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**JOURNAL OF THE
IL &
COLOUR
CHEMISTS'
ASSOCIATION**

*OCCA 60th Anniversary
Commemorative Issue*

From Gold to Diamond. OCCA 1968-1978

By S. H. Bell



The application of linear programming to paint and resin formulation
P. E. Kavanagh

Microanalysis of marine antifouling paints in the scanning electron microscope—its automation and application to less homogeneous paints
R. J. Bird and D. Park

The mathematical relationship between the viscosity of PVC plastisols including fillers and plasticiser number of fillers
J. Wypych and J. Walczak

A new method of determining the dispersibility of pigments and the optimal mill base formulation
J. Oyarzun

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JOURNAL of the OIL AND COLOUR CHEMISTS' ASSOCIATION

Hon. Editor: S. R. Finn, B.Sc, FRIC, FTSC

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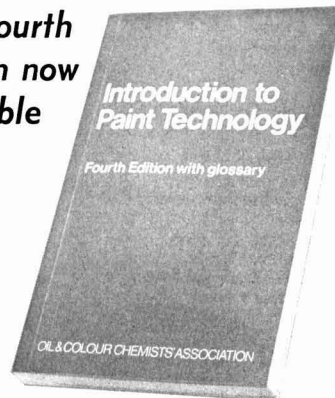
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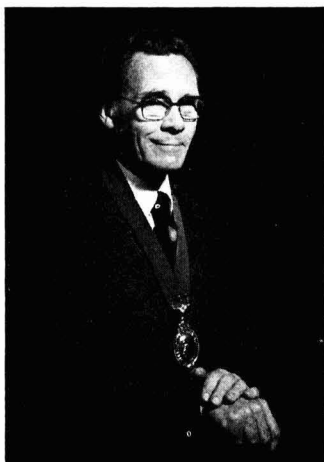


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Message from the President of the Association



Although—or, perhaps, because—we live in times when the organisation seems progressively to submerge the individual, there is a constant need to remind ourselves that organisations exist for people and that, in the last resort, they are people.

But the individual in isolation is equally anomalous. All meaningful existence involves relationships—with people, not with things. And no Learned Society, such as our own Association, can claim to be a live and creative thing unless real relationships are forged between its members.

In this, we are peculiarly fortunate in OCCA and I have been particularly aware of it in my first year as President. In that time, I have had the good fortune to visit all of the Sections in the UK and in Ireland, and it has been a great inspiration and encouragement to have seen, at first hand, the spirit of dedication which is so characteristic of OCCA and its members. Service is so willingly given at all levels; in Council, in Committees of Council and, above all, within the Sections themselves, where the real heart of OCCA beats.

In this year, which marks the 60th Anniversary of the foundation of our Association, I can look back over a membership which spans exactly half of this period. It is a period which has seen notable progress and many important changes. But, it is one in which, above all, people and personal friendships are the things which I shall most clearly recall and which are so characteristic of OCCA.

The 60th Anniversary affords me an excellent opportunity, as President, to offer my sincere thanks to all members who have contributed, in any way at all, to the advancement of our Association to the position of prestige and high esteem which it enjoys throughout the world.

I extend a warm welcome to all who attend our 60th Anniversary Celebration and trust that you will not only enjoy them, but will recall them with pleasure for many years to come.

East Kilbride,
Glasgow
May 1978

Angus M. Lean

Transactions and Communications

From gold to diamond. OCCA 1968-1978

By S. H. Bell, OBE, PhD, FTSC (President 1965-67)

The sixtieth year of our Association happily coincided with the Silver Jubilee year of Her Majesty Queen Elizabeth II, to whom a loyal message was addressed.

Introduction

Attempting to chronicle briefly the wide-ranging activity of our Association at home and overseas during the momentous years between our Silver and Diamond Jubilees has proved difficult but absorbing. The selection of events and developments for special emphasis and comment in this survey inevitably reflects the judgement and attitudes of the author. It is hopefully acceptable to the reader as a fair conspectus of changes, accomplishments and trends. However, the author has not presumed to select individuals for special mention later in the text from among the many who have made it all possible** except those whom the Association has itself honoured for exceptional past services to the Sections or to the Association as a whole.

But why the *Diamond Jubilee*† celebrations? Certainly not just because we have survived for sixty years, though it is noteworthy that we have remained without substantial change or amalgamation, as has proved necessary or appropriate for some associations through economic pressures or changing technology. It is because we find ourselves in good heart after a difficult period and ready to pause for a while, accepting the opportunity of a major anniversary to survey the past, to remember with gratitude our founders†† and many stalwarts active over the years, and to look to the future of our world-

*Major contributions are, of course, made by Honorary Officers, Council, Section Officers and Committee members, whose names are given year by year in the January issues of the Journal. Here appended are the names of the principal Honorary Officers of the Association during the period reviewed:

Presidents

-1969, F. Sowerbutts	1973-75, L. H. Silver
1969-71, A. Fraser	1975-77, A. T. S. Rudram
1971-73, the late A. W. Blenkinsop	1977-, A. McLean

Hon. Secretaries

-1969, the late J. C. R. Bews
1969-74, D. S. Newton
1974-, D. J. Morris

Hon. Treasurers

-1970, the late A. W. Blenkinsop
1970-76, F. Cooper
1976-, Dr H. R. Hamburg

Hon. Research and Development Officers

-1969, A. T. S. Rudram
1969-75, A. R. H. Tawn
1975-, C. N. Finlay

Hon. Editors

-1969, A. R. H. Tawn
1969-, S. R. Finn

**Many past and present Honorary Officers, Section Chairmen and Council members attend the Council Reunion Dinners, at which the reigning President outlines important developments, continuing the traditions of the Past Presidents' Dinners, of which the first was held in 1938.

†During the last few years major anniversaries have been celebrated by a number of associations founded in the years following the first World War. There was especial pleasure in the OCCA presentation of a congratulatory Scroll to the Paint Research Association on their Golden Jubilee in September 1976, five OCCA Presidents having served on P.R.A. staff.

††With the death of H. R. Wood in July 1977 at age 90, there are now no surviving founder members.

renowned Association, having especially in mind those younger members on whom that future so much depends.

The Golden Jubilee onwards

Where were we at the time of the Golden Jubilee? The first fifty years from our foundation in 1918 were surveyed in detail in *A Fascinating Story*, the final section of which recorded the main features of the *Forward Thinking* exercise launched in April 1967 which aimed, by seeking answers to searching questions, to assess "where we stand at present, how we got there, and . . . how to increase our readiness to meet new challenges . . . in a world which is rapidly changing industrially, scientifically and technically". The numerous questions included those about Council and Committee structure, range of membership, technical training, the *Journal* and its coverage and, indeed, almost all our activities; and a reiterated challenge—should OCCA become a qualifying or even an examining body?

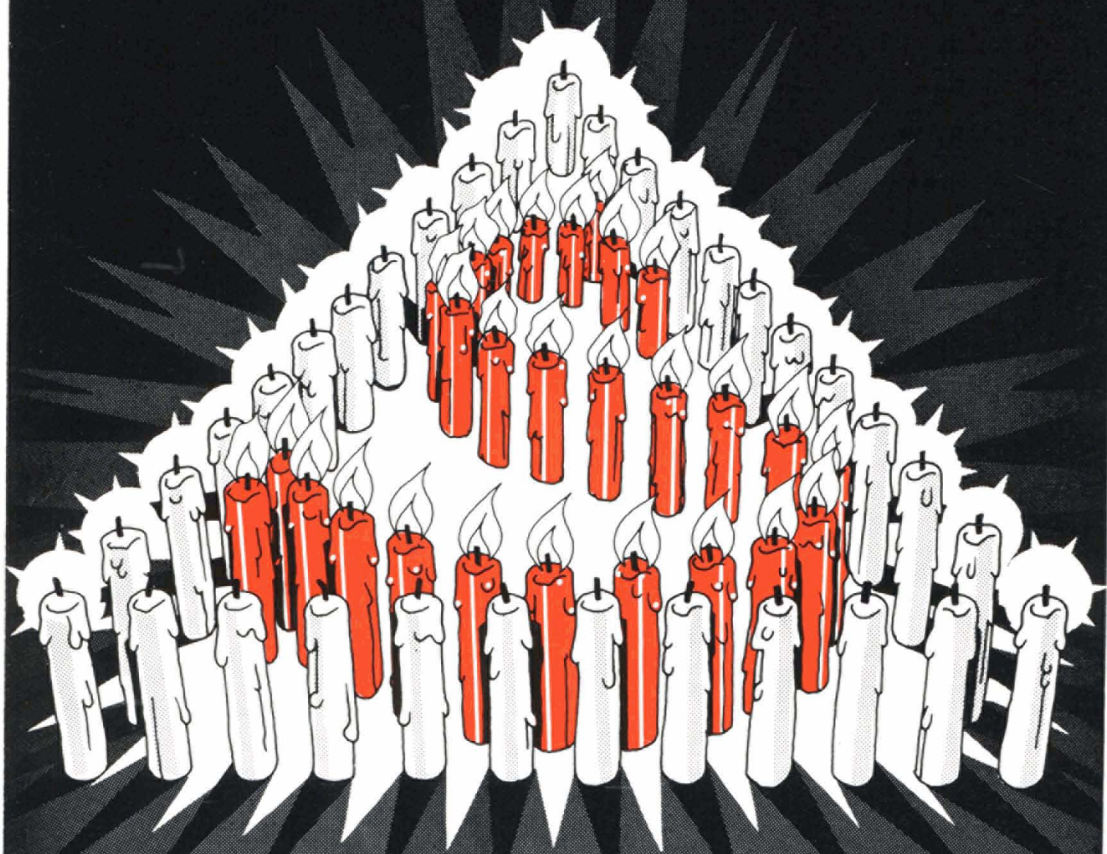
There was an immediate response with intensive consideration by Council and all Sections. In November 1967 a working party was formed to coordinate the views expressed, leading in February 1968 to Council's acceptance of proposals for setting up the *President's Advisory Committee* and other changes in Council and Committee procedures and in membership grading*, and surveys by some Sections provided information about age distribution among members, their daily work and technical interests, acceptability of technical and social programmes and much else of value in future planning.

O.C.C.A.—A Qualifying Body

Meanwhile, a working party on *Education, Training & Qualifications* had been formed in October 1967 to examine the many views on OCCA becoming a qualifying body; the pros and cons, hopes and fears, possible ways and means. By the autumn of 1971 a way had been found whereby "the desire of members to obtain an educational identity with the industries covered by the Association" could be achieved whilst still maintaining the established character of the Association, without (as it was formally expressed) "in any way amending the Memorandum & Articles of the Association or its name". Within months it was clear that there was widespread support among members and by January 1972 there were 103 successful applicants for the new "Professional Grade".

There are detailed Regulations (see eg. *JOCCA* February 1978) designed to establish sound standards and high status

*These included a new procedure for nominating the President Designate, reconstitution of the Technical Committee, revised terms of office for Honorary Officers and members of Committees of Council; also the change from "Junior Member" to "Registered Student".



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OCCA
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for these qualifications at home and overseas*. They are strictly applied by the *Professional Grade Committee*, taking account of the candidate's training, scientific or technical qualifications, publications, experience and technical responsibilities etc. at various levels for the Licentiate, Associate and Fellowship in the Technology of Surface Coatings. A technical dissertation and *viva voce* examination are required for all candidatures for the Licentiate and may be required in other cases.



A view of the Association's Information Centre at an OCCA Exhibition with one of the seating areas in the foreground

At December 1977 there were 440 members in the Professional Grade from 39 countries. Naturally, at the outset there were many members with well-established qualifications and experience who fulfilled the requirements for the Fellowship or Associateship. It is now pleasing also to see younger people coming forward for the Licentiate, and to note that OCCA liaison with Technical Colleges has resulted in registered students being given guidance in the preparation of their dissertations. By December 1977, 15 Licentiateships had been awarded, 9 applications had not been accepted, and 22 awaited fulfilment of the Regulations. Four of the earlier of those 15 Licentiates have since been upgraded to Associates.

The Professional Grade is steadily growing. It already covers over 15 per cent of the total *Membership* of about 2,800 which, although now increasing, remains less than the 1968 figure of a little over 3,000 after a period of severe economic pressures and declining manpower in many parts of industry. There has, however, been a steady and striking rise in overseas membership from 750 to over 1,000, almost wholly due to growth in overseas Sections and Branches including over 90 members in the new Ontario Section.

The Registered Student category (at low subscription rates for those under age 21 and up to 25 if following courses of tech-

*In *JOCCA* February 1977 there appeared the first advertisement for staff in industry mentioning the Professional Grade as a qualification to which preference would be given.

As reported in *JOCCA* April 1978, the Council of the Paintmakers Association of Great Britain Ltd has passed the following resolution:

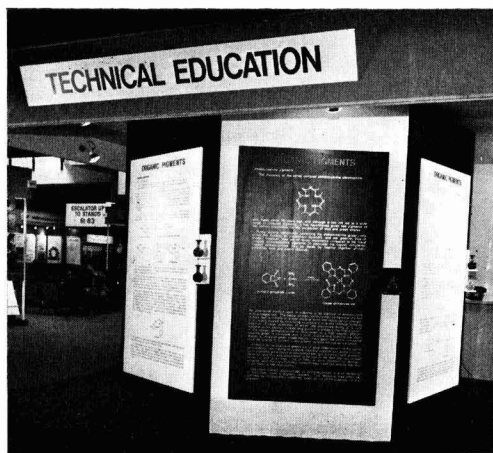
"The Council of the Paintmakers Association of Great Britain recognises the need for maintaining scientific and professional standards within the Industry and recommends Members to encourage their scientific and technical staff to apply for admission to the Professional Grade of the Oil & Colour Chemists' Association and to support the aims and activities of the Oil & Colour Chemists' Association."

nical training) has declined from 170 to only 50, thought to be due mainly to the changed pattern of training and industrial recruitment. Many more young people continue full time education into their early twenties and those suitably qualified are taken straight into Ordinary Membership. The Association owes much to the good offices of established members in stimulating interest among their younger working colleagues as well as through the special efforts of Sections and Branches.

Encouragement of the younger scientists and technologists was very much in the mind of the late Mrs M. R. Jordan when endowing *The Jordan Award* in 1967 in memory of her husband Dr L. A. Jordan, a Past President 1947-49 and an Honorary Member. It is awarded every two years for the best contribution to the science or technology of surface coating by a member of any nationality working in either the academic or industrial field who is under the age of 35. There have been seven recipients, the award having twice been shared, and the wide technical coverage has included film formation and optical properties of paints, milling of printing ink, pigment flocculation and opacity, and lacquer cracking; all but one of the papers appeared in the *Journal*, the other award having been made for a confidential technical report.

The Technical Exhibitions

At the beginning, in 1949, fourteen "exhibitors" sat at tables at the Borough Polytechnic, London (well known for its long and admirable tradition of technical training), "to discuss technical problems and offer advice" because "juniors, in particular, had little opportunity to meet the technical representatives of raw material suppliers". No one could have dreamt, much less expected, that, thanks to the work of successive *Exhibition Committees* and the organising flair of our professional staff, the annual Technical Exhibition would become a major international event, and one of considerable significance for OCCA finances.



For many years the Association ran and organised a Technical Education stand at the Exhibitions

The opening years of the period now reviewed saw continued development. In 1969 exhibitors' demands became so great that in some cases less space could be allocated than they had requested, and it was then decided (assisted by some uncertainty about the future of Alexandra Palace) to move to

the larger and more central facilities of Olympia, London. There, in 1970, "the most successful Exhibition" to that date attracted some 14,500 admissions, with visitors from 38 countries. There was a similar success in 1971.

Then followed downs and ups, for such a venture involving so many companies and countries inevitably reflects national and international economic climates and recurrent crises. Also there were sometimes "little local difficulties" such as "work-to-rules" by staffs of exhibition contractors and public transport. There was a fall-back in 1972, an upsurge of interest for the Silver Jubilee Exhibition in 1973, then described as a "resounding success"; but in 1974 there were the adverse effects of a U.K. economic crisis with its three-day working week which followed a period of acute raw material shortages. Some other exhibitions were cancelled or severely reduced.

Thereafter progress was renewed. There was the return in 1976 to the refurbished Alexandra Palace where the facilities and ease of access have been greatly improved; and so to the highly commended 1977 Exhibition having over 70 stands representing 120 organisations from 16 countries: Australia, Belgium, Denmark, East Germany, Holland, Hungary, Italy, Norway, Poland, Rumania, Spain, Sweden, Switzerland, U.K., U.S.A. and West Germany*. Over 10,000 admissions were recorded at the entrance hall from 50 countries for this "continuous dialogue between suppliers and manufacturers" on "new advances in raw materials, plant and equipment for the paint, printing ink, colour, adhesives and allied industries". The information leaflet was printed in six languages. Remember those tables at the Borough Polytechnic?

For various reasons, including the numerous commitments of exhibitors and visitors during a very busy week, the opening Exhibition Luncheons and their successors the Exhibition Dinners were discontinued; but on the 1977 opening day, after a buffet luncheon at Alexandra Palace, the Committee conducted the principal officers of other societies and of research institutes, and Members of Parliament around the Exhibition†.

The Association Conferences: International liaison

Except for the war years, our biennial Conferences have an unbroken history since the first at Buxton in 1937, and in the

post-war years they increasingly became major international events, technical and social.

Five were held during the period now reviewed* and the successive Honorary Research & Development Officers and their *Technical Committees* deserve commendation for their forward-looking policy in subject choice and in framing the programmes of lectures and discussions, and a variety of "workshop sessions". Much background research has been reported and there has been continuous attention to the efficient use of established and new materials, improving methods of product control, testing and application, and increasing attention to environmental, safety and health problems. Throughout, there has been stimulation of well-grounded speculation for the years ahead, emphasised in the choice of the 1973 Conference subject—"Towards 2000". At the most recent (1977) Conference the truly international character was again evident, with delegates from the U.K. and ten overseas countries taking part in exceptionally lively discussions on the topical theme—conservation of resources.

There has also been the desire to bridge that gap, which has frequently been the subject of comment, between the severely academic and the severely on-the-job practical. Lecturers have been drawn from the universities, technical colleges, governmental and other official bodies, research institutes, trade bodies and of course our own and associated industries. Lecturers and audiences have come from many countries including official representatives of our fellow associations within the *International Alliance*—the U.S. Federation of Societies for Coatings Technology (*FSCT*), the Federation d'Associations des Techniciens des Industries des Peintures, Vernis, Emaux et Encre d'Imprimerie de l'Europe Continentale (*FATIEPEC*) and the Scandinaviska Lackteknikers Forbund (*SLF*).

Wherever possible, our Presidents have accepted invitations to attend the Conferences of those bodies, and papers have been presented on behalf of OCCA in Boston, Chicago, Atlanta, Los Angeles, Houston and Washington (*FSCT*); in Brussels, Montreux, Florence, Garmisch Partenkirchen and Cannes (*FATIEPEC*) and in Copenhagen, Sandefjord and Helsinki (*SLF*). Our President was pleased to present a congratulatory scroll at the Fiftieth Anniversary Meeting of *FSCT* in October 1972 at Atlantic City.



Delegates attending a lecture at one of the Association's recent Biennial Conferences

*The April issue of *JOCCA* showed that at the 1978 Exhibition (OCCA-30) there would be 145 organisations from 16 countries represented.

†It is interesting and significant that, during the period reviewed, invitations as guests of honour at the Luncheons and Dinners were accepted by:—the Rt. Hon. Lord Erroll of Hale P.C. (1968), Lord Kings Norton (1969), Lord Sheffield, President, Parliamentary and Scientific Committee (1970), the Rt. Hon. Margaret Thatcher P.C., M.P., Secretary of State for Education and Science (1971), Lord Ironside, Vice-President, Parliamentary and Scientific Committee (1972), and Lord Limerick, Parliamentary Under-Secretary of State for Trade (1973).

