by weight MEK:Xylene

(4) Solvent mixture = 1:1 by weight Toluene:IPA

Table 7

The effect of heat and UV light on coating impact strength

	Reverse Impact Resistance (Joules)		
	Solid Epoxy LP-33 Prepolymer Coating	Solid Epoxy-ZL-1400C Prepolymer Coating	Solid Epoxy-Polyamide Coating
Initial Value	18	18	18
After 240 Hours UV at 70°C	2.3	4.5	2.3
After 480 Hours UV at 70°C	0.45	0.9	*

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Adhesives

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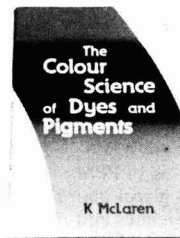
Benefits

Good wetting characteristics.
Adhesion to many substrates without priming.
Elastomeric - can withstand movement and vibration.
Reduces stress between dissimilar bonded materials.
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Resistant to thermal cycling.
Chemical resistance to oils and fuels.

*Solaglass Sealants, Unit 33, Bentley Avenue, Stake Hill Industrial Estate, Middleton, Manchester.

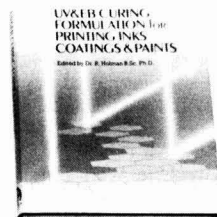
Polysulphides are not new polymers and are well established in the fields of sealant and rubber technology. Because of its proven track record, polysulphide chemistry is continually striving to reach new market areas. The new generation of LP modified epoxy coatings and adhesives represent one example of this expanding technology.

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New propylene glycol ethers for waterborne coatings

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Introduction

Glycol ethers have, for many years, been well known solvents in the paint and coating industry. They are widely used in both solvent and waterborne coatings. Until the beginning of the 80's, ethylene glycol based ethers, the so called E-series, were the commonly used products. As it was discovered, by the end of the 70's, that some members of the family of E-series glycol ethers could give rise to toxicological problems under certain circumstances, and in particular that they are teratogenic, people started to think about alternative products.

Today, the most questionable members of the E-series glycol ethers family, methyl glycol and ethyl glycol and their acetates, are in the process of being phased out or drastically reduced in many of their applications, e.g. solvent borne coatings.

The most widely used alternative products for methyl and ethyl glycol are glycol ethers based on propylene glycol, the P-series, which has been proved not to exhibit teratogenic effects. The most well known members of the family of P-series glycol ethers, methoxy propanol, is a common solvent today in the world of paints and lacquers, mainly replacing previously used ethyl glycol.

In the area of waterborne coatings another glycol ether, (butyl glycol) from the E-series is still widely used. Butyl glycol is a very special molecule: it combines a highly hydrophilic ethylene glycol "head" with a lipophilic "tail"; the n-butyl ether. This structure provides unique coupling properties for hydrophilic and lipophilic phases. For these reasons butyl glycol is an almost unique additive for water reducible resins. It also is an excellent, fast evaporating coupling and coalescing agent for dispersion coatings.

According to the latest glycol ethers toxicological information butyl glycol, unlike methyl and ethyl glycol, is not teratogenic. It possesses however pronounced hemolytic properties, which, combined with easy absorption through the uninjured skin make it less desirable for the use in products like cleaners, do-it-yourself paint etc, which might come in contact with the skin.

The latest revision of TLV (or MAK) values for glycol ethers in most countries of the EEC, in 1985/86, did not leave butyl glycol untouched.

Today the MAK value in UK is 25 PPM, versus 10 PPM for ethyl glycol. In West Germany both ethyl glycol and butyl glycol are at 20 PPM. Also labelling guidelines and emission control regulations are relatively unfavourable for butyl glycol in many countries today. In the EEC labelling regulations, butyl glycol is classed in group II B, as a harmful chemical. This gives rise to some usage limitations.

It is probably due to these reasons that there is a growing

interest in the industry, particularly in the coatings and cleaners area, for alternative products for butyl glycol.

As with the replacement of methyl glycol and ethyl glycol by propylene glycol ethers, it seems logical to look first at alternatives for butyl glycol of this group of products.

Dow Chemical, as a leading manufacturer of propylene glycol ethers has recently introduced Dowanol* PnB, n-butoxy propanol or propylene n-butyl ether, and Dowanol DPnB, n-butoxy methyl ethoxy propanol or di-propylene glycol n-butyl ether, as realistic technical alternatives for presently used co-solvents and coalescing aids for waterborne coatings.

**Trademark of The Dow Chemical Company.*

Dowanol PnB and DPnB

Comparing physical properties (Table 1) of Dowanol PnB with EB one can see that these are almost identical for both products in almost all respects. Strikingly different is the water solubility of n-butoxy propanol, which is only 6%, against EB, (n-butoxy ethanol) which is completely water soluble.

This difference already demonstrates, that PnB is more lipophilic than EB. Looking at the molecular structure provides no surprises: the propanol end of PnB is obviously less hydrophilic than the ethanol end of EB, resulting in a greater overall lipophilic character for PnB.

It should be mentioned that this figure for water solubility refers to pure water. In actual coating formulations, containing resins, fillers, surfactant etc, the water solubility can increase remarkably.

On the other hand, due to the more lipophilic character of PnB the coating layer will dry faster under humid atmospheric conditions and develop earlier and higher film hardness.

Comparing the evaporation speed of various, commonly used, co-solvents/coalescing aids (Figure 1), one notices two different groups: the fast evaporating and the slow evaporating groups. As a rule, the slowly evaporating types exhibit the higher coalescing performance, whereas the faster evaporating products offer more rapid film-drying and highest film hardness. Depending on requirements and application for a particular coating formulation, a choice may then be made.

In both groups the n-butanol based propylene glycol ethers—the mono-propylene glycol n-butyl ether (PnB) and the di-propylene glycol n-butyl ether (DPnB)—are the products which evaporate most rapidly and are often the best performers overall.

Table 1

Physical properties of PnB and EB

	N-BUTOXY PROPONAL		BUTYL GLYCOL	
	(PnB)		(EB)	
MOL. WEIGHT	132		118	
BOILING POINT 760 mmHG, °C	170		171	
VAPOUR PRESS. AT 25°C (mmHG)	0.6		0.88	
RELATIVE EVAPORATION RATE (BuAC=1)	0.07		0.06	
SPECIFIC GRAVITY (25°/25° H ₂ O)	0.88		0.90	
VISCOSITY mPa.S, 25°C	3.26		3.15	
SURFACE TENSION, 25°C, DYNES/cm	26.3		27.4	
SOLUBILITY. IN WATER (g/100ml)	ca.	6	ca.	∞
(20°C) WATER IN - (g/100ml)	ca.	16	ca.	∞
FLASHPOINT (C.O.C.), (°C)	66		74	
REFRACTIVE INDEX N _D 20	1.416		1.419	
MELTING POINT, °C	<-75		-75	

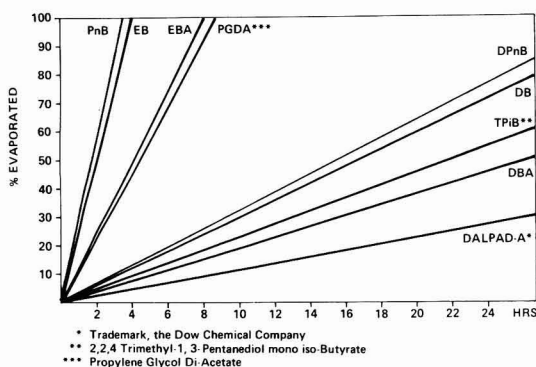


Figure 1. Relative evaporation speeds of various solvents/coalescing aids.