*The main subject headings have been:—

1969, Eastbourne, "Film formation and curing",

1971, Torquay, "Surface properties and appearance",

1973, Eastbourne, "Towards 2000",

1975, Scarborough, "Performance of surface coatings—does the reality match the theory?",

1977, Eastbourne, "Conservation of energy, materials and other resources in the surface coating industry".

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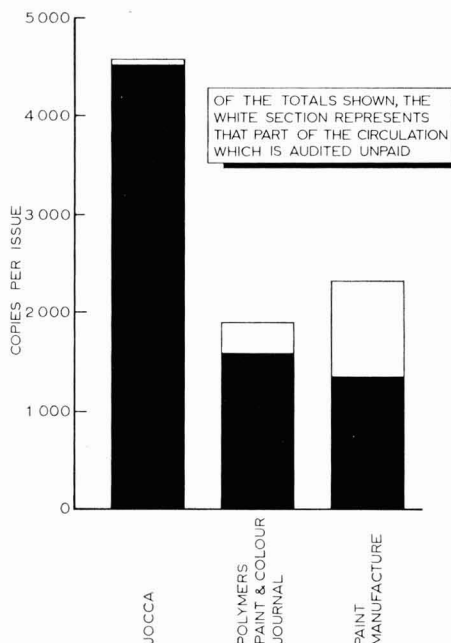
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The London Section has continued the exchange of lecturers with kindred European societies inaugurated in January 1965 in Copenhagen. Such exchanges have been made with Germany and Holland as well as Denmark, and in a further collaboration, members joined with those of the local section of the Association Française des Techniciens des Peinture, Vernis, Encres d'Imprimerie, Colles et Adhésifs in Calais in June 1973 and there was a return weekend visit to Hastings in February 1974.

Close liaison has, of course, also been maintained with our own Sections and Branches overseas. There have been a number of visits by our Presidents and other U.K. members, some of whom have presented technical papers, and overseas representatives have been warmly received at headquarters and at Association events in the U.K.

The Divisions, Sections & Branches

In the latter part of 1975 and early 1976 the *Manchester Section*, the first OCCA Section, enthusiastically celebrated its fiftieth Anniversary. On behalf of the Association, the President presented a congratulatory scroll, and at a special dinner received in return an Address on behalf of the Section. A commemorative booklet was issued and celebrations continued at their annual dinner-dance, a ladies' lecture and supper evening; and, not forgetting the technical side, the Section held a Symposium on "Films—formation and behaviour".

Also in 1976, new *Divisions* of the Association were created in *New Zealand* (Auckland and Wellington Sections) and *South Africa* (embracing the former Branches in the Cape, Natal and Transvaal which then received Section status) in meeting the desire to have organisations "reflecting their status in their own countries". In 1977 the Cape Section was operating an *Eastern Cape Branch* and the Transvaal Section a *Rhodesian Branch*. Also, in 1977 a Nigerian Branch was projected under the London Section. The Association now has 10 Sections and 2 Branches in the U.K. and Ireland, and 6 Sections and 2 Branches overseas, as well as the General Overseas Section. There are continuing close links with *OCCA Australia** formed in January 1968 from the five Australian Sections, through arrangements for their receipt of the *Journal* and representation by the President of the Australian Federal Committee who is coopted to our Council.

The recent increase in the number of centres of interest is notable, for the well-being of our Association is heavily dependent on the vitality of its local units, always remembering that we are bound together by the central organisation and collective activities governed by the Council on which all Sections are represented. Each local unit has its own character, partly dependent on the nature and extent of relevant local industry, but largely created and maintained by regular technical meetings and social events; horizons have been broadened through the increasingly popular seminars, symposia, colloquia, conferences, etc. attracting wider interest, frequently with speakers and visitors from other countries.

A number of successful Section symposia were held in 1977-78 not all yet fully reported, but a survey of the records to 1976 covering 24 such gatherings since 1968, sometimes held in collaboration with appropriate kindred societies and sometimes especially for students, reveals a wide coverage of

developments and trends in the preparation, assessment and use of materials and products. There have been papers and discussions on the characteristics of pigment surfaces, film formation and behaviour, paint properties such as gloss and colour, the life performance of paints, and on printing ink testing; also on acrylics, water-borne systems and ultraviolet and other radiation curing and industrial finishing generally; on paints for microbiological resistance, metal protection and use under immersed conditions, and ships' compositions; on paints for "industrial building" and for plastics; and on "wall-coverings". Environmental problems (atmosphere pollution, health and safety) have received detailed attention and there have been excursions into training and management, and requirements for paint exporting.

The same forward-looking attitude is revealed by the records of the annual conventions in New Zealand and conventions and symposia in South Africa, including, for example, discussions on "powder coatings", "modern trends in coatings and their application" and "environment and the surface coatings industry". The 1971 New Zealand Convention marked their 21st anniversary and included a display of latest materials and equipment. In 1969 the 21st anniversary of OCCA in South Africa was marked by the manning of a stand at the first Chemical Industries Exhibition in Johannesburg "to encourage technical training and attract technically trained personnel to the paint industry".

Stimulating and maintaining interest among younger people is not easy nowadays, and student liaison officers and others involved on Section committees deserve congratulations on their tenacity in arranging numerous special lectures, symposia and other events. Taking just two further and recent (1977) examples, the Manchester Section arranged for a party of 70 to visit the Technical Exhibition and the Scottish Section held a Junior Technologists Symposium on the "Fundamentals of paint technology".

Technical Training

A notable example of encouragement of sound technical training is provided by the London Section with the introduction in 1976 of annual "Leslie Kekwick Prizes" for the best students in our technologies in local colleges*.

Indeed, there is widespread concern among the Sections to encourage registered students and to assist in local technical training schemes, now with the added incentive of OCCA professional qualifications. Much "in-house" training is done by companies frequently linked with external facilities such as the short courses and seminars run by the Paint Research Association and regular, extensive, long-range technical college courses. However, availability of college facilities varies considerably and in a number of areas is non-existent, if only because the potential intake of students from local industry is too small. The new Technician Education Council (TEC) arrangements which can include paint technology (for example) as only one element in broader training schemes, may assist.

The work of our *Technical Education Committee* has continued in liaison with the development of the Professional Grade scheme and in knowledge of the many changes in national and industrial training arrangements. There is OCCA representation on the relevant Training Boards and committees of the City and Guilds Institute, the Scottish Technical

*In September 1977, a paper was presented on behalf of our Association at the First Pacific Convention of OCCAA.

*In memory of the late L. O. Kekwick, Chairman of London Section 1949-51 and President of the Association 1951-53.

Education Council, the Paintmakers Association and the Institute of Corrosion Technology. A recent notable development is detailed cooperation between our Professional Grade Committee and the Training and Technical Education Committee of the Paintmakers Association in respect of the latter's arrangements with the technical colleges for courses satisfying TEC requirements; and at our 1978 Exhibition, stand space was provided for the Paintmakers Association's presentation of such courses to the industry and to potential students. This was in appropriate succession to the technical training displays and information centres which were for many years special features. Many hundreds of school and college pupils have visited our Exhibitions for conducted tours and talks on careers in our various industries.

Association Honours

The Association has continued the happy practice of bestowing honours for notable service, and with so much to commend in the vital work of the Sections, there is particular pleasure in recording the establishment in 1969 of the *Commendation Award* "for members of outstanding and long service to the Association, particularly at Section level". This has been conferred on W. J. McWaters (Bristol), P. B. Hunt (Auckland), T. W. Slinn and A. C. McEwan (Wellington), T. R. Smith (West Riding), G. F. Jones (Irish), Dr W. Carr (Manchester), G. H. Hutchinson (Scottish), K. Engelbert (Natal), O. Rutledge (Auckland), and D. N. Fidler (Bristol).

Also during the past ten years, *Honorary Membership* has been awarded to T. Howard for outstanding service in South Africa, to R. W. Matlack who for many years fostered our liaison with the U.S. Federation of Societies for Paint Technology and to five Past Presidents, Dr H. W. Keenan (1944-47), the late L. O. Kekwick (1951-53), Dr H. A. Hampton (1961-63), F. Sowerbutts (1967-69), and Dr S. H. Bell (1965-67).

Publications: The Journal

Reference to the *Journal*, that major OCCA achievement, comes logically late in this review, for it mirrors month by month all those other activities already surveyed. Praise for its excellence in content and presentation is due to successive Honorary Editors, the *Publications Committee*, our professional staff and the printers.

JOCCA, issued almost from the beginning of our sixty years and in the now familiar A4 format since 1973, has a world-wide circulation of approximately 4,500 copies, with readers in 80 countries and including over 1,700 non-member subscribers. It presents numerous scientific and technological reports from many countries, and is an important news organ and advertising medium. The Consolidated (decennial) Index of Transactions and Communications, 1966-75, recently issued, covers the greater part of the period now reviewed and is of especial value in technical literature searches.

The extent to which the *Journal* has become a world platform for scientific and technological contributions is especially noteworthy. In the ten years 1968-77 there were 468 papers of which 290 came from the U.K. and Ireland. The remaining 178 came from overseas, some through Section meetings, symposia, and Association Conferences, etc., but as many as 138 by direct submission. In one year (1976) from a total of 46 papers, there were 21 from overseas, from Australia, Austria, Belgium, Czechoslovakia, Denmark, Egypt, West Germany, Holland, New Zealand, Switzerland

and U.S.A.; and there was one of joint U.K./Danish authorship.

A major achievement within the training area, though with a much wider audience, has been the compilation and publication since 1966 of the OCCA *Paint Technology Manuals* covering non-convertible and convertible coatings, solvents, oils, resins, driers, pigments, dyestuffs and lakes; paint application, testing and works practice—the whole gamut of paint technology. Many thousands have been sold and of the seven volumes (some in a second edition) only two remain available: No. 3 on "Convertible Coatings" and No. 7 on "Works Practice" which originally appeared as a series of Student Reviews in the *Journal*.

More than 18,000 copies of *The Introduction to Paint Technology*, now in its fourth (1976) edition, have been sold and this basic treatise for students and qualified personnel entering the industry continues to attract purchasers in many countries.

Finance: Headquarters

During the last ten years economic problems became all too familiar, with rapidly increasing costs, and disturbances in the state of trade and industry such as affected our Exhibitions.



In 1977, the Association acquired its own headquarters building, Priory House, situated on the outskirts of London between Wembley and Harrow

In three successive years, 1972-74, the Association had deficits in its income and expenditure accounts, and there were some very serious financial problems. Thanks to good responses to firm actions by the Council on all charges, including subscriptions, and critical economies in running costs, we have recently been in annual surplus. There are still problems and we remain, and rightly so, essentially dependent on undiminished support for all activities and recognition that they will continue to become increasingly expensive; but a major, long-standing ambition has been achieved. We now own our headquarters' premises, *Priory House*, Wembley, the purchase of the freehold having been recently negotiated without need of a bank loan or mortgage.

Nevertheless, in a review of the last ten years, it is proper to record that those financially critical middle years were further bedevilled by problems arising from the expiry of the lease on the former premises at Wax Chandlers Hall. The

1972 Annual Report pointed out that "difficulties seldom come singly" and that it was hoped to negotiate a favourable short lease renewal whilst the Association recovered from its financial reverses. This was not to be and the outcome was to move away from costly central London to the present address in suburbia; but unfortunately the move occasioned the loss of all the trained staff. Subsequently, the staffing situation improved, but throughout these difficult and changing times a very heavy burden has been carried by our *Director & Secretary*, *R. H. Hamblin*, to whom the Association is greatly indebted. It was with especial gratitude and pleasure that at the Annual General Meeting in June 1976 the Council marked Mr Hamblin's twenty-five years of service with the presentation on behalf of all members of a print by John Piper.

Now to The Future

During ten very momentous years the Association has steadfastly pursued the aims of its founders, with modifications in structure and activities to meet new needs arising from technological development and changes in the economic and social climate.

The Association has emerged with enhanced status, as an independent qualifying body with expanding overseas membership and unabated vigour in its technical and social activities at home and abroad. Moreover, after combating serious financial problems, it has emerged as the proud owner of its recently purchased headquarters.

Now, after sixty years, we look to the future, remembering that societies and associations are potentially immortal through continuous infusion of new blood; but as has been so well demonstrated during those ten years, continuing success demands frequent forward-looking re-appraisal of ends and means in ever-changing circumstances.



The small Committee Room in Priory House which has been used for meetings and viva voce examinations

OCCA

60th Anniversary



Celebrations

On the occasion of the Association's 60th Anniversary, the following functions have been arranged to be held on 11 and 12 May.

The actual date of the foundation of the Association was 16 May 1918, but it has been decided that the Thursday and Friday are more appropriate times to hold the celebrations.

Commemorative Lecture and Dinner



Sir John Methven

The Commemorative Foundation Lecture (instituted in 1963 in memory of the late H. A. Carwood, Esq., the first Honorary Secretary of the Association) will be given

in the Court Room at the Painters' Hall, Little Trinity Lane, London EC4, on the

evening of Thursday 11 May by Sir John Methven, Director General of the Confederation of British Industry. The title he has chosen is "The place of business in our society". Admittance to the Lecture will be by ticket only and will be followed by a short reception. Dinner will be taken in the Dining Hall of the Paint Stainers' Company at 7.45 p.m. Informal dress; will be worn.

Immediately following the lecture, a special commemorative silver medal will be presented to Sir John Methven by the Master of the Worshipful Company of Painter-Stainers, Sir Ralph Perring Bt., to mark the occasion.

Commemorative Dinner Dance

The Association's Dinner Dance will be held at the Savoy Hotel, London WC2, on the evening of Friday 12 May, and Presidents of other societies, together with their ladies, will be invited to attend.

The reception will take place in the River Room at 7.00 p.m., and Dinner will commence at 7.30 p.m. in the Lancaster Room. Arrangements for a cabaret have been made and dancing to the Jay Langham Orchestra will continue until 1.00 a.m. Dinner Jacket will be worn.

The application of linear programming to paint and resin formulation

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Summary

The application of linear programming to paint and resin formulation is discussed. As an example, an acrylic automobile lacquer was developed to equal or exceed the properties of a commercially

available lacquer. Linear programming was used in this process to assist in keeping the number of formulating steps required to a minimum.

Keywords

Miscellaneous terms

linear programming
formulation

Equipment primarily associated with analysis, measurement or testing

computer

L'utilisation de programmation linéaire pour le mis au point des formules de peintures et de résines

Résumé

On discute l'utilisation de la programmation linéaire pour le mis au point des formules de peintures et de résines. A titre exemplaire, on a développé une peinture acrylique pour automobiles dont les caractéristiques égalent ou dépassent celles d'une peinture de

commerce. On a utilisé la programmation linéaire dans ce procédé afin de contribuer à la restriction au minimum du nombre d'étapes requises pour établir la formulation.

Die Anwendung linearen Programmens auf die Ausarbeitung von Rezeptur für Lacke und Kunstharze

Zusammenfassung

Eine Besprechung der Anwendung linearen Programms auf die Ausarbeitung von Rezepturen für Anstrichmittel und Kunstharze. Als Beispiel wurde ein Automobilack auf Akrylharzbasis mit mindestens gleichguten oder besseren Eigenschaften, als denen eines

im Handel verfügbaren Lackes entwickelt. Lineares Programmieren wurde auf diese Weise benutzt um zu helfen, die zur Entwicklung der Rezeptur erforderlichen Schritte auf ein Minimum zu beschränken.

Introduction

Refs. 1-6

When a formulator is required to design a paint or resin for a specific purpose, or to match a competitor's product, there are certain minimum specifications which the paint must reach and, if possible, exceed. The cost must always be kept to a minimum, consistent with the required properties. A formulation of various components which the formulator, based on experience and knowledge, believes will meet the required specifications is then produced and tested. If the tests show that the formulation does not meet the required specifications then the formulator, again based on knowledge and experience, adjusts the quantities of the components, adds or deletes other components to try to obtain a formulation which does meet the specifications. The ability of the formulator is judged by how well the final formulation meets or exceeds the required specifications and how many steps of formulation and testing were required to reach the final formulation. The application of linear programming in the manner suggested here will help to ensure that the number of steps required to reach a final formulation is kept to a minimum, thus saving time and money. Some other benefits of this application of linear programming are that results are always quantified, making them easier to use in other formulations of similar products, and costs are always kept in mind and automatically minimised for the components tested.

Linear programming was first introduced by Dantzig¹ in 1947 to aid military planning. Since that time, many applications have been developed. Some of the more common are

economic analysis and the solution of production, sales, inventory and transportation problems. The general linear programming problem can best be summarised by the following equations:

$$c_1x_1 + c_2x_2 + c_3x_3 + \dots + c_nx_n = Z \dots\dots\dots (1)$$

$$a_{11}x_1 + a_{12}x_2 + a_{13}x_3 + \dots + a_{1n}x_n \geq b_1, \dots, \dots (2)$$

$$a_{21}x_1 + a_{22}x_2 + a_{23}x_3 + \dots + a_{2n}x_n = b_2 \dots \dots \dots (3)$$

$$a_{31}x_1 + a_{32}x_2 + a_{33}x_3 \dots a_{3n}x_n \leq b_3 \dots \dots \dots (4)$$

$$a_{m1}x_1 + a_{m2}x_2 + a_{m3}x_3 + \dots + a_{mn}x_n = b_m \dots \dots \dots (m)$$

where c_1, \dots, c_n are the costs of the variables x_1, \dots, x_n and a_{11}, \dots, a_{mn} are constants relating the variables to the constraints b_1, \dots, b_m . The problem is to find values of x_1, \dots, x_n which will satisfy the constraints b_1, \dots, b_m and at the same time minimise the total cost, Z . As an example, x_1, \dots, x_n might be the amounts of various components, such as titanium dioxide, resin, solvents, labour, *etc.* in a formulation. The costs per unit of these components are c_1, \dots, c_n . The constraint equations would represent various properties that the final formulation must have. For example, the hiding power must be equal to, or greater than, a value selected by the formulator. This selected value can be in any units, even the units of an arbitrary scale from 0 to 10. The linear programme solution would give the amount of each component in the formulation required to give the desired properties at minimum cost. The usual method of solving the linear

programme is called the "Simplex" method. Readers who are not familiar with this technique are referred to some of the many introductory texts²⁻⁶ on the subject.

At first sight, it may appear that linear programming would be of limited use in paint formulation because few properties are linear functions of the components, especially over wide concentration ranges. However, as the concentration range of a component becomes smaller then, generally, the relationship between the concentration of a component and the property of the paint concerned becomes more and more linear. It is this latter fact upon which the success of the proposed method depends.

After a first formulation has been produced and tested, then those properties of this formulation, which do not meet the specifications required, may be able to be written as equations of the form:

$$y = ax \dots \dots \dots (5)$$

where y is a numerical measure of a property, a is a constant and x is a weight fraction of a component on which this property depends. Thus, values of a may be found for various properties. By putting y equal to the required value of a property, then equation (5) becomes a constraint equation which can be solved for a new value of x . This new value of the weight fraction of a component may be used in the following formulation to give the desired value of a property. When the second formulation has been produced and tested then more information becomes available and the properties may be able to be written as equations of the form:

$$y = ax + d \dots \dots \dots (6)$$

where a and d are constants. Alternatively, the formulator may think that an equation of the form:

$$y = ax_1 + dx_2 \dots \dots \dots (7)$$

is more appropriate for some particular property. By making the values of y equal to the desired value of the property and solving the linear programme, a third formulation is arrived at which should be closer to the desired specifications. If this process is repeated, then a formulation will eventually be reached which has the desired properties, provided the components in the formulation are capable of giving the required specifications. Whether or not the components of a formulation are capable of giving the desired specifications would normally be indicated by a small change in the value of the property in successive formulations, and hence very large changes, perhaps greater than the formulation can accommodate, in the weight fractions of the components on which that property depends. When linear programming is applied in this manner, the total cost of a formulation is minimised subject to the specifications being met. Linear programming is applied repeatedly because the form of the equations relating properties to components is not known with the first formulation and only becomes clear after a number of formulations have been produced and tested. Thus, information is used as it comes to hand.

The application of linear programming to formulation is illustrated by matching certain properties of an acrylic automobile lacquer which is commercially available in Australia. The properties selected to be matched were hiding power, rocker hardness, adhesion and viscosity at 19 per cent solids in an equal parts mixture of toluene and ethyl acetate. The components selected to be variables were the proportion of titanium dioxide, methyl methacrylate, ethyl acrylate, acrylic acid, benzoyl peroxide, toluene and ethyl acetate.

To keep the illustration short, a number of simplifications were made such as the toluene/ethyl acetate ratio was kept at equal parts and no other monomer combinations were examined.

Experimental

Materials

All materials used were of commercial or laboratory reagent grade. The titanium dioxide was Tioxide R-CR6 (Tioxide Australia Pty. Ltd). The carbon black was Tintacarb 35 (Australian Carbon Black Pty. Ltd). The monomers methyl methacrylate, ethyl acrylate and acrylic acid were commercial grade donated by Rohm and Haas Australia Pty. Ltd. The benzoyl peroxide was 75 per cent active, moistened with water (Ajax Chemicals Ltd). The toluene and ethyl acetate used were laboratory reagent grade (Ajax Chemicals Ltd).

Preparation

Acrylic resins

The acrylic resins were manufactured in a three necked glass flask fitted with thermometer, stirrer, reflux condenser and dropping funnel. The flask was heated by an oil bath, magnetic stirrer and hot plate combination. Ten per cent of the monomers with 10 per cent of the benzoyl peroxide was added to refluxing toluene. The ratio of monomers added in this initial charge was determined by a computer solution of the copolymerisation equations from the 'Q-e' scheme. The Q-e values for the monomers were from Brandrup and Immergut⁷. The remainder of the monomers and initiator were added dropwise over a three hour period. Toluene was added as required.

Paints

The paints were made by dispersing the required amount of titanium dioxide in the acrylic resin diluted with ethyl acetate to the required consistency. The dispersing machine used was a Sardik High Speed Dissolver (Sardik Engineering Pty. Ltd) running at 6000 revolutions per minute. The paints were contained in a water cooled stainless steel container during dispersion. Dispersion was continued until no particles greater than ten microns were visible on a fineness of grind gauge (Sheen Instruments Ltd). The final paint was reduced to 37.5 per cent solids, so that the solvent composition was equal parts toluene and ethyl acetate.

Testing methods

All tests were carried out in an air conditioned room at a temperature between 20.5 and 21.5 °C.

Hiding power

Hiding power was determined by drawing down 0.15 mm wet film thickness of paint on to a Sheen 301 hiding power chart (Sheen Instruments Ltd). The resulting film was allowed to dry and rated subjectively for hiding power on a scale from 0 to 10. Because the commercial acrylic lacquer was an off-white colour, 0.08 parts of carbon black (Tintacarb 35) per one hundred parts of titanium dioxide was used in all formulations.

Hardness

Hardness was tested by drawing down a 0.2 mm wet film thickness of paint on to xylene washed 150 × 100 × 0.25 mm, tin plated steel. The dried film was tested with a Sward

Hardness Rocker (Gardner Laboratory Inc.). The average of six tests on different areas of the panel with the rocking direction parallel to the direction of drawdown was taken as a measure of the hardness.

Viscosity

Viscosity was tested by diluting the 37.5 per cent solids paint equal parts with one to one ratio toluene and ethyl acetate mixture and then measuring the time in seconds taken to empty a B4 cup to British Standard 1733 (Analite Pty. Ltd). Dilution of the commercial lacquer was done with the commercially available thinners.

Adhesion

Adhesion was measured by drawing a grid with lines 1 mm apart with a new E/11 shape scalpel on the same panels as used for hardness testing. Bear adhesive tape 6B (Norton Pty. Ltd) was placed over the grid and the air bubbles squeezed out. The tape was removed slowly by pulling the tape at 180° to the panel. The amount of paint film removed by the tape was assumed to be inversely proportional to the adhesion. The paint was given a subjective rating on a scale from 0 to 10 based on the amount of paint removed.

Impact resistance

Impact resistance was measured by dropping a 25 mm ball attached to a 0.25 kg weight from various heights on to a painted panel placed paint down over a 26 mm die. The painted panel was prepared in the same manner as for hardness testing. The height, in mm, from which the ball was dropped required to cause cracking in the paint film, was taken as a measure of the impact resistance.

Wedge impact resistance

In this test, 50 × 100 mm panels were cut from panels prepared in the same manner as for the hardness test. These smaller panels were bent around a 10 mm mandrel and placed on their side so that a one kilogram weight inclined at an angle of 7° to the horizontal could be dropped from a height of 700 mm to form a wedge. The wedge impact resistance was taken as the distance of cracking in the paint film measured from the small end of the wedge.

Flexibility

Panels (20 × 100 mm) were cut from panels prepared in the same manner as for the hardness tests. The panels were tested with the Sheen Bend test apparatus Reference 802 (Sheen Instruments Ltd). The mandrel sizes were 3.2 mm ($\frac{1}{8}$ inch) and 6.5 mm ($\frac{1}{4}$ inch).

Method

The commercially available lacquer to be matched had the properties listed in Table 1. These properties are the average of four control tests, as a control test on the commercial

lacquer was repeated as each formulation was tested. Four formulations were required satisfactorily to match the commercial lacquer. These formulations are listed in Table 2. The units used in Table 2 are weight fractions. The properties of each formulation are listed in Table 3. The values in brackets are the properties of the commercial lacquer.

Table 1
Properties of the commercial lacquer

solids content	37.5%
hiding power	8
hardness	17.3
viscosity	25.7 s
adhesion	8
impact resistance	103 mm
wedge impact	56 mm
flexibility	pass 3.2 mm mandrel

Table 2
Formulations

	1	2	3	4
titanium dioxide	0.1529	0.1370	0.1373	0.1372
methyl methacrylate	0.0894	0.1731	0.1404	0.1551
ethyl acrylate	0.1333	0.0647	0.0928	0.0789
acrylic acid			0.0048	0.0036
benzoyl peroxide	0.00046	0.00033	0.00029	0.00024
toluene	0.3124	0.3124	0.3124	0.3124
ethyl acetate	0.3124	0.3124	0.3124	0.3124

The first formulation was based on the author's experience and has a 13.7 per cent pigment volume concentration, the binder being a 60 per cent ethyl acrylate and 40 per cent methyl methacrylate copolymer. The linear programme for this formulation is:

$$70.0x_1 + 55.0x_2 + 70.0x_3 + 269.0x_4 + 16.8x_5 + 46.3x_6 = Z, \dots (8)$$

where Z is the cost of the formulation per kilogram. The variables x_1 to x_6 are the weight fractions of titanium dioxide, methyl methacrylate, ethyl acrylate, benzoyl peroxide, toluene and ethyl acetate respectively. The constants were the costs per kilogram of these materials in Australia at the end of 1974. The value of Z must be minimised subject to the following constraints:

$$x_1 + x_2 + x_3 + x_4 + x_5 + x_6 = 1.0, \dots (9)$$

$$0.625x_1 + 0.625x_2 + 0.625x_3 + 0.625x_4 - 0.375x_5 - 0.375x_6 = 0.0, \dots (10)$$

$$x_5 - x_6 = 0.0, \dots (11)$$

$$58.86x_1 \geq 8.0, \dots (12)$$

$$167.8x_2 \geq 17.6, \dots (13)$$

$$113.2x_4 = 0.039, \dots (14)$$

Table 3
Formulation properties

	1	2	3	4
hiding power	9 (8)	8 (8)	8 (8)	8 (8)
hardness	15.0 (17.6)	18.3 (17.2)	16.5 (17.3)	17.7 (17.3)
viscosity	19.2 (25.5)	23.5 (25.5)	24.3 (26.0)	26.1 (25.8)
adhesion	3 (8)	3 (8)	10 (8)	9 (8)
impact resistance	>1000 (120)	40 (100)	>1000 (90)	>1000 (100)
wedge impact	0 (60)	60 (50)	0 (56)	35 (59)
flexibility	pass 3.2 mm	fail 3.2 mm	pass 3.2 mm	pass 3.2 mm

Constraints 9, 10 and 11 are invariant constraints, in that they are constant for all formulations. Constraint 9 is to make the total mass of the formulation one kilogram. Constraint 10 keeps the solids content at 37.5 per cent, whilst constraint 11 keeps the toluene concentration equal to the ethyl acetate concentration. Constraints 12, 13 and 14 are variable constraints, in that they may change for different formulations. Constraint 12 is the hiding power constraint and ensures that the hiding power remains greater than or equal to 8 on the arbitrary hiding power scale. The value of the constant 58.86 was obtained by solving the equation:

$$0.1529 a_1 = 9.0 \dots \dots \dots (15)$$

that is, assuming the hiding power to be a linear function of the weight fraction of titanium dioxide. Constraint 13 is the hardness constraint and is obtained by assuming that hardness is a linear function of the methyl methacrylate weight fraction. Constraint 14 is the viscosity constraint, which was obtained by assuming the viscosity was inversely proportional to the benzoyl peroxide weight fraction. The other properties were not programmed as constraints for various reasons. Adhesion was not programmed because the author did not know how any of the components present affected adhesion. Impact resistance and wedge impact were not programmed because no specific values were obtained from formulation 1. Flexibility was not programmed because it was not a continuous function. A computer programme was written to solve the above linear programme, which was solved on an ICL 1901A computer. A number of extra constraints, called safety constraints, were automatically added by this programme to ensure that components did not disappear from the formulation. The solution to the above programme was formulation 2. In this formulation the weight fraction of methyl methacrylate was higher than predicted to give a rocker hardness of 17.6 because the constraint was written as a \geq constraint, and methyl methacrylate, being cheaper than ethyl acrylate, replaced ethyl acrylate in the formulation to the minimum amount of ethyl acrylate allowed by the safety constraint. From the properties of formulation 2 the following constraints were written for a second linear programme:

$$x_1 + x_2 + x_3 + x_4 + x_5 + x_6 = 1.0 \dots \dots \dots (16)$$

$$0.625x_1 + 0.625x_2 + 0.625x_3 + 0.625x_4 - 0.375x_5 - 0.375x_6 = 0 \dots \dots \dots (17)$$

$$x_5 - x_6 = 0 \dots \dots \dots (18)$$

$$0.2378x_1 - 0.1370x_2 - 0.1370x_3 = 0 \dots \dots \dots (19)$$

$$39.43x_2 = 5.73 \dots \dots \dots (20)$$

$$33077x_4 = 8.9 \dots \dots \dots (21)$$

Constraints 16, 17, 18 are the same invariant constraints as step 1, however, since the hiding power was now considered to be matched, constraint 19 was added to the invariant constraints to keep the ratio of titanium dioxide to polymer the same as formulation 2. The hardness constraint, equation 20, was obtained by solving the equations:

$$0.0894a_2 + D_2 = 15.0 \dots \dots \dots (22)$$

$$0.1731a_2 + D_2 = 18.3 \dots \dots \dots (23)$$

Thus hardness, H , was assumed to be the following linear function of the methyl methacrylate concentration:

$$H = 39.43x_2 + 11.47 \dots \dots \dots (24)$$

When H is put equal to 17.2, the constraint equation 20 is obtained. This time the constraint sign was made $=$ rather

than \geq . The viscosity constraint 21 was obtained by solving the equations:

$$0.00046a_4 + D_4 = 19.2 \dots \dots \dots (25)$$

$$0.00033a_4 + D_4 = 23.5 \dots \dots \dots (26)$$

The solution to the above linear programme gave formulation 3, excepting that the acrylic acid content was added to the methyl methacrylate. It was decided to incorporate some acrylic acid at this stage to improve the adhesion and this change resulted in formulation 3. In this formulation the acrylic acid replaced some of the methyl methacrylate.

The properties of formulation 3, Table 3, show that this formulation was not an acceptable match to the commercial lacquer. The linear programme for the next step, step 3, therefore became:

$$70.0x_1 + 55.0x_2 + 70.0x_3 + 269x_4 + 16.8x_5 + 46.3x_6 + 51.0x_7 = Z \dots \dots \dots (27)$$

where x_7 is the weight fraction of acrylic acid, and, as before, the constants are the costs of the components. The following are the constraints:

$$x_1 + x_2 + x_3 + x_4 + x_5 + x_6 + x_7 = 1.0 \dots \dots \dots (28)$$

$$0.625x_1 + 0.625x_2 + 0.625x_3 + 0.625x_4 - 0.375x_5 - 0.375x_6 + 0.625x_7 = 0 \dots \dots \dots (29)$$

$$x_5 - x_6 = 0 \dots \dots \dots (30)$$

$$0.2378x_1 - 0.137x_2 - 0.137x_3 - 0.137x_7 = 0 \dots \dots \dots (31)$$

$$55.1x_2 = 8.5 \dots \dots \dots (32)$$

$$20,000x_4 = 4.6 \dots \dots \dots (33)$$

$$1458x_7 = 5 \dots \dots \dots (34)$$

Equations 28-31 are similar invariant constraints to the step 2 linear programme. Equation 32 is the hardness constraint and was obtained by solving equations 35 and 36 and manipulating the hardness equation, 37, by putting H equal to 17.3:

$$0.1731a_2 + D_2 = 18.3 \dots \dots \dots (35)$$

$$0.1404a_2 + D_2 = 16.5 \dots \dots \dots (36)$$

$$H = 55.1x_2 + 8.8 \dots \dots \dots (37)$$

The viscosity constraint, 33, and the adhesion constraint, 34, were obtained in a similar manner by solving the appropriate equations. The solution to this linear programme gave formulation 4 which was judged to be an acceptable match to the commercial lacquer after testing. Slight differences between Table 2 and the predicted formulations may be observed due to weighing difficulties.

Conclusions

The above procedure shows the application of linear programming to a relatively simple formulation problem. The final formulation matches the commercial lacquer for those properties tested, however, many important properties were not tested, such as durability. It should be pointed out that a computer programme is not necessary to solve simple linear programming problems. Where a property of the formulation can reasonably be attributed to one particular component and the constraint signs are put equal to, then the equations are simple and may be solved by inspection. Only where properties of a formulation are functions of more than one component and the constraint signs are greater than or less

than, does computer programming of the problem become necessary.

The greatest advantage of the method is that it allows the inexperienced formulator to reach an acceptable formulation in a finite number of steps, provided the components selected are capable of giving this acceptable formulation. The experienced formulator should be able to use the method with much greater effect because he should be able to get closer to the optimum formulation on the first attempt.

Listings of the computer programmes used in this work may be obtained by writing to the author.

[Received 20 September 1977]

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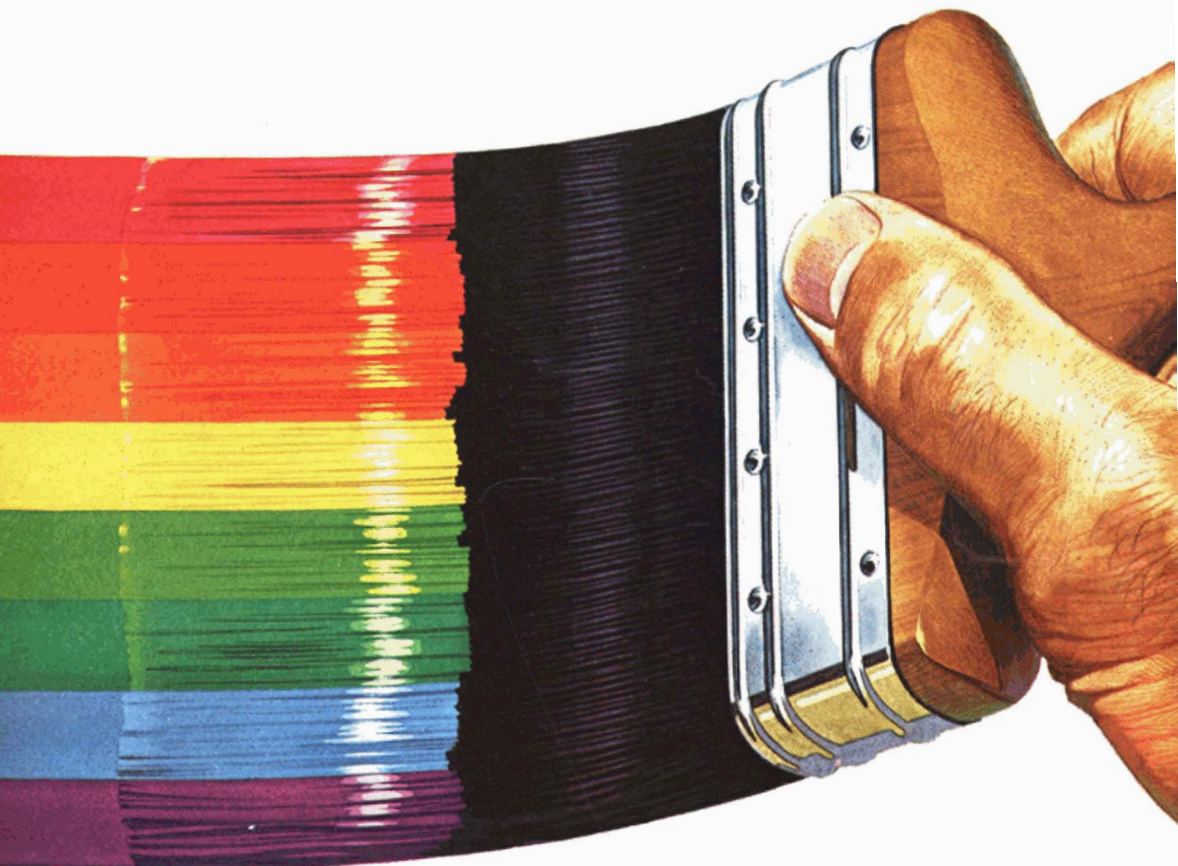
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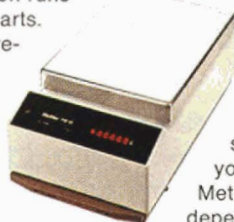




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Microanalysis of marine antifouling paints in the scanning electron microscope—its automation and application to less homogeneous paints

By R. J. Bird and D. Park

Shell Research Ltd, Thornton Research Centre, P.O. Box 1, Chester, CH1 3SH

Summary

In an earlier paper¹ the scanning electron microscope with X-ray microanalysis was shown to be a valuable tool with which to follow compositional changes occurring in antifouling paints during service. A description is now given of a procedure for speeding up such

work using some automated collection and computer processing of the data. Paints containing relatively large particles present added difficulties and means are discussed by which these may in part be overcome.

Keywords

Types and classes of coatings and allied products

antifouling coating

Raw materials

pigments

copper oxide

biologically active agents

copper salt

organotin

Processes and methods primarily associated with analysis, measurement or testing

electron microscopy

computer

X-ray analysis

Properties, characteristics and conditions primarily associated with the environment

leaching

Miscellaneous Terms

data processing

L'automatisation de la technique de microanalyse de peintures "antifouling" au moyen de la microscopie électronique à balayage et l'utilisation de cette technique pour étudier les peintures moins homogènes

Résumé

On a démontré dans un exposé précédent que la microanalyse par les rayons X au moyen de la microscopie électronique à balayage est une méthode utile par laquelle on peut étudier les altérations qui se produisent dans la composition des peintures "antifouling" au cours de leur emploi. On décrit actuellement un procédé pour accélérer ces études en se servant des techniques de l'automatisation

dans la (collecte) de certaines données et d'un ordinateur dans le traitement de l'information. Les peintures qui contiennent des particules relativement grosses présentent des difficultés supplémentaires, et l'on discute les moyens par lesquels en partie on saurait les surmonter.