Coalescing performance

In Figure 2 the coalescing performance of three typical water dispersible resins are compared: 1. An acrylic resin with high MFFT, 2. A styrene-acrylate copolymer resin with high MFFT and 3. A copolymer resin with low MFFT.

In all cases DPnB, together with PGDA, proves to be the most efficient coalescing aid, outperforming TPiB and the fast evaporating glycol ethers EB and PnB. The two latter products being, on average, equal in performance.

Looking at the physical properties of the series of slow evaporation coalescing aids (Table 2) one sees, apart from the water solubility, few differences. From a performance point of view, however, DPnB clearly outperforms the others. It exhibits the best coalescing performance, equal to PGDA, but has the advantage over PGDA and all the other esters in that it is stable against hydrolysis. This means that, over time, neither loss of activity nor development of undesired smell will present a problem.

Film hardness

An important parameter for the quality of a coating film is

the hardness and the speed with which this hardness develops after application of the film. In waterborne coatings the evaporation speed of the coalescing aid, or co-solvent plays a key-role here.

Hardness evolution testing performed with various coalescing aids for two different resins is shown in Figure 3.

The film hardness was measured according to DIN-standard method, using a Koenig Pendulum apparatus.

The amounts of coalescing aid were chosen in such a way, that the MFFT in all cases was approximately 3°C.

Hardness evolution as well as final hardness (after 96 hours) were very much dependent on the coalescing aid used.

As might be expected, EB and PnB showed the fastest hardness build up requiring, and thus have to be used in relatively large amounts.

How a resin can influence the evaporation characteristic of a coalescing aid is illustrated here by the behaviour of DPnB: In one case it typically behaves like a slow evaporating type. In the other case it almost equalled PGDA and therefore belongs to the group of fast evaporators.

For other resins similar interactions with particular coalescing aids can be expected. For that reason, no general rules can be given for performance of coalescing aids in various formulations. The right amount of the best performing coalescing aid for each particular coating formulation should always be arrived by experimental means.

It can be said however, that these new P-series glycol ether coalescing aids rank among the best in most formulations. Their features are:

- excellent coupling
- highest coalescing performance
- stability against hydrolysis
- good odour

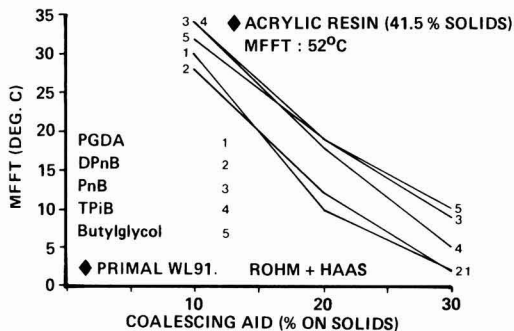
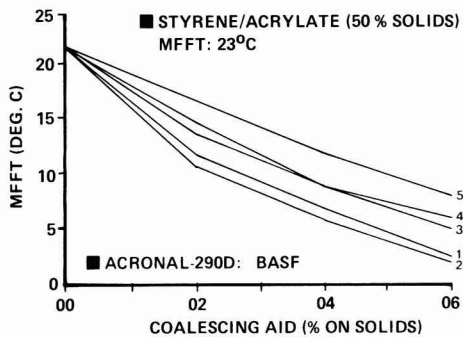
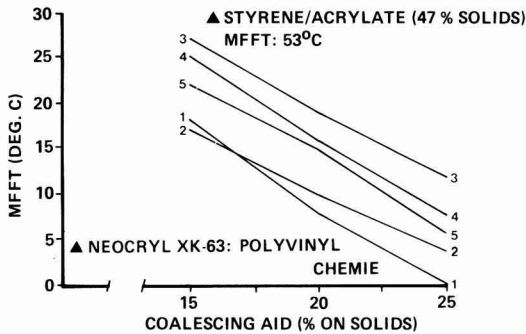
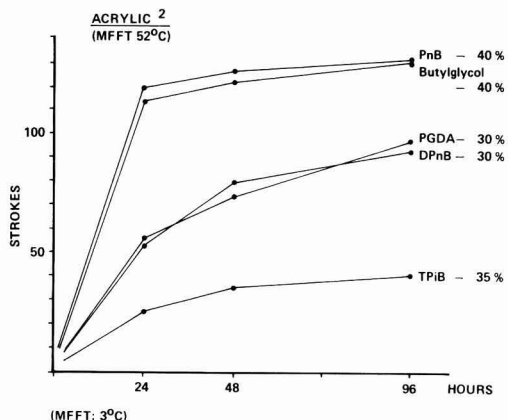
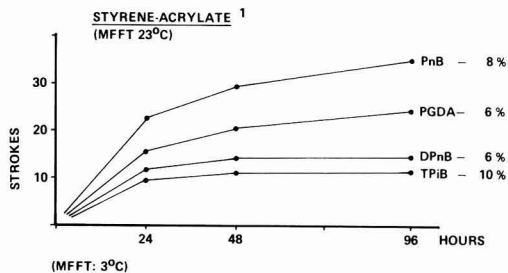


Figure 2. Coalescing performance of various coalescing aids for different resins.



1 ACRONAL 290-D BASF
2 PRIMAL WL-91 ROHM + HAAS

Figure 3. Film hardness (Koenig) against time (Acc. to DIN 53157) for two different resins.

- low toxicity, based on present data
- favourable labelling in comparison to mono-ethylene glycol ethers
- most lypophilic water soluble glycol ethers

These properties make them ideal as first choice for many applications and provide a guarantee for high quality products.

Table 2

Physical properties of slow evaporation coalescing aids

	DPnB	DB*	DBA	TPiB	PGDA
MOL. WEIGHT	190	162	204	216	160
BOILING POINT 760 mmHG, °C	229	230	247	245	190
VAPOUR PRESS. AT 25°C (mmHG)	0.06	<0.1	<0.1	<0.1	<2
RELATIVE EVAPORATION RATE (BuAC=1)	~0.01	<0.01	<0.01	<0.01	0.04
SPECIFIC GRAVITY (25°/25° H ₂ O)	0.913	0.952	0.981	0.95	1.06
VISCOSITY mPa.S, 25°C	4.6	5.17	3.2	—	2.65
SURFACE TENSION, 25°C, DYNES/cm	—	30.0	—	—	31.1
SOLUBILITY: -IN WATER (g/100ml)	ca. 5	∞	6.5	0	9.0
(20°C) WATER IN - (g/100ml)	ca. 12	∞	3.7	0.01	4.3
FLASHPOINT (C.O.C), (°C)	113	110	115	120	95
REFRACTIVE INDEX N _D 20	1.425	1.432	1.426	1.4423	1.4127
BOILING POINT, °C	-75	-76	-32	-50	-75

* Diethylene Glycol n-Butyl Ether

Some anticorrosive primers free of lead and chromate

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Summary

Red lead and chromate pigments are frequently used in anticorrosive primers for the protection of steel structures. Unfortunately these pigments are hazardous; lead pigments may lead to mental retardation in children, in addition to their cumulative toxic effects. On the other hand, chromium compounds cause dermatitis, skin sensitization and are a carcinogenic hazard. In the present work a number of safer and non-hazardous formulations has been investigated. They are based on zinc phosphate and barium metaborate pigments and these show promising results as anticorrosive primers.

Introduction

Anticorrosive pigments are tentatively described, and among these is red lead which is one of the oldest pigments that has been used for the protection of iron and has perhaps the longest history of exposure experience¹.

Red lead is an orange-red highly toxic pigment, it is a reactive pigment and its chemical reactions support the accepted constitution $2\text{PbO} \cdot \text{PbO}_2$, when dispersed in linseed oil or other oxidizing media, lead soaps are formed by reaction between the PbO and acidic by-products produced during the oxidation process. These soaps are thought to play an important part in the protection afforded to iron and steel by red lead primers².