Mikroanalyse von Marine Antifoulingfarben im Abtast—Elektronen—Mikroskop, ihre Automatisierung und Anwendung für weniger homogene Farben

Zusammenfassung

In einem früheren Artikel wurde aufgezeigt, dass das Abtast—Elektronen—Mikroskop zusammen mit Röntgenstrahlen—Mikroanalyse ein wertvolles Werkzeug für die Beobachtung von Änderungen in der Zusammensetzung von Antifoulingfarben während ihres Gebrauches ist. Nunmehr wird ein Verfahren beschrieben,

wie solche Arbeit durch automatisierte Ansammlung und komputersierte Behandlung der Befunde beschleunigt werden kann. Verhältnismässig grosse Teilchen enthaltende Farben verursachen zusätzliche Schwierigkeiten, und Massnahmen werden besprochen, wie diese teilweise überwunden werden können.

Introduction

Ref. 1

The selection of the most efficient antifouling hull paint is an economic factor of great importance in marine transportation. In the assessment of paint performance it is necessary not only to inspect the outer surfaces for the onset of fouling, but also to follow changes in the microscopic distribution of toxicants within the paint film by the examination of suitably prepared sections. In this type of work the scanning

electron microscope with X-ray analysis facilities has proved of great value, as discussed in an earlier paper¹.

The active materials in the paint films are most usually present in particulate form and often not very uniformly distributed. It is necessary, therefore, to obtain average figures. This is done along lines parallel to and at various depths below the paint surface. Such results give valuable concentration profiles of the active materials present and wherever possible these are referred to related paints of

known composition for the better assignment of actual concentrations.

The length of line that can be scanned in the microscope at the usual magnifications used is limited to about 100 μm , and for the more coarsely particulate toxicants this may intercept only a few particles. Concentration profiles produced under these conditions are not sufficiently smooth, and consequently it is necessary to obtain and average data from several traverses through the paint specimen. It is expedient, therefore, to record the analytical data on punched tape and subsequently to process them, including the final plotting of results, on off-line computing facilities.

The aim of this paper is firstly to give details of the procedure that is proving very valuable despite the fundamental difficulties in this type of analysis, as touched upon in the earlier paper⁴. The authors discuss further some of the problems of quantitative electron probe analysis of samples that are not microscopically homogeneous over the analysed regions, emphasising possible sources of error, whilst stressing the usefulness of the technique described.

Experimental

The technique is essentially one for the examination in section of small mounted pieces of test panels, usually with the paint still attached to the substrate. The mounting and preparation of the sections is described in the earlier publication.

The X-ray analysis equipment used is of the energy dispersive type, in which a lithium drifted silicon detector generates electrical pulses proportional in size to the quantum energies of the various X-radiations falling on it. These pulses, suitably amplified and digitised, are recorded in the memory of a small computer, the content of which is continuously displayed on a monitor screen. After a suitable accumulation time, which depends on the count rate, a spectrum such as that in Figure 1 is obtained. This relates to an antifouling paint and shows peaks for the biocide elements copper and tin as well as others due to pigment and binder.

The system may be set up to provide net integrated peak intensities corrected for background and to record these on

punched paper tape. With corresponding data from suitable standards and manually inserted identification and positional data, the tape is then transferred to a Univac 1108 computer where concentrations are derived and plotted with the aid of a small Fortran programme written for the purpose.

The more detailed procedure is illustrated by the flow diagram, Figure 2. Firstly, sample identification and other details that will be needed in the titling of the final presentation of results are typed. These are followed by plotting instructions, microscope screen magnification (needed in position plotting), concentrations of active elements in standards and the counting time over which spectra will be accumulated (usually 20 seconds). Output from the analyser is firstly the intensities found for the active elements in the

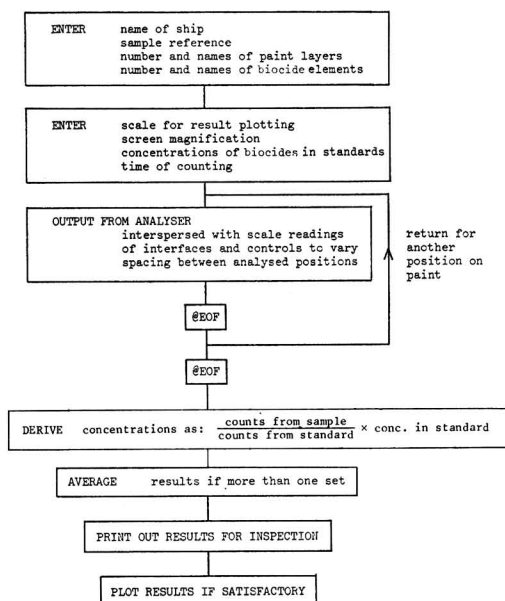


Fig. 2. Flow diagram showing data acquisition and handling procedures

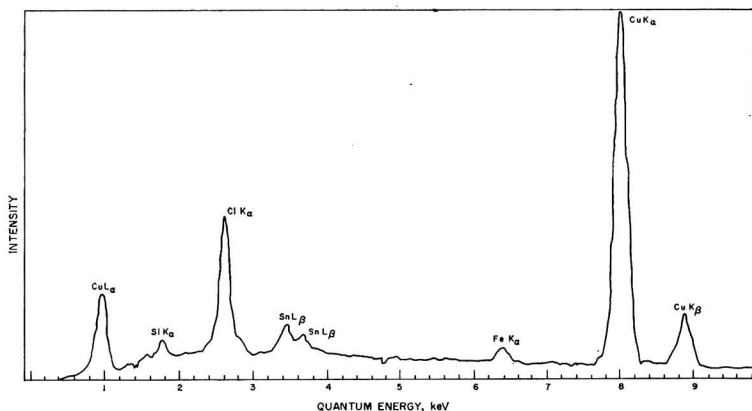


Fig. 1. X-ray spectrum of typical paint (paint 2)

standards, and then the intensity data from the sample, interspersed as necessary with positional information.

Analysis is carried out at each of as many as 30 positions selected by reference to a graticule marked out vertically on the viewing screen of the electron microscope. The zero of the graticule scale is on the centre of the screen and the paint section is moved by the stage controls so as to bring the interface between the antifouling layer and the undercoating on to the zero position and oriented at right angles to the graticule. In this orientation the interface is parallel to the line scan direction of the microscope, Figure 3. The graticule divisions are 5 mm, which at the $600\times$ magnification usually employed in this work corresponds to $8\text{ }\mu\text{m}$ on the sample.

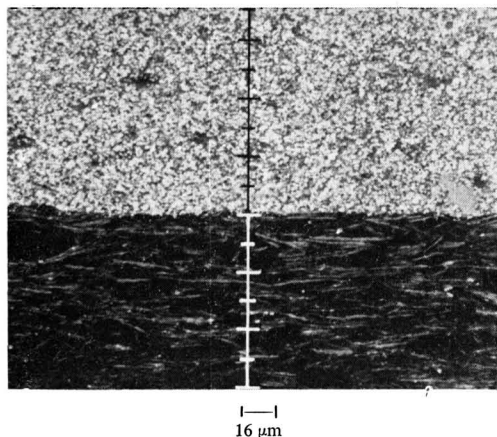


Fig. 3. Graticule (5mm divisions) superimposed on paint section

During analysis two kinds of positional information are inserted from the keyboard. The first of these are the positions of the outer surface and of the interfaces between the various layers encountered during the analytical sequence. The second kind defines the position at which analysis is carried

out. It is, of course, important to minimise the amount of positional information to be keyed in during the analysis and to this end the starting assumption is made that analysis will be made at regular intervals of position starting with the first integral reading on the graticule inside the outer surface of the paint. It is further assumed that the interval will be one scale division. However, the size of the interval can be reduced in the regions of rapid change or increased in the less interesting regions simply by keying in the new desired interval with an appropriate prefix. Thus, unless a change of position interval is required, it is unnecessary to enter positions of analysis.

In paint systems containing biocide in particulate form, the varying intersections of the line scans with particles can give erratic results in the plotted concentration profiles. Better results can be obtained by collecting data at more than one position in the section of the paint film and averaging the results. This is possible provided the thicknesses of the various paint layers on a given panel are reasonably constant from one position to another and the same set of analytical positions is used throughout. Small variations in interfacial positions can be accommodated in the treatment and average positions are indicated in final plots where more than one set of data has been obtained and averaged. Figure 4 illustrates a typical output.

With some paints spectral interferences occur; for instance, if it is required to measure the zinc content of a paint which also contains copper. The K_{β} radiation from the latter gives a peak that almost coincides with the preferred analytical line of Zn, the K_{α} radiation. It is a simple matter in the Fortran programme to build in a correction for this interference based on the measured intensity of the $\text{Cu } K_{\alpha}$. Some other simple correction procedures could also be added, but the established procedures widely used in accurate quantitative microanalysis are not applicable in this type of work where the sample is not homogeneous over any one analysed region, i.e. any one line. Because of the fundamental limitation on accuracy, element concentrations are derived simply from the ratio of intensities from sample and standard, and the known concentration in the standard,

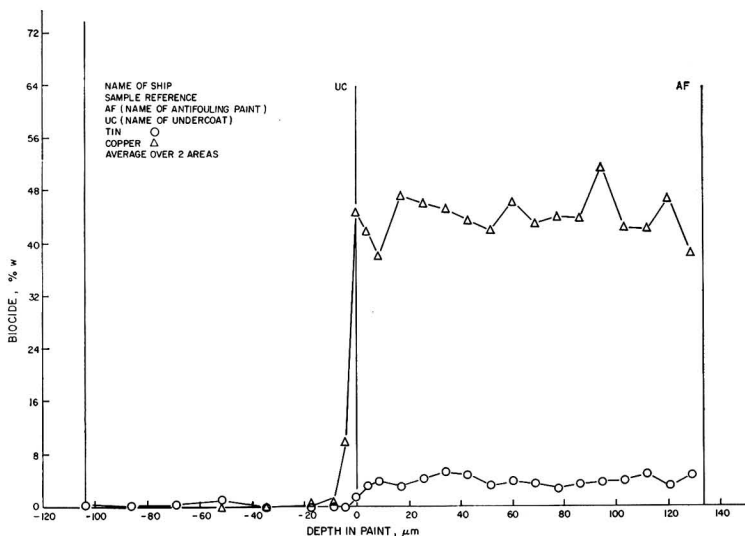


Fig. 4. Typical plotted output

assuming strict proportionality between intensity and concentration.

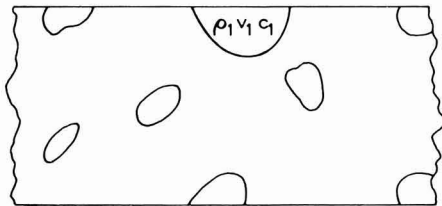
Analysis of paints containing coarsely particulate toxicants

Refs. 1, 2

Because of the particulate nature of the pigments and the fillers they contain, paints in general are far from ideal subjects for quantitative electron probe microanalysis. The particle size of the dispersed materials is of the same order of size as the depth of sample accessible to analysis, so that it is impossible to employ the correction procedures normally used in quantitative work to eliminate the errors due to inter-element effects.

Conventional copper oxide antifouling paints contain biocide particles almost as fine as the average pigment material; and as discussed in reference 1, the simple approximate approach to quantitative electron probe analysis can give line average results for these paints which are fairly close to copper contents known from manufacturing data. More recent types of paint utilising other active elements do, however, frequently contain coarser particles. It is for these that the automation in analysis becomes even more valuable in facilitating averaging of results at several positions. In extreme cases particles up to more than 50 μm have been seen.

The accuracy of analysis of such coarsely particulate systems cannot be high. As well as the difficulty of obtaining adequately smoothed representative data with a reasonable length of scan line, there are effects on the emitted X-ray intensity that will vary with particle size. There is also an important error if the particles are not of the same density as the surrounding paint film. This can best be considered for the case of very large particles, when the scanning electron beam will dwell almost entirely on the biocide-rich particles or on the surrounding paint composition. The fraction of time spent crossing boundaries will be small and boundary effects will therefore also be small. The average X-ray intensity from the active element will depend on the percentage area presented in the section by the particles containing it. As shown in standard works on stereology (*e.g.* Underwood²) the percentage area occupied by a disperse phase in a section is numerically the same as its volume percentage, as also is the percentage intercept of lines taken across the section surface.



- Let ρ_1 = density of particles
 v_1 = fractional volume of particles
 c_1 = concentration of element 1 in particles
 C_1 = concentration of element 1 in paint
 ρ = density of paint
 c_2 = concentration of element 2 inside particles
 C_2 = concentration of element 2 in paint

Adopting the basic assumption in electron probe analysis of proportionality between X-ray intensity and weight concentration of emitting element, the emission from the particles may be written as:

$$I_1 = A_1 c_1$$

But c_1 can be expressed in terms of the densities and volumes of particles, as:

$$c_1 = C_1 \cdot \frac{\rho}{\rho_1 v_1}$$

$$\therefore I_1 = \frac{A_1 C_1}{v_1} \cdot \frac{\rho}{\rho_1}$$

If the probe is now set to line scan, it will fall on particles for only v_1 as a fraction of time, and the average may be written:

$$I_1 \text{ average} = A_1 C_1 \cdot \frac{\rho}{\rho_1}$$

Thus the quantity ρ_1/ρ , which will be denoted by F_1 , is seen to be the correction factor to apply to the apparent result.

Along similar lines, for the element 2 in the main part of the film can be written:

$$I_2 = A_2 c_2 \\ = A_2 C_2 \frac{\rho}{\rho - \rho_1 v_1}$$

and:

$$I_2 \text{ average} = A_2 C_2 \frac{(1 - v_1)\rho}{\rho - \rho_1 v_1}$$

Hence:

$$C_2 = \frac{I_2 \text{ average}}{A_2} \cdot \left(\frac{1 - \frac{\rho_1}{\rho} v_1}{(1 - v_1)} \right)$$

where the factor $\left(\frac{1 - \frac{\rho_1}{\rho} v_1}{1 - v_1} \right)$ or F_2 , is the correction factor to apply to this result.

This is displayed graphically in Figure 5 for various values of v_1 and ρ_1/ρ .

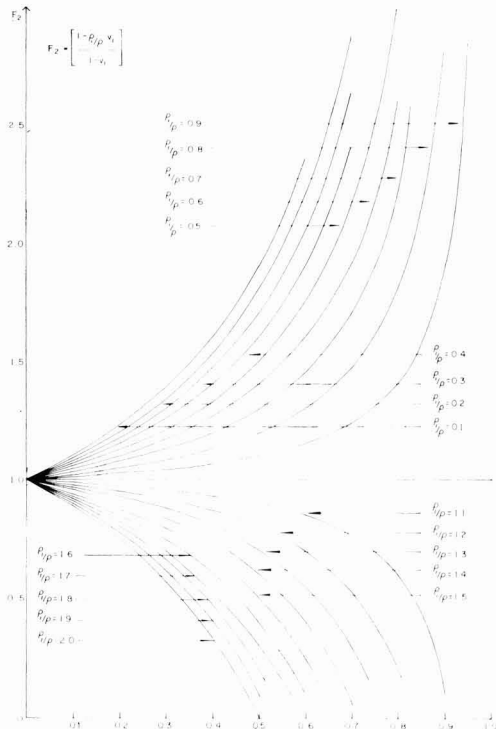


Fig. 5. Correction factor F_2 for the effect of density on the analysis of the distributed element

Some test results have been obtained for paints containing two active materials, one in coarsely particulate form. Sections through these paints are illustrated in Figure 6.

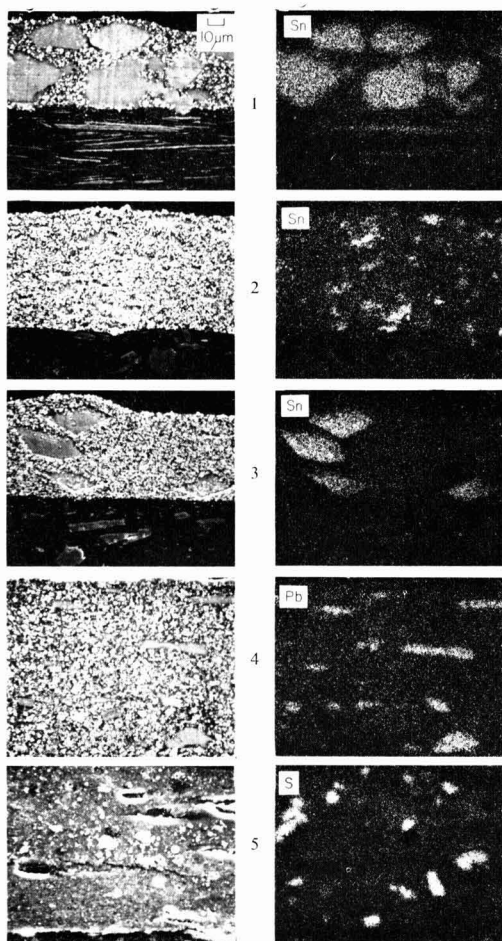


Fig. 6. Micrographs of paint films in unexposed condition

For each paint, in addition to line average observations as discussed above, local analyses were carried out by stationing the electron probe on selected large particles so as to obtain

the biocide concentration c_1 within the particles. The assumption was then made that the area fraction of large particles in the section (i.e. v_1 , their volume concentration in the bulk) would be obtained from the line average apparent concentration on dividing the latter by c_1 . In some paints a small amount of the toxicant was found generally distributed. This concentration was subtracted from the line average figure before it was used to deduce the area fraction of large biocide-rich particles. The overall densities of the film were obtained from simple measurements on detached films prepared on tin foil from which they could usually be satisfactorily stripped mechanically. Overall densities of the paint films and densities of the particles are given in Table 1. This information enabled the corrections described above to be applied. Table 2 shows the analytical results for the paint films before and after correction and compares them with other compositional information on the paints.

Table 1
Density information on paint films

Paint No.	Density of particulate biocide (ρ_1), g/cm ³	Overall density of film, g/cm ³
1	1.25	2.0
2	1.26	2.3
3	1.25	2.0
4	1.25	2.3
5	1.00	1.2

Paint 1

This paint contained copper oxide and an organotin compound. The latter was present in the unexposed paints as very large crystals up to about 30 μm in size. The tin X-ray intensity ratio from these particles relative to the pure element gave an approximate tin content of 37 per cent by weight. This figure was reduced to 32 per cent on applying a simple absorption loss correction and is to be compared with a theoretical figure of 28 per cent by weight. Point analysis outside the large particles indicated the presence of about 2 per cent tin generally distributed. Re-examination of this paint after 8 months of service on a ship revealed that the tin-rich particles had been lost, and the paint film had been deformed to leave a thinner void-free film containing practically no tin.

Paint 2

The active materials in this paint were copper oxide and an organotin compound present in particles up to about 10 μm in size. The approximate simple intensity ratio result for the tin content of these particles was 48 per cent by weight. This was reduced to 37 per cent by a simple absorption

Table 2
Analytical results on paints with coarse biocide particles, showing the effect of correction for particle density

Paint	Biocide present largely in particulate form								Distributed biocide					
	Element	Particle size μm	Concentration in particles, %w	% Area of particles	Concentration in paint, %w			According to manufacturer	Element	Concentration in paint, %w				
					By electron probe analysis		By chemical analysis			By electron probe analysis	By chemical analysis	According to manufacturer		
					Uncorrected	Corrected								
1-unexposed	Sn	30	37	32	14	10	9		Cu	24	28	33		
-exposed 8 months	Sn	10	48	8	<0.5	4	2.2		3.7	Cu	36	36		60
2-unexposed	Sn	50	46	11	5	3.1			4	Cu	56	58		60
-exposed 8 months	Sn	20	46	9	4	2.4	5.3			Cu	47	49		
4-unexposed	Pb	12	41	14	8	5.7				Cu	51	60	50	
5-unexposed	S		38	11	5	4.3				Sn	7	7	8	
-exposed 8 months					1	1					4	4		

correction, compared with a theoretical content of 33 per cent. Outside the large particles in the main body of the paint film no tin was detected.

Paint 3

The active materials in this paint were copper oxide and an organotin compound closely related to that in paint 2, with a theoretical tin content of 34 per cent by weight. The tin was present in particles up to 50 μm in size, and electron probe analysis results for them were 46 per cent tin, falling to 35 per cent after the absorption correction. No tin was detected outside the large particles. After 8 months service on a ship the tin had largely disappeared from the outer region of the paint, but large tin-rich particles were still to be seen in the interior of the film. Analytical figures for this inner region are included in Table 2.

Paint 4

This paint contained copper oxide and an organolead compound. The latter was present largely in separate particles up to about 20 μm in size. Electron probe analysis on these gave a lead content of 41 per cent by weight uncorrected. It was not possible to apply the absorption correction because no reference data were available for the Pb M radiation used in the analysis. The theoretical lead content of the toxicant was 42 per cent. Outside the lead-rich particles there was a small amount of lead continuously distributed and corresponding to a concentration of about 2 per cent by weight.

Paint 5

The active materials in this paint were a sulfur compound present largely as separate particles up to about 12 μm in size, and an organotin compound uniformly distributed outside the sulfur particles. There was some additional sulfur corresponding to about 1 per cent generally distributed with the tin. After 8 months of service on a ship the sulfur-rich particles were no longer to be seen and the concentrations of continuously distributed toxicants were reduced.

Discussion

This approach to the automation of paint film analysis requires the presence of the microscope operator most of the time. However, this is no great disadvantage because in the analysis of even the best prepared films the analyst must search for and select with great care areas of section suitably free from irregularities and imperfections. Automatic recognition of interfaces in a series of various paints might also be difficult to contrive and not economic in overall effort. There is the great virtue that little further manual effort is required when the microscopic examination is completed.

In seeking to apply this type of analysis to paints in which the active material is segregated into large particles it can be seen that errors up to 40 per cent can occur, much of it predictable if the particles are different in density from their surroundings. In the cases of tin in paint 1 and lead in paint 4 the appropriate corrections give reasonable agreement with chemical analysis, for paints 2 and 3 rather poorer agreement

is found with compositions according to manufacturers' information. The consequences of such segregation for results for a second constituent remaining more generally distributed are less severe. The maximum error incurred in the present examples is only about 16 per cent.

For particles of smaller size, boundary effects will become more important, and this will make the basis of the density correction less valid. At the same time it would be expected that the total effects of segregation should diminish with particle size and correction would become progressively less important, until, when the particles are small compared with the effective probe diameter, which is about 1 μm the, particulate nature of the active material would present no additional problem.

The agreement found between the electron probe results and other information is not always good. This must in part be due to the inadequacy of the relatively short scan line in deriving representative average intensities where the particles encountered are not small relative to the line length. Inter-element effects (principally X-ray absorption losses resulting from the presence of pigment and filler materials) will also cause errors; and, as discussed earlier, no correction can be applied. With the type of instrument employed in this work, a scanning electron microscope, some uncertainty of X-ray take-off angle must be expected. This could introduce error and may be responsible for some of the discrepancies between measured and theoretical compositions of particles of active material.

Conclusions

With paints in which an active material is present as relatively large particles, analytical difficulty is increased, but useful results can still be obtained.

When the density of a separated material is different from the overall density of the paint, substantial errors can be introduced.

Expressions for correction factors to be applied where the particle density is known can materially improve the agreement between electron probe results and other analytical information on known paints.

Where density corrections have been applied, test results can still show discrepancies compared with other sources of information, which if true can, at least in part, be attributed to the specimen heterogeneity.

Although the absolute accuracy of the results by electron probe analysis may not be very high for the types of paint discussed, the technique is nevertheless invaluable in that it facilitates the study of changes in biocide content and distribution during the service life of the paint.

[Received 27 September 1977]

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The mathematical relationship between the viscosity of PVCI plastisols including fillers and plasticiser number of fillers

By J. Wypych and J. Walczak

Central Laboratory for Technical Products, Lodz, Poland

Summary

The paper gives data on the viscosity of PVCI plastisols containing fillers and plasticiser number of 16 commercial grade fillers having different chemical constitution. The results obtained have been

used for the measurement of the parameters of an equation describing viscosity of PVCI plastisols, containing any quality of filler as a function of filler plasticiser number.

Keywords

Raw materials

binders (resins, etc.)

plastisol resin

vinyl chloride resin

plasticisers

plasticisers

Properties, characteristics, and conditions primarily associated with materials in general

viscosity

Le rapport mathématique entre la viscosité des plastisols de C.P.V., y compris les matières de charge, et l'indice de plastifiant des matières de charge

Résumé

Cet exposé présente les données sur la viscosité des plastisols de C.P.V. contenant des matières de charge et d'ailleurs sur l'indice de plastifiant de 16 matières de charge de commerce ayant les constitutions chimiques différentes. Les résultats obtenus ont été

utilisés pour déterminer les paramètres d'une équation qui décrit la viscosité des plastisols de C.P.V. contenant des matières de charge de toutes qualités en fonction de l'indice de plastifiant du matière de charge.

Die mathematischen Beziehungen zwischen der Viskosität von PVC-Plastisolen enthaltend Weichmacher und eine Reihe von Füllstoffen

Zusammenfassung

In dieser Abhandlung werden Angaben vorgelegt über die Viskosität von PVC-Plastisolen, welche Füllmaterial und Weichmacher enthalten und zwar 16 handelsübliche Qualitäten von sich in ihrer chemischen Konstitution unterscheidenden Extendern. Die erhaltenen

Resultate wurden zur Messung der Parameter einer Gleichung benutzt, welche die Viskosität von Füllmaterial irgendwelcher Qualität enthaltenden PVC-Plastisolen als Funktion der Extender-Weichmacherzahl angibt.

Refs. 1-4

Fillers are incorporated in plastic materials to improve the general and physical properties, to introduce particular characteristics, or to reduce their cost. For these reasons, the methods of use of the various fillers available, having different chemical constitutions and methods of preparation, and varying especially in particle size and morphological structure, are well known.

Since fillers are capable of absorbing the plasticisers, it is obvious that the addition of filler must have an effect on the viscosity characteristics of pastes in which they are incorporated.

According to Kerr¹, the commonly used fillers tend to increase the viscosity due to the value of their oil absorption. Bergan² pointed out that the rheological characteristics of filled pastes can vary depending on the rate of shear.

Further papers^{3,4} covering this subject give data on some fillers and their effect of usage.

Based on the above mentioned papers, it can be said that the precise formulation details regarding the influence of fillers on plastisol viscosity is now an important subject for investigation.

The relationship between plasticiser number of fillers and its effect on plastisol viscosity is the subject of this investigation.

Experimental

Materials

The types of the fillers tested are listed below in Table 1.

A dispersion of polyvinyl chloride in plasticiser was used as polymer [E-68 Pbs ($K=68$). K is Fikentscher's number denoting the molecular weight of the polymer]. The plasticiser used was di(2-ethylhexyl) phthalate (DOP).

Table 1
Types of fillers used

Filler	Commercial Grade	Chemical composition	Plasticiser Number
Barytes	<i>EWO</i>	BaSO ₄ — 94%; SiO ₂ — 6%	16.0
Dolomite	<i>Microdol 1</i>	Ca.Mg (CO ₃) ₂ — 99%	26.6
Silicious earth	<i>Gloxil weiss</i>	SiO ₂ — 85%; Al ₂ O ₃ — 10%	51.6
Kaolin	<i>Kaolin KG</i>	SiO ₂ — 49.5%; Al ₂ O ₃ — 35%	55.0
Barium sulphate	<i>Blanc Fixe Powder F</i>	BaSO ₄ — 99%	25.8
Kaolin	<i>Kaolin FI</i>	SiO ₂ — 48.1%; Al ₂ O ₃ — 36.5%	58.9
Barytes	<i>Barytmehl F</i>	BaSO ₄ — 92%; SiO ₂ — 8%	12.8
Dolomite	<i>Microdol Extra</i>	Ca.Mg (CO ₃) ₂ — 99%	32.5
Barytes	<i>Barytmehl N</i>	BaSO ₄ — 88%; SiO ₂ — 12%	30.5
Titanium dioxide	<i>Hombitan R101D</i>	TiO ₂ — 97%	55.8
Kaolin	<i>China Clay Bavaria 800</i>	SiO ₂ — 49%; Al ₂ O ₃ — 35.7%	15.3
Barytes	<i>Fleur</i>	BaSO ₄ — 94%; SiO ₂ — 6%	19.2
Barium sulphate	<i>Blanc Fixe Powder N</i>	BaSO ₄ — 99%	16.5
Barytes	<i>Albaryt</i>	BaSO ₄ — 95%; SiO ₂ — 4%	29.4
Titanium dioxide	<i>Hombitan R610K</i>	TiO ₂ — 95%	49.6
Kaolin	<i>China Clay Bavaria 600H</i>	SiO ₂ — 49.3%; Al ₂ O ₃ — 35.8%	

Methods

The determination of plasticiser number

This was determined for di(2-ethylhexyl) phthalate, a plasticiser according to the conditions defined by British Standard⁵.

The preparation of plastisols

The pastes were mixed in a ball mill (Pulverisette) using the agate balls for 10 minutes at 200 revolutions per minute.

The following formula was used:

PVCl (Solids)—	100 pts by weight
DOP —	60 pts
Filler —	2; 5; 10 and 20 pts.

The determination of viscosity

The viscosity was measured in a cone-plate viscosimeter (Ferranti-Shirley), recording the function $\tau = f(D)$ in the range of shear rates from 0 to 1660 s⁻¹, during 20 seconds.

The apparent viscosity was calculated from the recorded curves at shear rate of 1660 s⁻¹.

Results

Figures 1 and 2 show the relationship between the apparent viscosity of pastes, containing respectively 10 and 20 per cent of the fillers listed in Table 1, and their plasticiser numbers.

The regression lines, calculated on the basis of the results of these determinations are described by the following equations:

$$\eta_{2 \text{ per cent}} = 0.000815PN + 3.30 \text{ Nsm}^{-2}$$

$$r = 0.775$$

$$\eta_{5 \text{ per cent}} = 0.01236PN + 3.411 \text{ Nsm}^{-2} \dots (1)$$

$$r = 0.751$$

$$\eta_{10 \text{ per cent}} = 0.02545PN + 3.348 \text{ Nsm}^{-2}$$

$$r = 0.944$$

$$\eta_{20 \text{ per cent}} = 0.04514PN + 3.463 \text{ Nsm}^{-2}$$

$$r = 0.942$$

where:

$\eta_{2 \text{ per cent}}$; $\eta_{5 \text{ per cent}}$; $\eta_{10 \text{ per cent}}$; $\eta_{20 \text{ per cent}}$

—are apparent viscosities of the plastisols containing respectively 2; 5; 10 and 20 parts of filler for 100 pts of PVCl,

PN

—is the plasticiser number,

r

—is a correlation coefficient between η_i and PN .

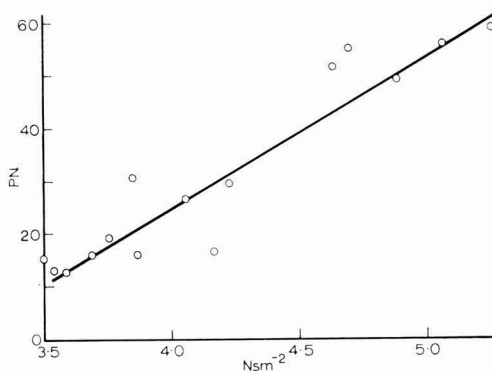


Fig. 1. The relationship between plasticiser number (PN) and viscosity of plastisols containing 10 parts of fillers

The above results indicate that the measurements for the pastes containing the smaller quantities of fillers are less accurate. This is easy to understand because relatively smaller quantities (2 and 5 per cent) cause little change in the viscosity; this affects the accuracy, as experimental error is constant. On the other hand, it is seen that the viscosity of pastes containing 10 and 20 per cent of fillers depends directly on the plasticiser number on account of the high correlation coefficient r .

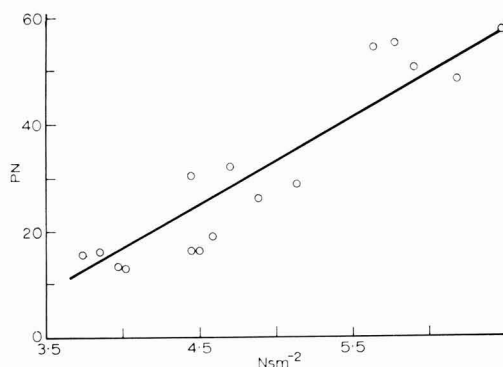


Fig. 2. The relationship between plasticiser number (PN) and the viscosity of plastisols containing 20 parts of filler

The values of the constants (3.30; 3.411; 3.348 and 3.463 Nsm^{-2}) show the calculated viscosity of the plastisol without filler. This value, measured according to the method used, is, in fact, equal to 3.32 Nsm^{-2} . Thus, the maximum error of the calculated results at γ_{20} per cent is 4.3 per cent and average standard deviation $s = 0.0999 \text{ Nsm}^{-2}$.

The equation (1) can be given in the general form:

$$\eta_{fc} = x \cdot PN + \eta_p \dots \dots \dots (2)$$

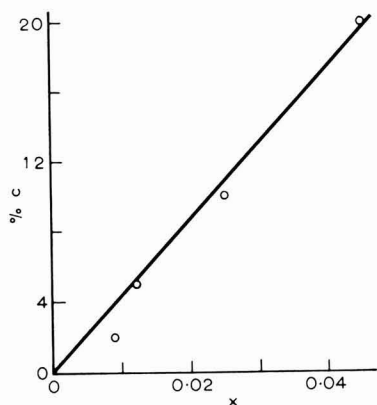


Fig. 3. The relationship between the concentration of filler (c) and multiplier (x)

where:

η_{fc} — is the apparent viscosity of the plastisol containing the filler in concentration c ,

x — is a multiplier, depending on the concentration of filler c ,

PN — is the plasticiser number,

η_p — is the apparent viscosity of the plastisol, when $c = 0$.

Figure 3 shows the relationship between the multiplier x and the concentration of filler c .

It was calculated that the correlation coefficient for these two values is 0.998, so these values can be used for the calculation of the multiplier x . The values found for a 2 per cent concentration of fillers were ignored because of their higher divergence.

If the calculated value of the multiplier x is used, the following form of equation (2) was obtained:

$$\eta_{fc} = \frac{c}{413.5} PN + \eta_p \dots \dots \dots (3)$$

where:

c — is the concentration of filler in weight per cent of PVC

The general form of this equation can be shown as:

$$\eta_{fc} = \frac{c}{a} PN + \eta_p \dots \dots \dots (4)$$

where:

a — is the coefficient depending on the rate of shear.

Recapitulating the above, it is found that equation (4) can be used for the calculation of viscosity of PVC plastisols containing any concentration of filler, but owing to the fact that Newton's Law is not valid for PVC plastisols, the apparent viscosity should be calculated at a definite shear rate after the determination of coefficient a . This is easy to determine for any process conditions by measuring η_{fc} ; c ; PN and η_p . Further data for viscosity at the constant rate of shear can be determined with a large degree of accuracy from equation (4) for any η_p and chosen concentration c of any filler.

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A new method for determining dispersibility of pigments and the optimal mill base formulation*

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Summary

After a short discussion on the method of the dispersion curve for the evaluation of the dispersibility of pigments, an attempt is made to establish the dispersibility by means of a new criterion, the "effectiveness", which takes into consideration not only the energy expenditure, but also the amount of dispersed pigment. By means of grinding tests it is shown that for every pigment/vehicle system there is an optimum proportion for the economy of the milling process or, in other words, an 'optimal effectiveness'.

The physical factors determining the shape of the 'effectiveness'

curve are briefly discussed and a method is suggested to obtain the optimal effectiveness with a lesser effort. If the volume ratios, instead of the weight ratios, are considered for the calculation of the effectiveness it can be shown that the effectiveness optimum in a given vehicle solution is variable within a narrow range for most pigments.

Further research has shown that the effectiveness curve can also be obtained by using the development of tinting strength instead of the fineness of grind as criterion for the dispersibility.

Keywords

Properties, characteristics and conditions primarily associated with materials in general

dispersibility

raw materials for coatings and allied products

tinctorial strength

bulk coatings and allied products

pigment volume concentration

Raw materials

prime pigments and dyes

prime pigments and dyes

Miscellaneous terms

mill base

Une nouvelle méthode pour déterminer la facilité de dispersion de pigments et la composition optimale de la masse broyante

Résumé

Après une brève discussion de la méthode basée sur la courbe de dispersion pour évaluer la facilité de dispersion de pigments, on essaie d'établir cette facilité au moyen d'un nouveau critère, "l'efficacité", qui tient compte non seulement de la consommation d'énergie, mais aussi de la masse du pigment dispersé. Au moyen des essais de broyage on démontre, dans le cas de chaque système pigment/véhicule, qu'il existe un rapport optimal à l'égard de l'économie du procédé de broyage, ou autrement dit, "une efficacité optimale".

On discute brièvement les facteurs qui déterminent le profil de la

courbe "d'efficacité" et l'on propose une méthode pour obtenir l'efficacité optimale par un effort diminué. Si l'on utilise les rapports par volume au lieu de par poids en faisant le calcul de l'efficacité, on peut démontrer au point de vue de la plupart de pigments que l'efficacité optimale à l'égard d'une solution de véhicule spécifique ne se varie qu'entre les limites assez proches.

Une étude de plus a démontré que la courbe d'efficacité peut aussi être obtenue en utilisant le développement du pouvoir colorant au lieu de la finesse de broyage en tant que critère de la facilité de dispersion.

Eine neue Methode zur Bestimmung der Dispergierbarkeit von Pigmenten und der optimalen Rezeptur des zu mahlenden Anteils

Zusammenfassung

Nach kurzer Besprechung der Methode der Dispergierungskurve für die Beurteilung der Dispergierbarkeit von Pigmenten wird versucht, die Dispergierbarkeit mit Hilfe einer neuen Kennzeichnung, der "Effectiveness" auszudrücken, welche nicht nur den Energieverbrauch, sondern auch die Menge des dispergierten Pigments in Betracht zieht. An Hand von Mahltesten wird gezeigt, dass für jedes Pigment/Lösungsmittel enthaltendes Bindemittelsystem ein optimales Verhältnis für die Wirtschaftlichkeit des Mahlvorganges besteht, in anderen Worten eine "optimale Effectiveness".

Die physikalischen Faktoren, welche die Form der "Effectiveness" Kurve bestimmen, werden kurz besprochen, und eine Methode

vorgeschlagen, mittels welcher die optimale "Effectiveness" mit geringerem Aufwand erhalten werden kann.

Wenn anstelle von gewichtsmässigen volumenmässige Proportionen für die Berechnung der "Effectiveness" in Betracht gezogen werden, kann aufgezeigt werden, dass die optimale "Effectiveness" in einer gegebenen Bindemittellösung für die meisten Pigmente innerhalb enger Grenzen variierbar ist.