Zinc chrome is another anticorrosive pigment and is used in priming steel and light alloys. $\text{ZnCrO}_4 \cdot 4\text{Zn}(\text{OH})_2$. It contains very little soluble chromate. The steel protection is thought to result from the presence of soluble chromate in the primer. Moisture permeating the film carries chromate ions to the steel surface where they polarize the anodic areas of potential corrosion cells³.

Contrary to compounds which cause dermatitis, skin sensitization, asthma and have carcinogenic hazards⁴, zinc phosphate $\text{Zn}_3(\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$ has been proved to be an anticorrosive and non-toxic pigment⁵. The mechanism of the protection by zinc phosphate is not completely understood. According to Clay and Cox⁶, zinc phosphate reacts by polarizing both anodic and cathodic areas as a

result of slight solubility. On the other hand, the pigment appears to pack in the film in a manner which presents a high resistance to the passage of water molecules and salts. The protective qualities are likely to be a combination of electrochemical suppression of corrosion cells with a barrier effect.

The Meyer theory⁷ might illustrate the mechanism of zinc phosphate protection as outlined in Scheme 1 below.

Modified barium metaborate (Busan 11 M1) pigment imparts a number of desirable properties to paints and paint films. In addition to corrosion resistance, modified barium metaborate can be used for different aspects, e.g. bacterial control, fungal resistances, enzyme inhibition, chalk resistance, fire resistance, and increased colour retention⁸. Modified barium metaborate has the formula $\text{BaB}_2\text{O}_4 \cdot \text{H}_2\text{O}$, it is, probably, the least hazardous pigment used as a corrosion inhibitor or fire resistance agent, it does not generate toxic fumes when paints are welded or when products containing it burn⁹.

Methods of preparation, testing and evaluation

Preparation

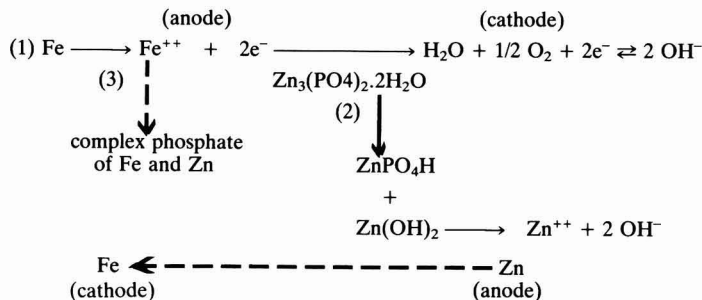
1 Kg. samples of paint were prepared in the laboratory using a three roll mill. Twenty anticorrosive primers were prepared (I-XX) in five sets. Primers based on zinc phosphate are ranked (I-IV), barium metaborate (V-VIII), zinc tetraoxochromate (IX-XII), red lead (XIII-XVI) and zinc phosphate/barium metaborate (XVII-XX). The formulations of primers of each set is shown in Tables 1-5.

Methods of testing and evaluation

These formulations (I-XX) were subjected for testing and evaluation according to well-known and standard methods. These tests include:

- Preparation of glass panels¹⁰
- Preparation of steel panels¹¹
- Detremination of viscosity by Ford cup¹²

Scheme 1



Applying coatings by using wire-wound drawdown bar¹³
 Determination of hardness by pendulum hardness tester¹⁴
 Dry film thickness (minitest FN 252)¹⁵
 Evaluating degree of blistering of paints¹⁶
 Degree of rusting¹⁷
 Adhesion by cross-cut¹⁸
 Testing the ductility (deformability) by cupping test machine¹⁹
 Drying time²⁰
 Determination of gloss²¹
 Resistance of organic coatings to the effects of rapid deformation (impact)²²
 Elongation of attached organic coatings with conical mandrel apparatus²³
 Operating light and water exposure apparatus (fluorescent UV-condensation type) for exposure of non metallic materials²⁴.

Table 3

Primers based on zinc tetroxychromate

Primer	IX	X	XI	XII
Additives	0.3	0.3	0.3	0.3
Bentone gel	5	5	5	5
Zinc tetroxychromate	8	10	12	16
Talc	40	38	36	32
Alkys resin binder	35	35	35	35
Drier	2	2	2	2
Thinner	9.7	9.7	9.7	9.7
	100	100	100	100
Pigment/binder ratio	2.49:1	2.49:1	2.49:1	2.49:1

Table 4 illustrates the composition of primers based on red lead.

Table 4

Primers based on red lead

Primer	XIII	XIV	XV	XVI
Additives	1.2	1.2	1.2	1.2
Red lead	65	68	71	75
Talc	7	7	7	7
Linseed oil	26.2	23.2	20.2	16.2
Drier	0.6	0.6	0.6	0.6
	100	100	100	100
Pigment/binder ratio	2.7:1	3.2:1	2.68:1	5:1

Table 5 illustrates the compositions of a set of primers based on a mixture of zinc phosphate and barium metaborate.

Table 5

Primers based on zinc phosphate and barium metaborate

Primer	XVII	XVIII	XIX	XX
Additives	0.5	0.5	0.5	0.5
Titanium dioxide	20	20	20	20
Barium metaborate	26	24	22	20
Zinc phosphate	6.2	8.2	10.2	12.2
Alkyd resin binder	38	38	38	38
Drier	2	2	2	2
Thinner	7.3	7.3	7.3	7.3
	100	100	100	100
Pigment/binder ratio	2.49:1	2.49:1	2.49:1	2.49:1

Results, discussion and conclusions

The evaluation data are collectively given in Tables 6, 7 and 8. The following aspects were taken into consideration during the formulation of anticorrosive compositions:

1. 'Natural' oxide layer on iron and steel surfaces possess many defects, at which local corrosion cells become active in a corrosive environment via their soluble fraction. Iron oxide pigments which are present in formulations (I-VIII) are able to overcome this activity by forming iron oxide directly in contact with the local electrodes, thus stifling the action of these elements under favourable circumstances²⁵.

Tables 1-5

Table 1 illustrates the primer compositions using zinc phosphate.

Table 1

Primer based on zinc phosphate

Primer	I	II	III	IV
Additives (antisetling & antiskinning)	0.5	0.5	0.5	0.5
Yellow iron oxide	14	12	10	—
Red iron oxide	11.2	10.2	9.2	9.2
Talc	10	9	8	—
Zinc phosphate	17	21	25	23
Titanium dioxide	—	—	—	20
Alkyd resin binder	38	38	38	38
Drier	2	2	2	2
Thinner	7.3	7.3	7.3	7.3
	100	100	100	100
Pigment/binder ratio	2.49:1	2.49:1	2.49:1	2.49:1

Table 2 illustrates the primer compositions based on barium metaborate.

Table 2

Primers based on barium metaborate

Primer	V	VI	VII	VIII
Additives (antisetling & antiskinning)	0.5	0.5	0.5	0.5
Titanium dioxide	20	20	20	20
Barium metaborate	20	22	24	26
Red iron oxide	12.2	10.2	8.2	6.2
Alkyd resin binder	38	38	38	38
Drier	2	2	2	2
Thinner	7.3	7.3	7.3	7.3
	100	100	100	100
Pigment/binder ratio	2.49:1	2.49:1	2.49:1	2.49:1

Table 3 illustrates the compositions of primers based on zinc tetroxychromate.