Weitere Untersuchungen zeigten, dass die "Effectiveness" Kurve auch bei Benutzung der Entwicklung von Färbekraft anstelle von Mahlfineinheit als Kennzeichen der Dispergierbarkeit erhalten werden kann.

*A paper on this subject appeared in *Farbe und Lack* 1973, 79, 518, to whom we are indebted for permission to reproduce certain parts which are common to both papers.

Introduction

Ref. 1

Among the direct methods for determining the dispersibility of pigments, that of the dispersion curve, according to Hegman, is one of the most commonly used, as it is quick and easy to carry out. For this purpose the fineness of grind is usually plotted as a function of the energy expenditure expressed indirectly as time in the case of dissolvers or ball mills, or as the number of passes on the three-roll-mill. Such a graph is called a dispersion curve. If two such dispersion curves are compared with each other, that pigment which demands the lesser energy expenditure to attain the desired fineness of grind is considered to be more easily dispersible.

A study of this method raises a question regarding which of the dispersing conditions should be kept invariable. It is evident that the binder solution should be the same in all tests and that the physical factors implied by the dispersing device, such as ball size and specific gravity, ball mill volume ratios, pressure between the rolls of the three-roll-mill, must also be kept constant. However, in relation to the pigment concentration, there are two possibilities:

Using the same pigment concentration

In this case it is possible to compare directly the energy expenditure required to disperse a given amount of pigment. However, this method does not take into account the different rheological behaviour of the pigments which has a considerable influence on the dispersing process, especially in the case of ball mills. This fact may have a misleading effect on the results.

Using the same consistency

Here the pigment concentration is variable. The mill base consistency chosen depends on the type of dispersing device. This method is more adequate because it takes the different rheological behaviour of the pigments into account. Nevertheless, its application may imply two sources of error:

- The determination of the proper pigment concentration in the mill base is usually carried out following the Guggenheim method, which is based on the flow point at different binder concentrations according to Daniel and Goldman¹. Nevertheless, with many pigments, especially organic pigments, the flow point cannot be established with the required accuracy. Anomalous flow of the paste may lead to an erroneous flow point.
- The mill base consistency does not remain constant during the milling process, so that the pastes may be milled under quite different conditions of consistency, even when the flow point and the mill base composition have been accurately determined initially.

By virtue of these considerations it was decided to search for a direct method for determining the dispersibility of pigments which would be more reliable than the dispersion curve. The basic idea was to find out which would be the optimum pigment concentration in the mill base in order to attain the best dispersion result by means of tests carried out in the ball mill itself.

Experimental

Refs. 2, 3

The trials were carried out with steatite ball mills having an internal volume of 250 ml. They contained 44 ml of steatite pebbles of 8 mm diameter. The mill base volume was 90 ml

in all tests. It was known that this was not the optimum volume ratio for the ball mill; however, it was chosen in order to be able to record small differences by a sort of 'slow-motion effect'.

A manganese lake of Pigment Red 52 was milled at nine different pigment weight concentrations (PWC) in a binder solution consisting of 30 per cent of a soya bean and linseed oil modified alkyd resin in white spirit. The rotation rate of the mills was 90 rpm. In all tests the running time necessary to attain a fineness of grind of 10 μm was determined. The values thus obtained are shown in Table 1.

Table 1
Milling time for Pigment Red 52-Mn lake in an air-drying system at different pigment weight concentrations to attain a fineness of grind of 10 μm

PWC %	Milling time h
3	15.5
6	16
9	16.5
12	17.75
14	19
16	21.5
18	25
20	32
23	58

The determined values were plotted. Because of the errors implied in the measurements on the fineness of grind gauge, the values were not entered as points, but as short lines. Thereafter, the fitted curve resulting from these values was drawn. (Figure 1a)

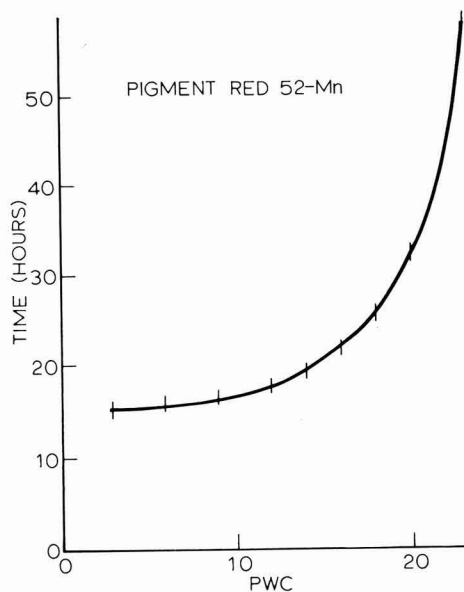


Fig. 1(a)

If the dispersibility is judged as dependent only on the milling time, test No. 1 (containing 3 per cent pigment) would be considered as the best. Nevertheless, this would be wrong. The short milling time in this case cannot be attributed to a

favourable formulation of the mill base, but to the low pigment content. From the economic point of view, which is probably one of the most important for the processing industry, a pigment can be considered as easy dispersible if a large amount of it can be dispersed in a relatively short time. This means that in order to judge the dispersibility in a proper manner, not only the milling time, but also the amount of pigment dispersed, must be considered.

Consequently, each milling process has a degree of effectiveness which is characteristic of its efficiency. This effectiveness is directly proportional to the amount of pigment dispersed and inversely proportional to the required milling time.

Thus:

$$\text{Effectiveness} = k_1 \cdot k_2 \frac{\text{pigment concentration}}{\text{milling time}}$$

If $k_1 \cdot k_2$ is written as K

it can be said that the effectiveness E is directly proportional to the ratio between pigment concentration PC and milling time t .

$$E = K \frac{PC}{t}$$

The resulting proportionality constant K is doubtless a characteristic of each combination of pigment/vehicle solution.

Now it becomes evident that the formulation attaining the highest value for E is the best one. This optimal value for E was determined with the help of the graph (Figure 1b). For this different pairs of values from the fitting curve were taken and the corresponding effectiveness was calculated. Thus, the values shown in Table 2 were obtained.

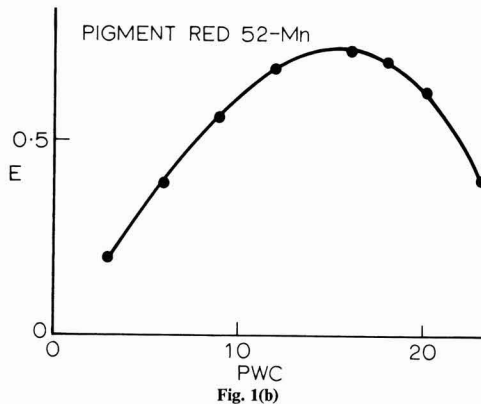


Table 2

Effectiveness as a result of the pigment weight concentration and the milling time

PWC	$\frac{t}{h}$	Effectiveness
3	15.4	0.195
6	15.6	0.385
9	16.1	0.560
12	17.5	0.686
14	19.2	0.730
16	21.8	0.734
18	25.6	0.703
20	32.0	0.625
23	58.0	0.397

The calculated effectiveness values were plotted in a new graph against the pigment concentration and connected with each other by means of a fitted curve (Figure 1b). At the abscissa value of 15.4, the curve presents the maximum value of 0.736. This is the 'optimal effectiveness' for the pigment tested under the milling conditions described.

The course of the effectiveness curve is evidently similar to a bell-shaped Gaussian distribution curve. Such curves always emerge when the process they represent is determined by chance. In this case this could be explained as follows:

The more the pigment concentration increases, the greater is the probability that pigment agglomerates get caught between two balls and broken down. However, at the same time, the influence of the increasing viscosity makes itself felt by slowing down the fall speed of the balls. This causes the frequency of the impacts to drop and the number of ground agglomerates in relation to the pigment concentration to decrease. Beyond a determined pigment concentration the negative influence of the growing viscosity becomes greater than the positive effect of the increasing pigment concentration. Eventually, the high viscosity causes the milling process to come to a halt. If the viscosity and the tendency to re-agglomeration had no influence on the milling process, the effectiveness would rise linearly with the pigment concentration.

Řehaček² has already shown in his investigations on pigment dispersion by means of ball mills that the dispersion speed drops after the pigment concentration has exceeded a certain value.

Since the effectiveness is a measure of the economics of the milling process, the optimal effectiveness is at the same time a measure of the dispersibility of pigments. If several types of pigment are tested in the same binder solution, the product yielding the highest value for the optimal effectiveness will be the most easy to disperse.

Based on these considerations, fifteen pigments were tested for their dispersibility. Thus the optimal values for the effectiveness were obtained as shown in Table 3 (see also Figs. 1 to 15)

Table 3
Determined optimal effectiveness for different pigments

Pigment	Optimal PWC %	Optimal effectiveness	Fig.
Cu-Phthalocyanine blue I	15.4	0.52	2
Pigment red 52-Mn	15.4	0.74	1b
Cu-Phthalocyanine blue II (easy dispersible)	16.4	0.82	3
Toluidine red	26.0	0.92	4
Iron oxide yellow (transparent)	27.6	0.99	5
Prussian blue (micronized)	21.5	1.05	6
Chrome yellow I	36.0	1.65	7
Pigment red 48-Mn	15.2	1.69	8
Molybdate red I	36.8	1.72	9
Cadmium red	36.5	1.86	10
Nickel titanium yellow	37.0	2.01	11
Cobalt blue I	36.7	2.05	12
Chrome yellow II (easy dispersible)	45.2	3.42	13
Titanium dioxide (micronized)	50.4	3.60	14
Molybdate red II (easy dispersible)	36.9	4.21	15

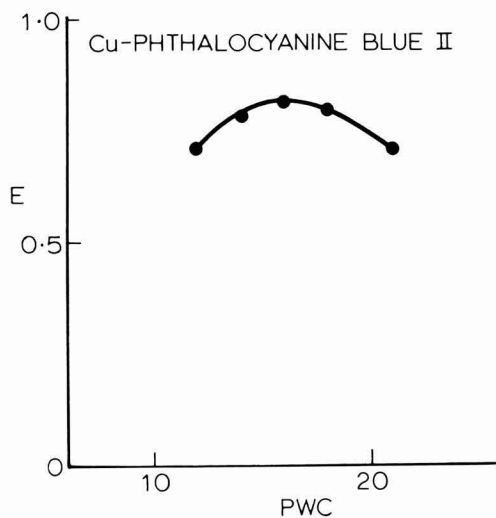


Fig. 2

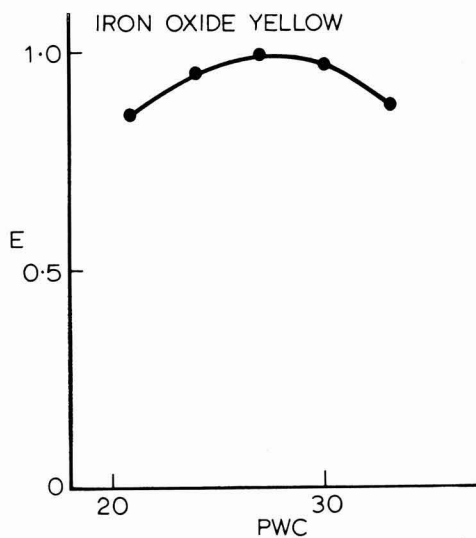


Fig. 5

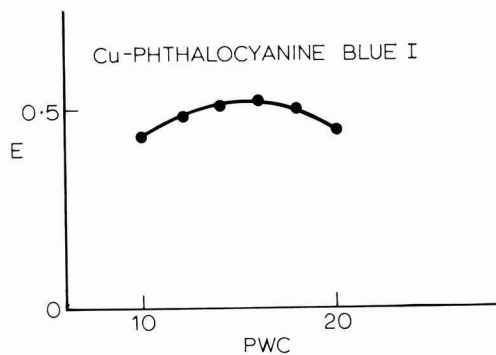


Fig. 3

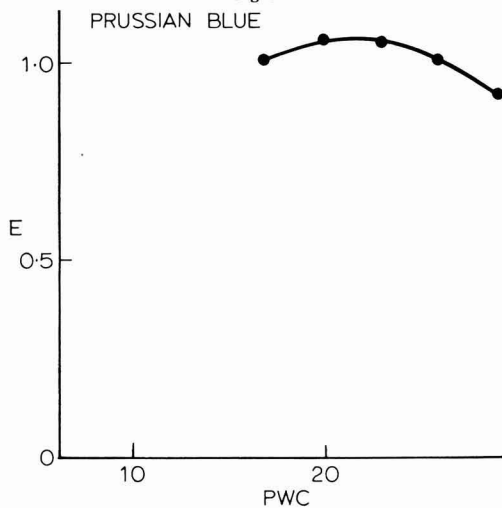


Fig. 6

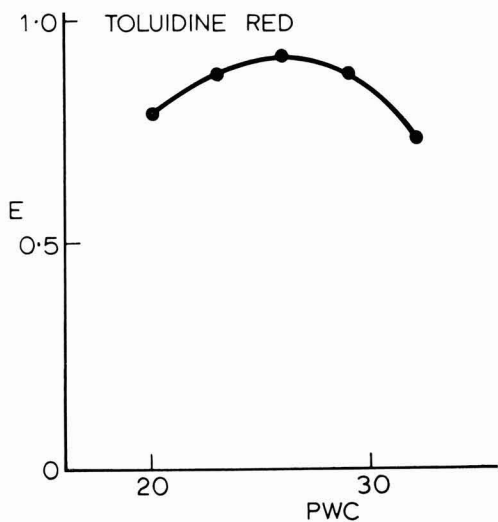


Fig. 4

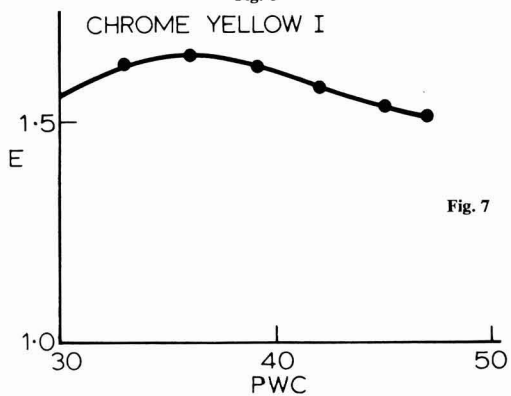


Fig. 7

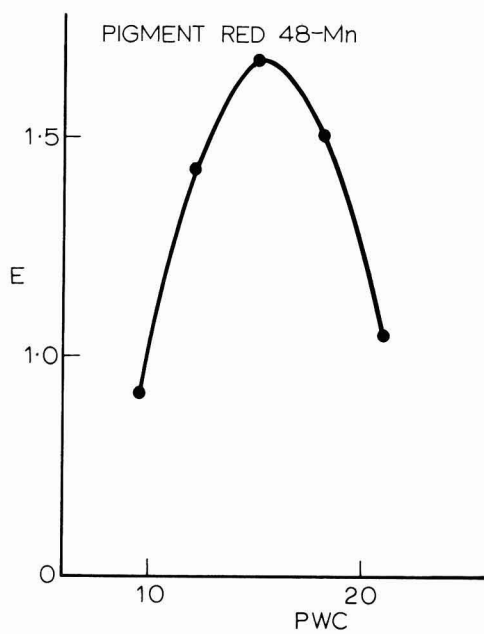


Fig. 8

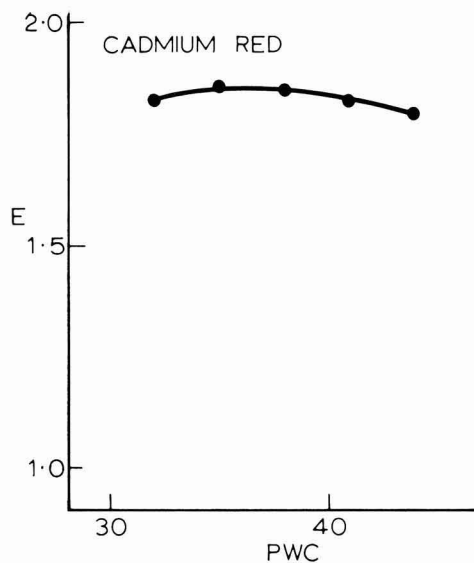


Fig. 10

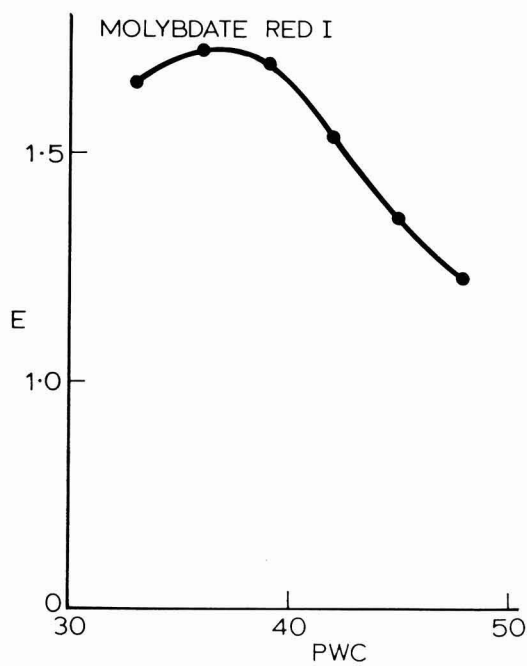


Fig. 9

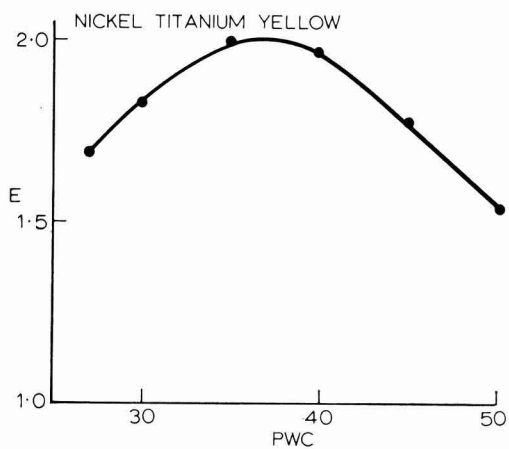


Fig. 11

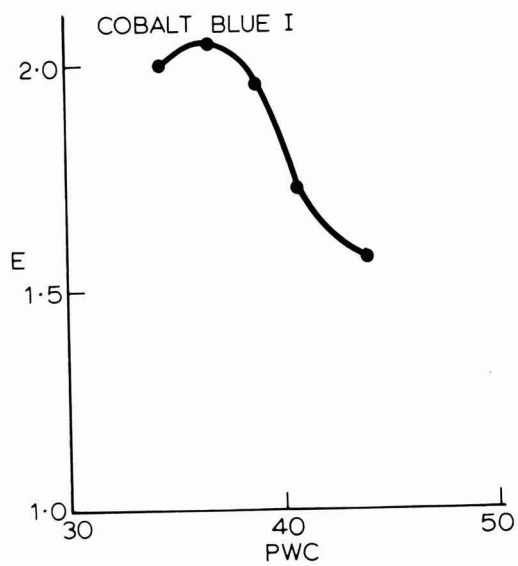


Fig. 12

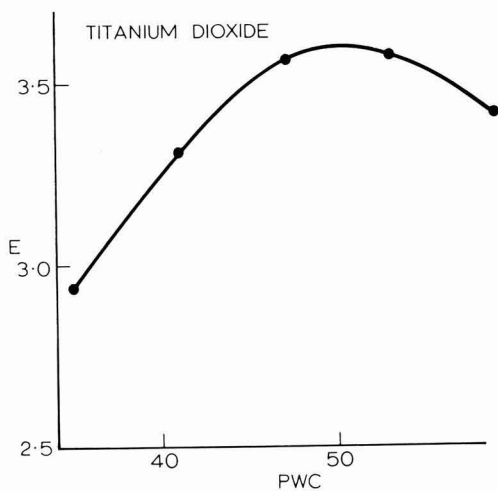


Fig. 14

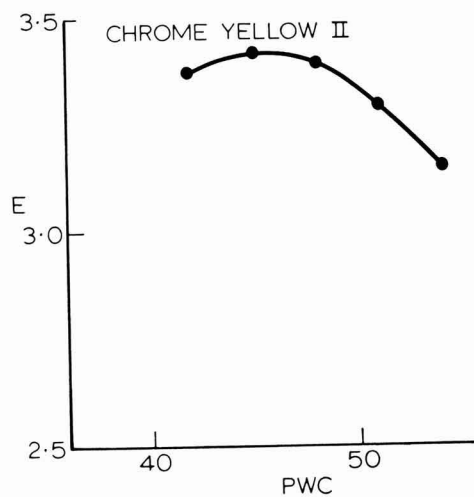


Fig. 13

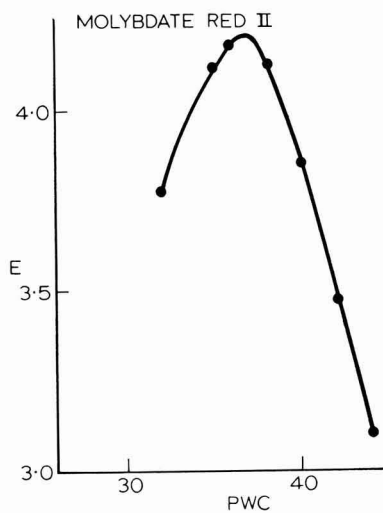


Fig. 15

The procedure is somewhat expensive, since at least five or six trials must be performed in order to determine the optimum effectiveness with reliable accuracy. Nevertheless, with the help of some calculations a way to do it with only three trials was found.