Table 6

Film characteristics

Primer No.	Type of anticorrosive pigment	Drying time, Hr.	Film thickness		Viscosity Ford Cup	
			wet	dry	min	sec
I	Zinc phosphate	8.5	100	30	3	29
II	Zinc phosphate	5.0	100	40	1	22
III	Zinc phosphate	8.5	125	30	1	31
IV	Zinc phosphate	5.5	100	35	—	51
V	Barium metaborate	5.0	100	30	1	10
VI	Barium metaborate	2.5	100	25	1	15
VII	Barium metaborate	3.0	100	45	3	32
VIII	Barium metaborate	4.0	125	35	2	15
IX	Zinc tetroxychromate	8.2	200	55	2	33
X	Zinc tetroxychromate	8.5	100	30	1	49
XI	Zinc tetroxychromate	1.0	100	35	1	48
XII	Zinc tetroxychromate	5.0	100	40	1	41
XIII	Red lead	52.0	125	40	—	36
XIV	Red lead	40.0	100	40	—	47
XV	Red lead	24.0	175	45	1	18
XVI	Red lead	24.0	175	50	1	21
XVII	Zinc phosphate/barium metaborate	1.5	175	65	3	29
XVIII	Zinc phosphate/barium metaborate	3.0	150	60	3	57
XIX	Zinc phosphate/barium metaborate	2.0	225	60	3	—
XX	Zinc phosphate/barium metaborate	3.0	150	45	3	50

Table 7

Mechanical properties of films

Primer No.	Gloss geometry degree	Elongation, % (conical Mandrel)*	Ductility (Erichsen cupping)†	Hardness pendulum (Koing)‡	Adhesion cross- cutΔ	Impact denting□
I	3.7	26	5	50	Gt 0	100
II	3.9	26	4	56	Gt 0	100
III	4.0	26	5.5	56	Gt 0	100
IV	39.0	26	6.0	64	Gt 1	100
V	31.3	26	5.5	73	Gt 1	100
VI	22.0	26	4.0	72	Gt 1	100
VII	22.0	26	3.5	70	Gt 1	100
VIII	19.2	26	4.0	67	Gt 2	100
IX	6.4	26	2.5	67	Gt 0	100
X	6.5	26	6.0	63	Gt 0	100
XI	7.0	26	5.5	62	Gt 0	100
XII	6.5	26	2.0	54	Gt 0	100
XIII	73.7	17.1	7.0	15	Gt 2	100
XIV	58.5	19	4.5	18	Gt 2	100
XV	52.8	14.2	10	16	Gt 2	100
XVI	26.6	11.4	1.5	20	Gt 2	50
XVII	18.3	26	3.5	65	Gt 1	60
XVIII	21.0	26	4.0	63	Gt 2	50
XIX	23.1	26	4.5	65	Gt 0	60
XX	33.3	26	5.0	58	Gt 1	100

* Elongation by conical Madrel is determined from a curve relating the distance of the first crack against elongation in percent. A correction needed for thickness of films is derived from another curve. The elongation in percent ranges from 2-26% and the correction for thickness of films ranges nearly from 0.2-1.2%.

† Ductility (deformability) of paints determines the depth of a dent in the coating system at which the coating tears or flakes.

‡ Hardness of films is evaluated through damping effect of an oscillation. The damping will increase with increasing "softness" of the coating to be tested. The reference value is the time (in seconds) expressed as the damping period, which the pendulum requires to slow down from the initial value to a lower value.

Δ Adhesion was determined by the cross-cut adhesion tester. The evaluations are ratings Gt 0, Gt 1, Gt 2, Gt 3 and Gt 4. Where Gt 0 means that the cut edges are completely smooth; no part of the coating chipped off, Gt 4 where the coating has chipped off along the cut edges in wide stripes and/or totally or partially from individual parts, and the chipped off area about 65% or more of the intersectional areas.

□ The impact adhesion tests the paints for elongation and bending and by derivation, adhesion due to sudden deformation. It determines the impact force necessary to crack a coating due to deformation of the base. When the test panel film is facing downwards, it refers to buckling (reverse). Denting is referred when the test panel film is facing upwards (direct).

Table 8

Q-UV accelerated weather testing data

Prime No.	Size	Blistering†	Q-UV accelerated weathering testing*		Rusting‡	Efficiency
			Frequency	Re		
I	4		Few	4		6
II		No blistering		2		8
III		No blistering		0		10
IV	8		Few	0		10
V	8		Few	0		10
VI	8		Few	0		10
VII	8		Few	0		10
VIII		No blistering		0		10
IX		No blistering		6		4
X		No blistering		3		7
XI		No blistering		6		4
XII		No blistering		6		4
XIII		No blistering		0		10
XIV		No blistering		0		10
XV		No blistering		0		10
XVI		No blistering		0		10
XVII	6		Medium	0		10
XVIII	6		Medium	0		10
XIX	6		Few	0		10
XX	4		Few	0		10

* Blistering and rusting were carried out after 61.7 Hr. (total time), and 31.7 Hr. UV exposure.

† Blistering is surface defect. Two characteristics are shown, size and frequency. The size is described on an arbitrary numerical scale, and the frequency is described qualitatively. For size, four steps as to size on numerical scale from 10 to 0, in which No. 10 represents no blistering. Blistering rate 6, 4 & 2 represents progressively larger size. Frequency is rated dense D, medium dense MD, medium M and few F.

‡ Degree of rusting is rated at Re 0=rust free, Re 9=whole surface rusty. Degree of paint protection efficiency 10=rust free, 1=whole surface rusty.

In general the incorporation of red iron oxide imparts good chemical resistance, colour fastness, excellent heat resistance and high tinting strength. Yellow iron oxide has excellent durability, and it has excellent chemical and alkali resistance²⁷.

2. Talc is incorporated in formulations I-III & I-XVI to combine both durability and cost reduction²⁷.

3. Barium metaborate was incorporated in formulations V-VIII as the safest pigment.

4. In formulations IX-XII, zinc tetroxychromate was used in amounts from 8-16% by weight. Bentone gel is also included among the ingredients as a viscosity controller, antisag and antisetling agent.

5. Formulations XIII-XVI based on red lead as anticorrosive primer contained linseed oil as the binder base. These compositions were introduced for the sake of comparison since red lead formulations are well-known traditional anticorrosive primers.

6. Formulations XVII-XX are pigmented with a combination of zinc phosphate and barium metaborate together with other pigments.

It should be noted that in all formulations the same alkyd resin was used as a base binder except for formulations XIII-XVI (where linseed oil is used as binder). Also the drier employed is of the lead-free type.

From Tables 6-8 it can be concluded that:

1. Red lead composition showed, as expected, the longest drying time of all formulations examined.

2. The gloss value of most primers studied showed moderate values as measured at 60° incident angle. Relatively higher values were obtained with formulations based on linseed oil. Increasing the percentage of barium metaborate resulted in lower gloss values.

3. All films of compositions based on alkyd resins show high elongation percentages compared with films of linseed oil base. Similar behaviour was also noticed regarding hardness and adhesion measurements.

4. Formulations based on titanium dioxide, barium metaborate and zinc phosphate showed the lowest values of impact strength. No definite conclusions were drawn from ductility studies, since the data was highly variable.

5. Formula I and XVII-XX showed tendency to blister, while films VIII-XVI show no blistering after exposure for 61.7 Hr. Films IV-VIII show minor tendency to blistering.

6. Formulae III-VIII and XIII-XX show no rusting whilst films I, II & IX-XI and XII are the worst; they also showed a tendency to rust.

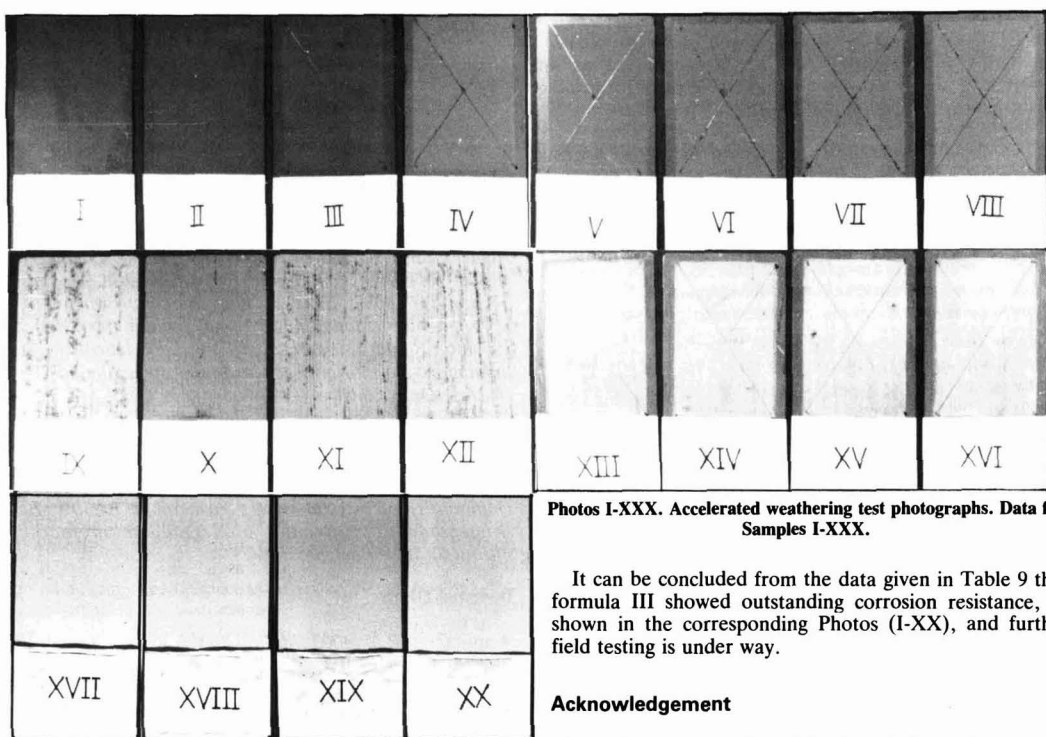
The most important findings drawn from the above record of study of the Q UV accelerated weathering testing are that anticorrosive primer compositions based on yellow iron oxide 10%, red iron oxide 9.2%, talc 8% and zinc phosphate 25% offer outstanding corrosion resistance.

Table 9

Results of the Q-UV accelerated weathering testing for 150 Hr.

Total time, Hr.	Condensation*, Hr. at 60°	UV, Hr. at 60° C*	Non-affected primer No.	Affected primer No.	Types of change
82	38	44	III, IV & V VI & VIII	XIII & XIV XVI	chalking & discoloration
93	43	50	III, IV, V & VI	VIII	rusting,
110	51.1	58.9	III, V & VI	XIII & XVI IV & VIII	chalking & discoloration
122	58	64	III, V & VI	XIII & XVI IV & VIII	rusting,
129	61	68	III, V & VI	XIII & XVI IV & VIII	chalking & discoloration
150.7	71.3	79.4	III	IV, V, VI & VIII XIII & XVI	rusting, chalking & discoloration

* Cycle is 4 hours.



Photos I-XXX. Accelerated weathering test photographs. Data for Samples I-XXX.

It can be concluded from the data given in Table 9 that formula III showed outstanding corrosion resistance, as shown in the corresponding Photos (I-XX), and further field testing is under way.

Acknowledgement

The authors are greatly indebted to Professor Dr. A. M. Naser, Faculty of Science, Al-Azhar University, for his invaluable guidance, revising and co-operation during this work.

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2. Morgans, W. M., Outlines of Paint Technology, Vol. 1, Materials, Charles Griffin & Co., Second edition, England 1982, p 95-96.

Those belonging to zinc tetroxychromate showed lower efficiencies.

From previous studies it seems that formula III showed promising results as anticorrosive composition.

This pigment was subjected to an extensive study to confirm its efficiency. Selected compositions were subjected to further evaluation in order to assess their anticorrosive properties. The results are given in Table 9.

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4. Hess's Paint Film Defects, Their Causes and Cure, Third edition, London, *Chapman & Hall*, 1979, p 347.
5. Ref. 2. p 59-60.
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9. Ref. 1, p 945.
10. ASTM D 3891 - 80.
11. ASTM D 609 - 73 (reapproved 1980).
12. ASTM D 1200 - 82.
13. ASTM D 4147 - 82.
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15. DIN 50 981.
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19. DIN 53 156.
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21. BS 3900.
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27. *Ibid*, 3, p 26-29.

conference discussions

Following the presentation of each paper at the OCCA Conference a period of time was allotted for discussion. Those who put the questions were asked to fill in discussion slips with the details of their question. Edited versions of the discussion (recently received) that followed the paper published in this, the January and the December issue, appear below: questions in ordinary roman type, answers in italic. Only those questions for which discussion slips were received are published.

Development of novel driers for paint by N. Usman

R. GOUGH: Can Mrs Usman expand on the problem of toxicities with benzoyl peroxide as suggested as an alternative drier to lead. Its use in face creams for healing spots suggests it cannot really be toxic?

N. USMAN: I quote the Toxic Hazard Rating for benzoyl peroxide from the book "Dangerous Properties of Industrial Materials" by N. Irving Sax: Acute Local = Irritant 2; Ingestion 2; Inhalation 2. Acute System = U. Chronic Local = Irritant 2. Chronic System = U. Explosion Hazard = Reacts violently in contact with various organic or inorganic acids metallic naphthenates etc. Disaster Hazard = Dangerous and may explode spontaneously.

Key to Toxic Hazard Rating Code

2 = Moderate: May involve both reversible and irreversible changes not severe enough to cause death or permanent injury. U = Unknown.

In Comparison for both cobalt and calcium compounds the Toxic Hazard Rating given in the same book = 1 (slight: causes readily reversible changes which disappear after end of exposure).

R. H. E. MUNN: A comment rather than question concerning the toxicity of benzoyl peroxide. It seems to me the word should be "dangerous" since it is well known that this peroxide will react violently with cobalt driers. Paint manufacturers normally mix their driers together before adding to the paint, if a benzoyl peroxide/cobalt drier mixture was being used, this would lead to a potentially violent explosion.

N. USMAN: Your comment supports my conclusion drawn for the unsuitability of benzoyl peroxide as an alternative drier with cobalt and calcium. Obviously at low concentrations (0.01% benzoyl peroxide) the risk of an explosion is much less than in a paint manufacturing plant where large quantities of materials are mixed.

E. A. JACKSON: Have you tried any other rare earth metals in your tests, vanadium, etc?

N. USMAN: We evaluated a wide range of metal driers as alternative driers to lead. This included driers based on zirconium, cerium, neodymium, barium and bismuth. Vanadium was not included in the evaluation work.

New propylene glycol ethers for water borne coatings by J. Spauwen

T. FJELDBERG: Which toxicity studies have been undertaken so far regarding PnB?

J. SPAUWEN: A summary of toxicological data generated so far for PnB and DPnB is given below:

Dowanol PnB

Acute Oral Rat: 3300 MG/KG/BW; Acute Dermal Rat: > 2000 MG/KG/BW; Skin Irritation Rabbit: Moderately irritating at 100%; EEC labelling; moderately irritating at 75%, slightly irritating at 50%, not irritating at 25%; Eye Irritation Rabbit; Moderately irritant; EEC labelling; Acute Inhalation: No mortality at maximum attainable dose level; 14-Day Oral Gavage 0-100-200-400 MG/KG: Nael 400 MG/KG; 3 Mo Dermal Tax Rat 0, 0.1, 0.3, 1.0/KG; In progress; Dermal Teratology Rat 0, 0.3, 1.0, ML/KG; Nael 1.0 ml/Kg; Sensitisation Test: No Sensitisation; Ames Test: Negative; Chromosome Aberration Test: Negative; Daphnia; EC50 > 1000 MG/L; Fish: 560 < LC50 < 1000, Biodegradability: Readily biodegradable in modified sturmtest, September 1987/HV.