The upper part of the bell-shaped curve, the part which is of interest for the evaluation of the optimum effectiveness, corresponds very nearly to a parabola with its vertex turned upwards. Such a parabola has the basic equation:

$$y = -ax^2 + bx + c$$

If P = pigment concentration

and E = effectiveness

the equation in this case would be:

$$E = -aP^2 + bP + c$$

Now, in order to be able to find for every pigment/binder solution system an equation showing the dependence of the effectiveness on the pigment concentration, the values of the constants a , b and c must be found. For three unknowns three equations are needed for the solution. For this reason three trials at least are needed.

Consider, for instance, the case of nickel titanium yellow. Taking into account the results of three trials only, the equations are:

$$1.82 = -30a^2 + 30b + c \dots \dots \dots (1)$$

$$2.00 = -35a^2 + 35b + c \dots \dots \dots (2)$$

$$1.96 = -40a^2 + 40b + c \dots \dots \dots (3)$$

When solving this system of equations, the following values for a , b , and c were obtained:

$$a = 0.0044$$

$$b = 0.3220$$

$$c = -3.8800$$

Consequently, for nickel titanium yellow the equation is:

$$E = -0.0044P^2 + 0.3220P - 3.8800$$

The optimum effectiveness is represented in the parabola by a maximum; for this reason the first differential quotient of the equation at that point must be zero. The differentiation with respect to P of the equation

$$E = -aP^2 + bP + c$$

$$\text{gives } \frac{dE}{dP} = -2aP + b = 0$$

$$\text{or } P_{op} = \frac{b}{2a}$$

Thus in the case of nickel titanium yellow the optimal pigment concentration is:

$$P_{op} = \frac{0.3220}{0.0088} = 36.59 \approx 36.6$$

Therefore, the optimum effectiveness will be:

$$E_{op} = -0.0044 \cdot 36.6^2 + 0.3220 \cdot 36.6 - 3.8800$$

$$E_{op} = 2.0115 \approx 2.01$$

This value which was found on the basis of only three trials coincides with the value determined graphically using the results of six trials. A comparison between the optimum effectiveness values obtained in this manner and the values found graphically is shown in Table 4.

Table 4
Values for the optimum effectiveness of different pigments obtained graphically and by calculation

Pigment	Optimal effectiveness	
	Graphically	Calculated
Cu-Phthalocyanine I	0.52	0.52
Pigment red 52-Mn	0.74	0.74
Cu-Phthalocyanine II (easy dispersible)	0.82	0.82
Toluidine red	0.92	0.92
Iron oxide yellow (transparent)	0.99	0.99
Prussian blue (micronized)	1.05	1.05
Chrome yellow I	1.65	1.65
Pigment red 48-Mn	1.69	1.69
Molybdate red I	1.72	1.72
Cadmium red	1.86	1.86
Nickel titanium yellow	2.01	2.01
Cobalt blue I	2.05	2.05
Chrome yellow II (easy dispersible)	3.42	3.42
Titanium dioxide (micronized)	3.60	3.60
Molybdate red II (easy dispersible)	4.21	4.22

For the calculation of the optimum effectiveness the upper section of the bell-shaped curve has been considered as a parabola. For this reason the further the values chosen for the evaluation differ from the vertex, the more the line obtained differs from a parabola and the greater is the error resulting in the calculation. For example, in the case of nickel titanium yellow the abscissa values 30, 35 and 40 were used and an optimum effectiveness of 2.01 was obtained. However, if the abscissa values 27, 30 and 35 had been taken, an optimum effectiveness of 2.27 would be the result. Because of this it is advisable to choose the pigment concentrations in the neighbourhood of the optimum region. The best result would be given if the vertex is within the chosen range. In other words the calculation of the optimal effectiveness should be achieved by interpolation and not be extrapolation.

Of course, when a pigment is first tested in a given vehicle solution, it is not possible to know beforehand which pigment concentration region is the right one. In this case, it is best to carry out several trials and determine the optimum effectiveness graphically. But when the object is to control different batches of the same product or to test different commercial grades of chemically identical pigments to check dispersibility, only initially are several tests required. For all the comparisons which follow, the optimal concentration region is already known and three trials are sufficient. At any rate, it is advisable to confirm the calculated optimum effectiveness by carrying out a test at the optimal pigment concentration obtained. The value determined experimentally should coincide or only slightly deviate from the calculated one.

According to the shape of the effectiveness curve the pigments tested can be classified in three groups:

Group A consists of pigments showing a flat curve with a wide optimum region. These are:

Cadmium red
Chrome yellow I
Prussian blue
Cu-Phthalocyanine blue I

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Organics Division



Hexagon House, Blackley, Manchester M9 3DA.

The types of Group *B* yield a steep curve with a narrow optimum range. These types are:

Cobalt blue I
Molybdate red II
Pigment red 48-Mn

In Group *C* the shape of the curves lies between Groups *A* and *B*. To this group belong:

Toluidine red
Iron oxide yellow
Nickel titanium yellow
Chrome yellow II
Titanium dioxide
Cu-Phthalocyanine blue II
Pigment red 25 - Mn

In the case of the products classified in Group *A*, it is possible to diverge slightly from the optimal concentration without affecting the economics of the milling process significantly. With certain reservations, this is also possible for the products classified in Group *C*. In contrast to this, with the pigments belonging to Group *B* it is absolutely necessary to stay within the narrow optimal field, otherwise high production costs will result.

The optimum effectiveness values of different pigments can be compared with each other only if they have been determined under the same conditions. Tests in other vehicles or other dispersion devices will yield different values. In addition to the tests already mentioned, four of the pigments were tested in a stoving lacquer based on a combination of a short oil alkyd and a melamine resin. A comparison of the results is shown in Table 5.

Table 5

Graphically determined optimum effectiveness of four pigments in a medium oil alkyd resin in white spirit (system A) and in a stoving system based on a short oil alkyd and melamine resin in xylol/butanol (System B), both containing 30% solids

Pigment	System A	System B
Pigment red 52-Mn	0.74	0.71
Cadmium red	1.86	1.76
Nickel titanium yellow	2.01	1.93
Molybdate red II	4.21	3.71

In the stoving lacquer, all four pigments show a slightly more unfavourable dispersibility.

To what extent can the results obtained by means of the ball mill be related to high speed dispersing devices like Perl or sand mills?

When confronted for the first time with this question it was difficult to form an opinion on the rapid-motion of these types of devices causing slight and medium differences to disappear.

On the other hand, the viscosity of the mill base in these continuously running machines must be kept relatively low in order to guarantee a constant pumping and flow-rate. However, it can now be asserted that there should be no difficulty in predicting the behaviour of pigments in high-speed dispersing devices from results obtained with laboratory ball mills. This assertion is based on the following:

A large pigment producer in West Germany delivered pre-dispersed iron oxides as pastes which were processed in Perl mills. The vehicle was a medium oil alkyd resin solution

in a mixture of hydrocarbons. Since the pastes were intended for automotive paints, heavy demands were made as to their quality. To fulfil these high demands the production batches had to undergo up to eighteen passes through the Perl mill. Using the method described here the optimal effectiveness and the corresponding optimal pigment concentration were determined in the laboratory by means of ball mills. After the formulations in the plant had been modified, the pastes could be produced with only nine passes of the batches through the mill. The quality remained unchanged.

When considering the volume ratios instead of the weight ratios in the mill base for the calculation of the effectiveness, different values are obtained. In the case of the pigments tested the values are shown in Table 6.

Table 6

Optimal pigment volume concentration (PVC) and the corresponding optimum effectiveness of different pigments

Pigment	Optimal PVC %	Optimum effectiveness
Iron oxide yellow (transparent)	8.8	0.295
Cu-Phthalocyanine blue I	10.0	0.305
Molybdate red I	8.2	0.345
Chrome yellow I	10.9	0.410
Pigment red 52-Mn	10.4	0.475
Cadmium red	10.6	0.480
Cu-Phthalocyanine blue II (easy dispersible)	10.3	0.490
Prussian blue (micronised)	10.5	0.500
Nickel titanium yellow	9.7	0.530
Cobalt blue I	10.3	0.545
Toluidine red	17.3	0.615
Chrome yellow II (easy dispersible)	13.3	0.890
Molybdate red II (easy dispersible)	8.5	0.890
Pigment red 48-Mn	10.1	1.025
Titanium dioxide	20.7	1.385

The effectiveness curve as a function of the PVC for Pigment red 48-Mn is shown in Fig. 16.

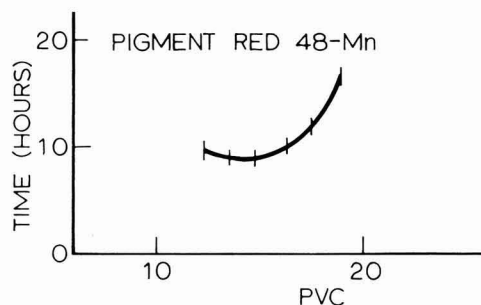


Fig. 16(a)

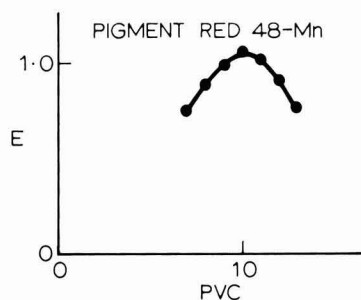


Fig. 16(b)

The determination of the optimum effectiveness taking as a basis the volume ratios in the mill base results in some interesting changes of position. Molybdate red II is now inferior to titanium dioxide and on a par with chrome yellow II. Iron oxide yellow is the most difficult to disperse amongst all the tested pigments; the micronised prussian blue is almost as good as nickel titanium yellow and the Mn-lake of Pigment red 48 is second only to titanium dioxide.

On the other hand, it is noteworthy that for most of the tested pigments PVC values of about 10 were obtained. To confirm this six further products were tested which yielded the results shown in Table 7.

Table 7

Optimal PVC and corresponding optimum effectiveness of different pigments

Pigments	Optimal PVC	Optimum effectiveness
Chrome oxide green	10.0	0.365
Cobalt green	10.3	0.415
Cobalt blue II	12.6	0.480
Iron oxide red	11.0	0.510
Ultramarine blue	10.8	0.560
Cadmium yellow	10.1	0.590

Evidently there exists an optimum viscosity range which determines the PVC of the mill base. For most pigments the optimal PVC varies within a narrow range, in this case between 8 and 11 per cent.

There are, however, products which exhibit rheological characteristics which may affect positively or negatively the milling process, resulting in a higher or lower PVC than the average. This different rheological behaviour can probably be attributed to a special surface property (conditioned for example by finishing) and can vary according to the vehicle. It is not dependent on the specific surface alone.

It had been intended to carry out further tests in order to determine if possible the optimum effectiveness using the

development of tinting strength according to von Pigenot³ instead of the fineness of grind, but the work had to be finished at this stage. Fortunately a paint manufacturer in West Germany was able to show that optimum effectiveness and optimal pigment volume concentration can also be determined by the development of tinting strength.

Conclusions

A suitable method has been found for determining the dispersibility of pigments, which takes into account the characteristic behaviour of each product. Every pigment is tested under the conditions determined by its inherent properties, and the values found under the optimal conditions are compared. In the authors' opinion, fair comparisons are only possible in this way.

The method can also be employed to establish optimal mill base formulations. In this case not only is the pigment concentration varied, but also the vehicle/solvent ratio.

Results obtained with laboratory ball mills can be applied for plant ball-, Perl- or sand mills.

The optimal effectiveness can also be determined if the development of tinting strength instead of the fineness of grind is considered.

[Received 23 August 1977]

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4. Patton T. C. "Paint flow and pigment dispersion", *John Wiley and Sons, Inc.*, 1964 New York.
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Next month's issue

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

New developments in ultraviolet curable coatings technology by C. B. Rybny and J. A. Vona

Cure behaviour of photopolymer coatings by R. Holman and H. Rubin

Photoinitiator problems in clear coatings by M. De Poortere, A. Ducarme, P. Dufour and Y. Merck

A new simple method of determining the oxirane oxygen content of vegetable oils

SIR—I have read the paper by Badran on the NMR method for estimating oxirane oxygen content in vegetable oils, published in *JOCCA*, 1978, **61**, 52 with much interest and found the values obtained interesting. However, the use of NMR spectroscopy is rather common for the estimation of the epoxy equivalents in the epoxy polymers, such as epikotes.

It has been suggested that the method has several advantages compared to the other methods, because of its simplicity and accuracy. However, there is no doubt about the accuracy of the method, but the main difficulty arises in obtaining a good NMR spectra which needs very careful sample preparation. This in turn limits the application of such a sophisticated method in industrial laboratories. Therefore, I consider that the chemical method^{1,2} for the estimation of oxirane oxygen in such products is the most simple and perhaps the most useful one. This method has been shown to give reproducible and accurate results with an error of less than one per cent.

SIR—Dr S. Paul refers in his letter to the NMR method published in *JOCCA*, 1978, **61**, 52. Some difficulty arose when I was treating epoxidised linseed oil with *p*-substituted aniline and I was unable to follow the reaction which took place by the usual volumetric methods, such as Nicolet's, Swern's, Jay's and Durbetaki's. The aim of the work was to study the reaction of the amines with epoxidised oil (*Europ. Poly. J.* 1977, 155) and to produce a bifunctional product which could act as an antioxidant-plasticiser for rubber mixes and vulcanisates (*Brit. Poly. J.*, submitted). I believe that the volumetric methods are not satisfactory in all cases and other methods should be available. The NMR method is not sophisticated, but it was one of the alternatives proposed.

With respect to the method proposed by Dr Paul, I would consider that it is not suitable for my study, and also that all the volumetric methods would fail in this case because they are based on hydrohalogenation; hydrogen halides react with amines to form amine salts.

The chemical method comprises mainly of the hydrochlorination of the oxirane groups in aqueous or dimethylformamide (DMF) solutions of the sample, followed by the determination of the free chloride ions by Mohr's method, after neutralisation of the free acid groups with anhydrous CaCO_3 . This method has been found to give inaccurate values only in the presence of primary amines, since the latter hinder the hydrochlorination reaction which, in turn, necessitates a longer hydrochlorination period.

Yours faithfully,

SWARAJ PAUL

AB Wilh. Becker,
Resin Development Dept.,
Fach, S-10270
Stockholm 9,
Sweden

3 March 1978

References

1. Paul S. and Ranby B., *J. Polym. Sci.-Polym. Chem.*, 1976, **14**, 2449.
2. Iwakura Y. et al., *Makromol. Chem.*, 1966, **97**, 128.

Dr Paul stated that in order to obtain a good NMR spectra, very pure samples must be prepared, and that this limits the application of the NMR method. To the best of my knowledge, the value of oxirane oxygen content obtained depends principally on the purity of the sample to be measured, whether the chemical or the NMR method is used.

I admit that it was my mistake to concentrate my laboratory work on epoxidised vegetable oils and not on epoxy resins, and would like to thank Dr Paul for his valuable comments.

Yours faithfully,

B. M. BADRAN

National Research Centre,
Laboratory of Polymers and Pigments,
Sh. El-Tahrir,
Dokki,
Cairo,
Egypt

15 March 1978

Review

Polymer rheology

By L. E. Nielsen

Marcel Dekker, Inc. NY and Basel, 1977

Pp x + 203. Price U.S. \$17.50

This book, the author states, emphasises general principles and practical implications of all phases of polymer rheology for people working practically with plastics and rubbers.

There are 203 pages, which include an author and subject index. The subject index is too small to be useful, but the author index is much larger and comprehensive. The chapters are short and each one has an extensive bibliography. Among the chapters are the relatively newer subjects of extensional viscosity and the rheology of powder and granular materials. Although extensional viscosity is only of academic interest to the paint technologist, the chapter on powder flow is of interest to those working with powder coatings. The intro-

ductory chapter and the following one on instruments are short and perhaps too concise, but the subject matter has been dealt with many times before.

For the paint technologist there is an interesting chapter on suspensions and paragraphs on emulsions, latices and plastisols which are far too brief.

The style of writing is clear and concise, but in an effort to explain complex phenomena simply, it can sometimes cause annoyance to the reader. The book, however, summarises well some of the important aspects of the subject, although thixotropy is not discussed.

Mathematics is confined to final equations without proofs and the clear and informative graphs are well drawn from plastic-polymer data.

The book is useful for explanations of rheological behaviour and a handy reference to some of the simpler equations and relationships that have been found.

M. Camina

Manchester

Purchasing strategy

Mr D. F. ("Doug") Brocklehurst of Berger Group Supplies Ltd delivered a lecture entitled "Purchasing strategy in the chemical industry with reference to paint" to 50 members and visitors at the Manchester Literary and Philosophical Society, George Street, Manchester, on Friday 10 February, 1978.

Indicating that his lecture was based on personal views, the speaker began by stating that purchasing should not be based on costs alone, but should be a balance between purchasing, technical, production and marketing and that strategy must change as marketing changed. He admitted only gloom on future prospects and stressed that the decorative paint section of the industry had been reduced to the level of the commodities market, whereas the industrial paint section was relatively profitable. Claiming that research and development follows the Law of Diminishing Returns, he quoted the decline in USA Patent applications, and on this theme stated: "Scientists publish—Technologists patent".

Stimulating statements such as "Lost sales have influenced formulations more than technical innovation" were followed by his general advice to choose supplies based on sound technology, GP grades and compilation of a standard material list. It should be noted that Mr Brocklehurst was not entirely gloomy and his many anecdotes ranged from the Papal to the Martian!

A lively question period encompassing the subject of technology and the increasing trend of chemical companies purchasing paint companies was followed by a vote of thanks proposed by Mr W. McCullum, which was well received.

Student film evening

This student function was held at the Manchester Literary and Philosophical Society, on Thursday 23 February, 1978, at 4.30 p.m.

Fifty students, Ordinary members and visitors were present to hear Mr G. T. Flood introduce three films which were the highlight of the Section's student programme.