Dowanol DPnB

Acute Oral Rat: 4000 MG/KG/BW; Acute Dermal Rat: > 2000 KG/KG/BW; Skin Irritation Rabbit: Slightly irritating, no EEC labelling; Eye Irritation Rabbit: Mildly irritant, no EEC labelling; Acute Inhalation Rat: No toxicity at maximum attainable dose level; 9-Day Inhalation Rat: Dose levels 0-20-40 PPM, no significant effects at maximum attainable dose level; 14-Day Oral Gavage 0-100-200-400 MG/KG: No haemolysis, nael including other criteria: 400 MG/KG; 3 Mo Dermal Rat 0, 0.1, 0.3, 1.0 ML/KG; In progress; Dermal Teratogenicity Rat 0, 0.3, 1.0 ML/KG: No teratogenic effect at 1ML/KG/DAY; Sensitisation G Pigs: No sensitisation; Ames Test: Negative; Daphnia: EC50 > 1000 MG/L; Fish: LC50, 840 MG/L; Biodegradability: 64% Biodegraded in modified sturmtest; September 1987/HV.

J. R. TAYLOR: Has any work been carried out on the effect on azeotropes in stoving finishes due to the statement

that PnB has lipophilic characteristics?

J. SPAUWEN: Laboratory evaluation with respect to azeotropic evaporation of PnB and other solvents still has to be started.

Advances in the science and technology of pigments by Dr B. L. Kaul

J. R. TAYLOR: The Chemistry of Pigment 155 shows esters in the pigment structure, do these groups give better wettability or give crystallization in organic solvents?

B. L. KAUL: Pigment Yellow 155 is indeed easy to disperse and, because of its total insolubility, does not give any crystallization in organic solvents.

Advances in the jet printing/inks by W. G. Erskine

K. MEGURO: Is it possible to write the complicated characters of Chinese writing (which is of considerable importance to the Japanese/Chinese market) by the jet

printing system at high speed and low cost?

W. G. ERSKINE Japanese companies have been studying this problem for many years and in spite of advances in printing technology have not to my knowledge developed a low cost high speed printer for the full Japanese or Chinese character set. To print Japanese or Chinese characters a good or very good print resolution in terms of dots per mm is required. Even the simpler Japanese language Katakana may require a dot matrix of 22 dots vertically and 16 dots horizontally. This can be printed by continuous ink jet printers at high speed providing that special software is available. The Chinese language requires even better resolution and while continuous ink jet printers can be modified to meet this resolution, it is unlikely that the printer would be considered to be "low cost". Depending upon the print head, certain drop-on-demand printers should be able to print Katakana but at low speed. Again it is a matter of resolution having to compress a large number of nozzles into a line of length equivalent to the height of a character. Alternatively the row of nozzles can be slanted with respect of the characters but this in turn reduces the printing speed.

reviews

Optical Properties

J. Semple

Module 4141 of an HNC course in science. The Borough School of Surface Coatings. Faculty of Continuing Education. Polytechnic of the South Bank.

£39 all learning material including BBC software. £84 as above with marking services and access to laboratories.

This is an example of "distance learning" a form of off-campus education more familiar in the guise of the Open University, (although O.U. differs in requiring no qualifications for entry). The O.U. uses television and radio to present lectures to a national audience as part of degree courses in subjects with national appeal and to large numbers of students. In the narrower and less popular field of surface coatings, less elaborate methods are necessary. In this case, audio cassette tapes carrying the lectures are supported by printed text, and computer programs generate explanatory 3-D graphics. Perhaps a more elaborate form may develop which uses video cassette recordings.

The course is self-contained in a hard backed ring binder with about 150 text pages, and a nest for eight cassettes. The text divides into five sections: intro-

ductions to distance learning(4p), and to the module(20p), a block of stationery, and theory(67p) and practical(29p) blocks. The cassettes carry introduction to distance learning, and to the course(0-1), text of lessons(2-5), commentaries on computer programs (6), while the programs themselves are on tape 7. The alternative of a disc of programs is available.

The programs are written to operate on BBC machines, ranging from the Electron, through 'B', Compact, and Master, to Archimedes. Display can be on standard (color) TV sets, but monochrome and color monitors give better detail, and color enhances the presentations. They can be used on an IBM machine under MS-DOS or on the popular Amstrad PC 1512 by the additional purchase of the language disc 'BBCBASIC(86)'. The use of a green monochrome monitor produced no problems, the colors of the original being shown as various (distinctive) depths of green, although a demonstration of additive colour mixing could not be appreciated.

In the course sent for review, the first audio cassette gave a much higher recording (almost studio) quality than the others, the female voice being completely clear, and no words were missed. In contrast the lecture tapes, recorded by a male voice, needed much more concentration and occasional replay to ensure correct understanding. There was also some evidence of intrusive background noise and voices.

There were no problems with the programs provided on disc, all loaded and 'ran' satisfactorily. Lack of a suitable computer cassette recorder prevented testing of the programs on cassette.

The lessons or lectures are controlled from the theory block in the text, and each starts with a list of objectives. "At the end of this you should be able to . . ." followed by a list of questions of the type "Explain what is meant by a uniform colour space". The audio cassette is played while following the text, which displays diagrams and also carries logos referring to the use of exercises and various types of assessment. The logos are PAX, practical attendance exercise, SAQ, self assessment question, CMA, computer marked assessment, and TMA, tutor marked assessment.

There are 18 PAX, 13 being computer based, the rest laboratory. SAQ's are spread through the audio tapes, a question is posed, the tape is to be stopped while the answer is written down, and the answer follows on restarting the tape. CMA's follow lessons 1, 6, 10 and 14, contain 10, 40, 20 and 20 questions. One of five possible answers is selected (A-E) and entered on a CMA form provided and sent to the Poly, where it is 'marked' and the result returned to the student. Two TMA's are indicated, after lessons 10 and 14. These are question papers set by the tutor, answered under Poly invigilation, and marked by the tutor. No directions were found to the sitting or dating of TMA's.

reviews

It is not proposed to examine in detail, the contents of the lessons, but to list their titles. The first is a revision of basics, which ends with a CMA, and ensures that the student's basic knowledge is sufficient to support the ensuing course. Seven lessons on colour follow: Illumination, The eye, Colour vision, Visual colourimetry, Instrumental colourimetry, Trichromatic colourimetry, The CIE system, Colour matching, Uniform colour spaces. The series is completed by four lessons on the optics of paint films: Hiding power, The Kubelka-Munk equation, The concept of gloss, Gloss measurement. The title "colour matching" could be altered since the subject covered is not related to the prediction of colorant recipes.

After working through the course the student should have acquired a soundly based knowledge of the fundamentals of colour science, and be able to use this in work on surface coatings needing the application of colour technology. A large organisation could use the course as an integral part of training, and supply the local counsellors required, as well as providing computer facilities, and the smaller company may be able to use the system in the absence of formally organised training. The availability of low cost Electron computers, linked to portable or second hand TV sets, reduce the capital cost involved in setting up. Only individual students in small firms may be less likely to benefit, on the grounds of cost and lack of local supervision.

It is appreciated that the course is

designed to follow the examination syllabus, but this is necessarily restricted to fundamentals. The addition of a section on applications emphasising the main industrial uses of the subject in the formulation of colorant recipes, the setting up and use of pass-fail tolerance limits and the correction of off-shade material, would provide a rounding off and whet the student's appetite to apply the new found knowledge.

D. A. Plant

Industrial Solvents Handbook

E. W. Flick (ed)

3rd Edition Noyes Publications 1985
xxiii + 648pp, \$86.00

Revised and expanded, this third edition of the handbook follows a similar format to the second edition (edited by Mellan, but not to be confused with his 'Industrial Solvents'). It consists mainly of tables of solvent properties, taken usually from the manufacturer's literature. Since the methods used to measure these properties are not always given, comparison of values for different solvents could be misleading. For some solvents, there is a general description of main uses but often there is little indication of suggested or recommended applications. The foreword states that one purpose of the book is to enable a formulator to select a new solvent when an

existing one is not available or is prohibited, but there is no guidance on how to make such a substitution. A trade name index would have been a useful addition. 82 references.

S. C. Haworth (PRA)

Organic coatings science and technology Vol 8

Edited by G. D. Parfitt and A. Patsis
Marcel Dekker Inc (N America) 1986
552pp, \$107.50

This is the 1986 publication of the 1984 papers presented at the 10th International Conference in Organic Coatings Science and Technology held in Athens. Its advantage is that these conference papers are now generally available, as compared with the original proceedings, which were only available to the delegates. As usual, most of the papers are useful contributions, edited since their presentation, representing the state of the art at the time of writing. For example, this volume contains one of the first published papers on group transfer polymerisation. Unfortunately, there will no doubt be considerable confusion due to the differing numbers between the conference proceedings and this later published version, with their very similar series titles. Despite the high price, this would be a useful addition to a library.

S. C. Howarth (PRA)

occa meetings

Bristol Section

Lectures for the first half of 1987/88

The First lecture in the series was given by Mr J. Pymmer of Neville Clarke Ltd management Consultants of Swindon on the BS 5750 Organisation Requirements for Quality Assurance. Mr D. S. Newton was in the Chair.

This important specification for quality assurance is now being universally studied by British Industry and Mr Pymmer gave important aspects of the requirements necessary to acquire certification to this specification. Every step in the production of an article in a factory must be examined to assure that no difficulties can arise in the quality of the final product. Training of staff to be aware of the problems is essential and his company run courses to give such guidance.

The documentation necessary to obtain approval can be considerable. When a company has been approved to Regulation of BS 5750 it will be allowed to display the

registration mark on letterheads etc. The certifying body for the specification must be accredited by the Government in the NACCB.

The lecturer answered many questions from those present.

The Second lecture in the series was on the 27 October, 1987 and was the Ladies' Lecture. This was of general interest and well attended under the Chairmanship of Don Newton and held as usual at the Bowl Inn, Lower Almondsbury, Bristol.

The lecturer, on this occasion, was Mr B. W. Beckett, the Bristol Trading Standards Officer, well-known for his appearances on Bristol HTV regarding Trading Standards.

He showed general excerpts from the HTV series during his lecture. He discussed the origin of his department from just interest in Weights and Measures etc, to the present day when the whole aspect of manufactured products may come under his terms of reference.

Items of general interest include the need to modify the design of the longer-life alkaline batteries as his Department had received complaints that the central electrode could, under certain circumstances, be blown out of the cell, with considerable force. Modifications to avoid any build-up of pressure during use were instituted, but the original valve designed to overcome the problem tended to block and when he demonstrated the modified products on TV the cell in question propelled the electrode across the studio. Further modifications to these cells have now been made and the electrode should remain within the cell during use.

Other problems shown included the usual dangerous children's toys, dolls, etc, which could easily come to pieces, allowing the child to come in contact with sharp pins and other dangerous objects. He showed an HTV video tape of a folding dolls pram which could collapse and trap the fingers of the child and had, in one case, taken off the tops of the fingers of a small child.

The Department now looks after problems with mortgages and other housing problems. Some electrical appliances were also shown to be dangerous on the HTV video tape.

In the future, the Trading Standards Offices throughout the country will be closely linked to ensure that early warning of problems can be made known quickly to all regions.

Many questions were answered by the speaker and the evening concluded with an excellent buffet.

The Third lecture was on 24 November, 1987 when Mr Brian Gregory of Tioxide Ltd read a paper on 'The Effect of Flocculation in Coatings'.

The lecturer discussed the effect of flocculation of TiO_2 on increased costs, decrease in opacity and covering power of a coating. In this respect, the use of thixotropic alkyds can give increases in flocculation and costs of decorative products unless great care is taken in the formulation.

The effect of extenders on the flocculation gradient, a subject documented by Tioxide Ltd previously, in particular the effect of extender particle size was clearly demonstrated. Very fine extender particles can flocculate around the TiO_2 particle, whilst extenders having similar particle size to the TiO_2 pigment have a neutral effect.

Flocculation in emulsion paint systems with different polymers can show porosity in the dry polymer film and thus voids in the dried film.

Other important effects of flocculation are settlement, colour and gloss, and these can have an effect on the reduction of durability of a film due to increasing breakdown of polymer as a consequence of photochemical reaction. Examples of the flocculation effect on Contrast Ratio were shown.

The lecture was well received and thanks expressed to

Tioxide Ltd for the provision of an excellent buffet at the close of the meeting.

J. R. Taylor

Manchester Section

Aluminium organics

The first meeting of the new year was held at the St James Club, Manchester, on Monday, 11 January, 1988, and was attended by 50 members and guests. A paper entitled "The Application of Aluminium Organics in Surface Coatings" being presented by Mr D. J. Love of Manchem Ltd, a member of the Manchester Section Committee.

The lecturer initially outlined the areas in which aluminium organics are used, these being: rheology, pigment wetting, cross linking and water repellants.

The basic intermediates used are the aluminium alkoxides, which are very reactive compounds, reacting with: water, alcohols, phenols, carboxylic acids and chelates to give polyoxo compounds, the resulting commercial products being described.

Each area in which aluminium organics are used was outlined in detail.

Simple aluminium acrylates with free hydroxides give gellation in hydrocarbons and their use as anti-setting agents was outlined.

Simple gels of this sort also have a limited use in greases and solvent gels, but are of little value in other fields. More complex compounds are used in printing inks, and these were described, the importance of temperature control was emphasised, as too high a temperature ($200^\circ C$) structure loss can occur due to different chemical reaction taking place. The overall advantages in inks were described, and the problem of local gelling and seeding, and the steps required to minimise this outlined.

For cross-linking purposes, Aluminium ketoenolates have to be used so that the reaction is not too fast, they are used to impart drying properties in very low viscosity high solids alkyd systems, and the reactions involved were outlined. These systems have better through dry, higher final hardness, and better weather resistance, than coatings using cobalt drier.

In water repellants, polyoxoaluminium stearates are used, the primary areas being masonry, timber and textiles. The comparison between a water repellent and a pore blocker was illustrated, and their detailed use on masonry and timber discussed.

A Zinc/Aluminium complex is used in the field of pigment wetting, shorter dispersion times, and higher pigment loadings are possible, the advantages being primarily found with inorganic pigments and extenders.

A short question and answer session was followed by a vote of thanks proposed by David Clayton, the session

being concluded with a buffet courtesy of Manchem Ltd.

M. Langdon

Midlands Section Trent Valley Branch

Confessions of a small paintmaker

The first meeting of the season entitled "Confessions of a Small Paintmaker" was presented by David Penrice of Newtown Industrial Paints on 23 October, 1987, and kindly sponsored by Via Gellia Colour Co Ltd.

Forty-five members and guests enjoyed this highly amusing talk and slide show which, amongst other things, gave some insight into the setting up and running of a small paint company.

Envious glances were noticeably cast by the audience at pictures of the bike and hardness tester in particular but not forgetting all the other items of laboratory and office equipment which received similar audience approval.

It was later commented that the scantily dressed young lady appearing on most of the slides may well have added to people's enjoyment, but it is questionable whether it is possible to enhance views of paint test equipment and the like. A lively period of questions followed which predictably centred around finding out Fifi La Belle's address and telephone number.

This excellent evening was suitably rounded off with a vote of thanks from Derek Grantham and warmly approved by all present.

Basic wood science and decay control with surface coatings

The second lecture of the current season entitled "Basic Wood Science and Decay Control with Surface Coatings" was presented by Mr Neil Tasker, Product Manager of Buckman Laboratories on 26 November, 1987.

Accompanying Mr Tasker was a strong contingent from Buckman who added further weight to the question/answer session. We are further grateful to Buckman Laboratories for their generous sponsorship of the whole evening which included a buffet before the lecture.

A good sized audience were treated to some of the basics of wood science which differentiated between the fungi that colonize wood and additionally reference was made to moulds, stains and fungi that cause decay. The characteristics of fungi and the environmental requirements of wood deteriorating fungi were also outlined.

White rot, brown rot, soft rot, dry rot, their causes and cures were illustrated with an in depth discussion of decay prevention. Comparisons were made of the effectiveness of commercially available preservatives which included TCMBP vs TBTO, Zinc Napthenate, and PCP, the TCMBP showing excellent results. This was further

strengthened with evidence of low oral and dermal toxicity an obvious bonus in today's concerned industry.

Mention must be made of the question/answer session which followed the lecture. There was so much interest in the subject from the audience that questions were fired for a good 45 minutes afterwards and probably represents something of a record for Trent Valley Branch.

The evening was brought to its conclusion with our usual vote of thanks from Jonathan Bourne of Mebon Paints.

History and development of Mebon Paints

The last meeting of 1987 was a social function held at the splendid new Technical Centre of Mebon Paints and included a most interesting insight into their history and development, the presentation being made by Mr John Bourne co-founder of the company.

Twenty-five years of Mebon's history was expertly encompassed with an impressive audio-visual display which complemented John's talk. The emphasis on technical excellence and well defined marketing strategies were as evident in the beginning as they are today, and everyone present I am sure, felt the pride generated by watching the company grow so successfully over a relatively short period.

We were thankful to have with us Mr Pacey-Day, our new General Secretary, who witnessed a most enjoyable part of the evening when a wall clock and tankard were presented to John Ellis in recognition of his many years of hard work and loyal service to Trent Valley Branch.

A cheese and wine buffet followed the more formal part of the evening and Derek Grantham [Via Gellia Colour Co] proposed the vote of thanks to John Bourne and Mebon Paints for their most generous sponsorship.

M. J. Hannah





FROM THE GENERAL SECRETARY

Priory House at Your Service

It is traditional of learned societies such as OCCA that the General Secretary is responsible for signing correspondence, is the primary telephone recipient and generally is the point of contact with the membership. However, in practice the role of General Secretary is very different, and in order to fulfil my responsibilities to Council in the administration of the Association, to service Council and its Committees and to promote and develop the Association, I must rely on the support of the other staff at Priory House. Indeed, without such staff I would be unable to fulfil my duties.

The Association is well served by its staff who maintain a most cost-effective level of service and activities and comprises fewer staff than other comparable bodies. Who then are the staff at Priory House and what is their role?

Yvonne Waterman, Senior Secretary and assistant to the General Secretary, deals with all matters relating to Council and its Committees and acts as principal contact with the President and Honorary Officers.

Peter Fyne, Assistant Editor, is responsible for the production of the *Journal* and other Association publications. Peter works closely with the Honorary Editor and during the year will be expanding his activities to include company visits and attendance at Section meetings and symposia.

Frank Dixon Craik, the Association's Advertising Manager, is spear-heading the promotion of the *Journal* and the drive for additional advertising revenue.

Hilary Pooley and Pam Stringer, provide secretarial services for the *Journal* and they also deal with subscriptions and sales of all Association publications and ties.

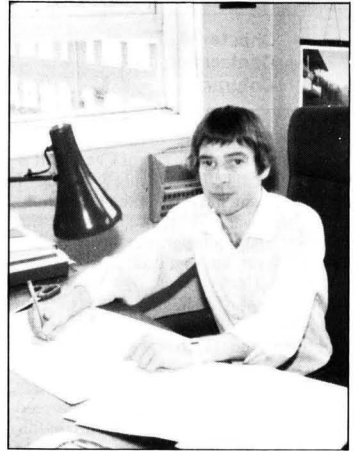
Irene Jenkins, maintains and administers the financial records of the Association. Irene also provides catering for Committee meetings at Priory House.

Jacqueline Raynaud, maintains the Register of members and deals with routine correspondence with the Sections and the election of members.

I am, of course, always pleased to talk to members on any matter relating to the Association, but I would encourage you to make direct contact with the appropriate member of staff, all of whom are willing and able to help you.



Yvonne Waterman



Peter Fyne



Frank Dixon Craik



Hilary Pooley and Pam Stringer



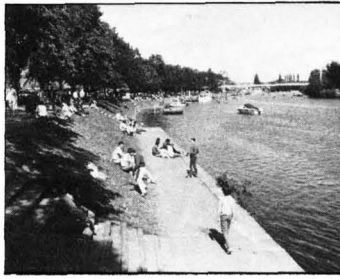
Irene Jenkins



Jacqueline Raynaud



Adjacent is a picture of the River Dee which runs through the city of Chester. It is on this river that the evening trip aboard the Lady Diana boat will take place during the Conference. The cruise will take the delegates right through the heart of the Duke of Westminster's estate.



Eastgate Street, Chester

John Taylor is having a reasonably successful time in collecting together the papers for the Conference. Apart from asking for authors he has received a number of unsolicited offers. As soon as we have the first eight papers confirmed beyond doubt we will list them in this column. However, we expect this should not be so far off, especially as a result of the "Call for Papers" insert in January JOCCA. If for some reason you need an extra copy of the insert please contact Priory House.

We will be interested to see how much enthusiasm there is for having a mini-exhibition at the Conference. The Chester Grosvenor is quite amenable to having rooms set aside for this purpose.

A. C. Jolly

new members

Ordinary Members

- Albert, D., BSc. (Ontario)
- Baines, P. F. (Manchester)
- Beedle, H. V. A., BSc. (London)
- Brown, A. (Newcastle)

- Emerson, D. M., MSc. (Ontario)
- Farrell, P. J. (Newcastle)
- Fletcher, R. J., BSc. (West Riding)
- Flynn, J. A., BSc. (Thames Valley)
- Goodman, J. H. (Manchester)
- Greaves, E. O., Ph.D., MSc. (Manchester)
- Hammett, D. (Ontario)
- Hardjasa, F., BSc. (Ontario)
- Haworth, P. D. (Manchester)
- Jolly, B. S., BSc. (Newcastle)
- Kidd, G. (Newcastle)
- Latchman, P., BSc. (Ontario)
- Loh, W. S., MSc. (General Overseas-Singapore)
- Mustafa, I., Ph.D., MSc. (London)
- Pitt, J. G. (Ontario)
- Rayner, J. (West Riding)
- Rogerson, M. (Newcastle)
- Songhurst, D. (Ontario)
- Vardi, S., MSc. (Transvaal)
- Viljoen, D. J., BSc. (Cape)
- Wall, P. V. G. (Manchester)

Associate members

- Dunn, A. A. (Cape)
- Marshall, D. D. (Midlands)
- Naidoo, G. J. (Transvaal)

Registered Student

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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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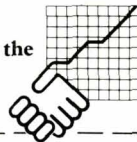
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