The first film was simply entitled "Paint" and described in detail the growth of the title subject from the early cave dwelling artists to modern times. The second was also a "Shell" production, and its title of "Epikote anti-corrosive paints" fully illustrated their use in oil refinery, chemical and marine environments.

The final film entitled "Email facade—France" was basically case histories of Farbenfabriken Bayer based coatings applied to building surfaces in France by Etan Protec. This film confirmed the viability of using long-life maintenance-free surface coatings for the protection and improvement in appearance of large building complexes.

F.B.W.

Transvaal

Sasol II

A meeting of the Transvaal Section was held at the Devonshire Hotel, Johannesburg on 24 November 1977 when Dr A. Brink, Head of the Research and Development

Department, SA Coal, Oil and Gas Corporation Ltd, delivered a lecture entitled "Sasol II with special reference to its significance for the surface coatings industry".

The Fischer-Tropsch synthesis, as practised at Sasolburg, is the only commercial oil-from-coal venture in the world at present. Although the F-T synthesis is mainly intended as a producer of fuels, it also is a source of petrochemicals and petrochemical precursors, and the relative importance of the latter outlets may increase in future.

Synthesis gas for the F-T synthesis is generated in Lurgi gasifiers and after purification is converted to a mixture of hydrocarbons, ranging from methane to waxy oils, over a promoted iron catalyst in the Synthol reactors. The hydrocarbons are mostly aliphatic in nature and highly olefinic. The aromatic content is low in the petrol range but increases with carbon number. Some oxygenates, mainly alcohols, ketones and fatty acids are also produced.

By-products from gasification include phenols, recovered by solvent extraction from the gas liquor, and tar, from which naphthas, rich in benzene, toluene and xylene, creosotes and pitch, are obtained.

The major building blocks for the production of petrochemicals are ethylene, propylene, the butenes, benzene, toluene and xylene. These are all produced from an oil-from-coal plant and, with the expanded production from the Sasol II plant, there is an abundance of these raw materials. Add to these the phenols recovered from the gas liquor and the olefins from the Synthol oil, which with some purification can constitute the raw materials for oxo-alcohols and detergents, and it can be seen that an oil-from-coal operation is a source of almost all the major chemical building blocks that go into the surface coatings field: polymers, resins, driers and solvents.

P.A.J.G.

Midlands

Trent Valley Branch

Anticorrosive primers

A joint meeting of the Trent Valley Branch and the Midlands Section of the Institute of Corrosion Science and Technology was held at the Steering Wheel Hotel, Hinckley, on Monday 6 March at which Mr W. Schleusser of Bayer Chemicals, Germany, presented a lecture entitled "Anticorrosive primers based on inert inorganic pigments and ferrites".

At the present time, calcium and zinc ferrite pigments are being manufactured on a pilot scale only. To date they are exhibiting extremely interesting corrosion inhibition properties, although it is not claimed that either product would be as active as zinc chromate in this respect.

The behaviour of these products was assessed in a wide variety of media, although the bulk of the investigation was undertaken on standard types of medium and long oil length alkyls. For comparative purposes, the basic substrate selected was phosphated mild steel panels, with back-up work on abrasive blasted steel once parameters had been established. Long term salt spray tests were the prime means of performance comparison, backed up by coastal and industrial exposure.

The many slides displaying the performance variations with differing pigment/binder ratios, variation being made in small steps, were themselves an education in paint technology. Each medium was pigmented in turn with rutile and anatase titanium, zinc chromate, zinc phosphate, red lead and each of the ferrites, with control tests on each of the media cast as clear films. The excellent performance achieved by inert pigments such as rutile TiO_2 at 30 per cent of critical P.V.C. on an inert, chemically clean, surface was a revelation.

On abrasive blasted surfaces the value of zinc chromate, even at levels as low as 10 per cent of C.P.V.C., was obvious, whilst zinc phosphate had little, if anything, to offer above the performance of TiO_2 alone. The ferrite pigments certainly show to advantage over all other pigments apart from zinc chromate, although their optimum is reached at *circa* 25 per cent of C.P.V.C.

The step by step approach had been extremely thorough in evaluation of these materials, each of which give an alkaline pH at the film to steel interface under humid conditions. Mr Schleusser stressed that the products are not soluble, and therefore not solution-active in the manner of the chromates, but effectively behave as basic pigments in reaction with the media under conditions of moisture.

The question time was lively, all present having been deeply interested both in the superb presentation by Mr Schleusser and the long term possibilities of the products under discussion.

Mr M. Jefferies proposed a vote of thanks for a most interesting and entertaining lecture.

J.E.F.S.

Information Received

New technical oil refinery

A new £500,000 technical vegetable oil refinery has been opened by BOCM Silcock at Selby, Yorkshire. The new refinery, which will process mainly linseed, soya and rape oils, replaces the firm's refinery at Hull which was closed earlier this year. Selby was chosen because it already has an oil seed crushing plant which produces crude oil for refining.

The refined oils are used mainly in the manufacture of paints, resins and printing inks, but there is a significant export trade, especially to the Middle East construction industry.

Ecology and toxicology centre

A new association, the European Chemical Industry Ecology and Toxicology Centre (ECETOC) has been established in Brussels to improve the knowledge of the toxicological and ecological effects of chemicals. Founded by some forty research-based chemical companies in Western Europe which have special expertise in the production, handling and uses of a large number of chemicals in different product classes, the Centre will co-ordinate their efforts. It will maintain close contact both with responsible authorities and the public to ensure that there is full understanding of the relevant information.

Synthetic fatty amines plant

Plant which will produce up to 4,000 tonnes per year of *Synprolam* synthetic fatty amines is to be built on Teeside by ICI Petrochemicals Division. It is expected to be producing by the end of 1978. Fatty amines and their derivatives are used in a wide variety of industrial and domestic applications including emulsifiers, corrosion inhibitors, powder anti-caking aids, fibre processing aids, biocides and fabric softeners.

Spray centre

R.D.M. Industrial Services Ltd have set up a Binks-Bullows spray centre in Manchester to serve the North West of England. The spray centre incorporates a laboratory where the latest techniques for spray finishing can be demonstrated and where training of customers' operatives can be carried out. The laboratory has facilities for powder coating, conventional, airless and electrostatic painting and stoving.



A view of the centrifuges and control panel at the new BOCM Silcock technical oil refinery

Change of address

U-E Chemicals (UK) Ltd have moved to Hawke House, Green Street, Sunbury-on-Thames, Middlesex TW16 6RA.

New pigment plant

Ciba-Geigy is investing £5 million in a new manufacturing plant to modernise its azo pigment production at Paisley, Scotland. In 1977 Ciba-Geigy announced a £2.5 million investment plan for the modernisation of phthalocyanine production at Paisley and the new phthalocyanine plant is due to come on stream in the second half of 1978.

The new azo plant is due to be completed in 1980 and will incorporate the latest manufacturing methods and techniques in classical azo pigment production.

Bayer christens its iron oxides

Iron oxides produced by Bayer AG, are now being marketed with a new trade name, *Bayferrox*. The registered trade mark is being introduced by Bayer worldwide, with

the British launch coordinated by the Inorganic Chemicals Division of Bayer UK Ltd. Previously the iron oxides, of which Bayer is now the world's most important producer, have been sold without an identifying name, and the use of the new trade name is hoped to assist customers in linking the products with the name of Bayer and to the advanced production technology which is used.

U K distributor

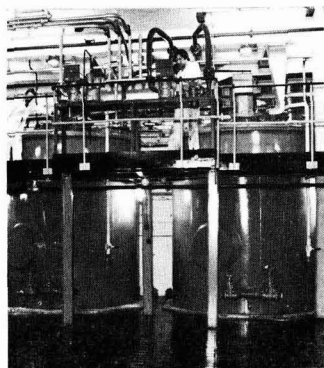
Cole Chemicals Ltd has been appointed the sole distributor in the U.K. and Eire for the Resindin division of Sybron Italia S.p.A. for their range of Relite and Ionac ion exchange resins.

Expanded European operations

The Bee Chemical Company, a leading international manufacturer of coatings and liquid colourants for plastics, has formed Bee Chemical Company S.A.R.L. and opened offices in Paris.

New mixing plant

As part of the modernisation programme for re-equipping their Silvertown plant, International Paint, Industrial Coatings have commissioned four new mixing vessels, each of 15,000 litres capacity. In addition to increasing the plant's capacity, the new mixers will enable International Paint to meet industry's growing requirement for higher quality coatings and to manufacture a greater number of bulk batches.

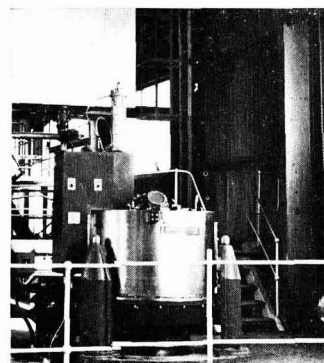


Two of the new 15,000 litre vessels which have been installed at the London works of International Paint

Increased resin catalyst capacity

Croda Synthetic Chemicals has commissioned an expansion to its facilities for the production of the Crodafonic range of sulphonic acids which are of particular importance as catalysts for resin systems. The new expansion doubles the capacity of the sulphonic acids plant and will enable Croda to meet a rising demand for these chemicals.

The Crodafonic range includes toluene, xylene, benzene, phenol and mixed aromatic sulphonic acids, all produced in a variety of concentrations and purities.



A new Broadbent Centrifuge, part of the expansion at the Sulphonic Acids Plant at Croda

Tioxide Group Ltd

Following the successful outcome of the offer by Dalgety for Federated Chemical Holdings Ltd, all the equity capital of Tioxide Group Ltd is now held by ICI and Lead Industries in equal shares.

Resin plant restarts

ICI Petrochemicals Division has restarted its petroleum resin plant at Wilton following prolonged reconstruction work. This plant produces the petroleum resin Imprez 100 and the tack resin Imprez T85. The major use for Imprez 100 is in road markings, rubber tyres, paints, wax blending and adhesives, and the speciality tack resin Imprez T85 is used in pressure-sensitive and hot-melt adhesives.

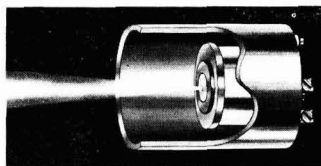
New products

Plastic films

Two types of plastic film for glazing solar collectors are available from Chemplast Inc., U.S.A. Tedlar (PVF) and Teflon (FEP) films offer the advantages of reduced cost and weight when used as the outer and inner glazing materials in either liquid or air type solar flat-plate collectors.

Low energy ions

A new low energy saddle field ion source, the Model B13, has now been introduced by Ion Tech Ltd. It produces ions with energies from less than 100eV up to 3650eV. This new product development offers unique opportunities to research workers specialising in surface interaction studies of ions with polymers, semi-conductor materials, biological specimens or any other application which requires a clean ion beam source free of contamination, giving a choice of continually variable ion energies and ion current density.



A new Low Energy Saddle Field Ion Source, model B13

New XRF materials analysis systems

Link Systems Ltd have available two X-ray fluorescence analysis systems, the MECA 10-42 and MECA 10-44 which offer the analyst full flexibility and operator convenience together with unprecedented sensitivity and stability. The MECA Systems can cope with samples ranging from bulk solids to suspensions, through to microsamples on a filter paper, with the minimum of sample preparation.

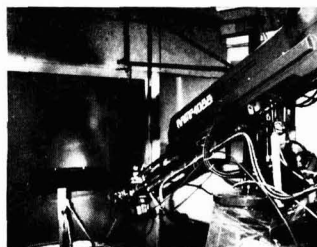
New Albany filters

A new advanced design based on their successful 3000 Series multiple filters, has been introduced by Albany Engineering Systems (Europe) Ltd. Designated the 3000 H & J range the new filters are the result of two years extensive operating trials. The new filters incorporate a simplified automatic control, modular construction with standard actuator valve configuration as well as space saving and cost reductions. The filters are self-cleaning and provide a continuous flow even while backwashing.

Robot finisher

Space Models Ltd, manufacturers of moulded polyurethane baffles for loud-

speakers, have installed a Binks-Bullows electronic robot for spray painting. The Binks-Bullows RAMP robot needs only one skilled operator to programme it by guiding the machine through the spraying sequence for each component. The movements are recorded on magnetic tape in a cassette. Playing back the tape causes the RAMP to duplicate the operator's actions and apply a perfect finish each time.



Binks-Bullows R.A.M.P. electronic robot spraying machine

Canespa-Tronic paint available

Canespa-Tronic electro-conductive paint for the dissipation of unwanted static charges is now available to industry for use in applications which cannot be served by the standard range of Canespa-Tronic electro-conductive foam systems and floor and bench coverings. Possible uses for the new paint include coating rollers on electrostatic copying machines.

New paint additives

Three new developments by the German company, Borchers, are now being marketed in the UK by their agents, Chemitrade Ltd. They are Borchigel L 75 a polyurethane based non-ionic thickening and levelling agent for acrylic emulsion paints, Borchigen HMP a liquid product which improves the adhesion in stoving enamels and Borchitherm EP 10, a liquid accelerator especially designed for use in amine or amino-amide hardening two component epoxy resins.

New red shade yellow pigment

A further extension of the already wide range of yellow pigments marketed by Burrell Colours Ltd for use in printing inks has been announced. The latest pigment to be introduced by Burrell Colours is a new transparent red shade yellow, CI Pigment Yellow 12. Called Fastona Yellow 12RT, the new quality has been developed to meet market demand for a red shade version of the company's Fastona Yellow 12XT. The pigment offers outstanding dispersibility and flow properties in addition to a high degree of transparency.

New aluminium paste

A new aluminium paste resistant to gassing in waterborne coatings is now marketed by Claremont Polychemical Corporation, New York. Excellent shelf stability has been obtained in a variety of acrylic formulations stored at room and elevated temperatures for extended periods of time. The new product, known as Claribrite Aluminium Paste XP-24050, contains 60 per cent aluminium and is easily added to aqueous formulations by simple mixing.

Sample concentrators

ChemLab Instruments, agents for Brinkmann Instruments Inc., have available a new range of sample concentrators designed for use wherever large numbers of samples need to be concentrated simultaneously, for example, eluent fractions in column chromatography, organic solvent extractions, scintillation counting samples. Concentration is achieved in approximately half the time normally required in evaporation procedures using a water bath.

Gel strength measurements

C. Stevens & Son (Weighing Machines) Ltd has available the Boucher Electronic Jelly Tester which automatically measures the gel strength and produces a digital readout of accurate, reproducible grams Bloom values. The instrument has been designed to enable non-skilled operators to obtain accurate results rapidly: a 200 gram Bloom measurement takes 30 seconds. Applications are numerous and the instruments are in use in research, testing and controlling the manufacture of foods, pharmaceuticals, paints, glues and photographic materials.



The new Boucher Electronic Jelly Tester, which measures gel strength

New micro-processor

Nelson Computer Services are marketing a micro-processor based checkweighing system, developed jointly by themselves and Aegis Components Ltd. The unit, called Checkweigh System 3 can easily be used by operators in industries where materials are dispensed or compounded by weight. The unit is capable of controlling batch weighing processes to any desired accuracy, and provides the user with a comprehensive documentary record of each weighing, together with data on stock usage and total batch weights. The system significantly reduces material waste and improves the consistency or quality of the finished product.

Modular pump drum

Bowser Pumps Ltd, a newly formed company which has bought out both Barnes Bowser Petrol Pumps Ltd and Willis Pumps Ltd, has introduced a new modular pump drum to the market. The pump drum is suitable for all petroleum non-corrosive products, normally dispensed from 25 litre or 45 gallon drums.

Urethane enamel coating

Inmont Corporation, has available a new two-component urethane enamel coating for the automotive refinishing market. It is claimed to be the fastest-drying clear urethane on the market. This new Rinshed-Mason product, 893 Clear and 894 Hardener, used in combination, provides a rich, tough shine that needs no compounding or polishing.

Functional labelling

Avery Label Systems, inventors of the first self-adhesive label and pioneers of the most important advances in labelling techniques, have published an illustrated booklet detailing the development and uses of self-adhesive labels. The booklet covers the construction of the label, the use of adhesives and face materials and the development of application systems.

New laser Raman systems

The latest Raman system from Glen Creston, the Spex Ramalog 5M, has the new revolutionary spatial filter which optimises the performance, particularly close in to the laser line. The system incorporates a high light gathering power F/1 lens system with variable working distance, a photomultiplier which has a guaranteed low dark count and a photon counter tailor made to the needs of Ramalog 5M to provide unsurpassed signal noise ratio.

Drying recorder

The Mickle Laboratory Engineering Company has developed a Ten Track BK Drying Recorder. Each track is provided with its own needle carrier which operates independently, greatly increasing the capacity of the instrument.

Conferences, courses etc.

Facts of life

Monsanto has published a booklet entitled "The chemical facts of life" which is a

candid discussion of chemicals, their history, use and misuse, testing procedures, benefits and risks to human beings. The publication marks the start of a major public information campaign in the U.S.A. to create a more balanced perspective in the mind of the public of chemicals and their use.

Safety information

Revertex has published an industrial health and safety information bulletin covering aqueous polymer dispersions (latex and emulsion), aqueous polymer solutions, and aqueous compounds derived from them.

Financial survey

Jordan Dataquest has issued its latest survey of the British paint industry which comprises the financial reports of some 120 companies with economic analyses and forecasts.

Industrial finishing conference

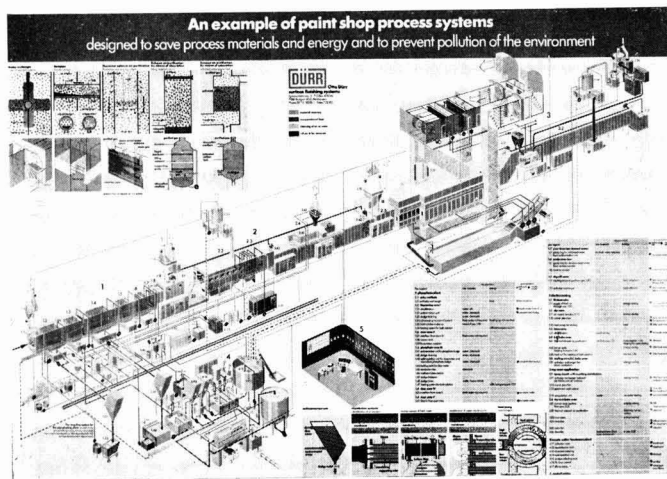
The Paint Research Association has organised its third international conference entitled "The new technologies for industrial finishing", to be held at the Excelsior Hotel, London Airport on 23-25 May.

Safety leaflet

Blundell-Permoglaz has issued a new technical information leaflet as an aid to managers, safety officers and others faced with deciding how to ensure that painted substrates have acceptable surface spread of flame properties. Entitled "Safety in fire", the leaflet is designed to assist in the selection of decorative paints to suit safety regulations.

Paint shop poster

Otto Dürr & Anlagenbau GmbH has published a poster demonstrating how raw materials and energy may be conserved in processes for industrial paint shops. It demonstrates how almost total recycling is achieved and how the various processes for materials recovery and removal of troublesome components have been so combined as to give optimum results.



The paint shop poster from Otto Dürr & Anlagenbau GmbH, which shows how raw materials and energy may be conserved

Obituary

C. J. A. Taylor MSc, ARIC, FTSC

Cyril Taylor died whilst playing golf at Walsall Golf Club on 2 December 1977. With his death the paint industry has lost one of its most respected and distinguished personalities.

Cyril Taylor was educated at Queen Mary's School, Walsall and the University of Birmingham, where he had an outstanding record which included periods as the Priestley Scholar in Chemistry 1922-3 and the Dudley Docker Research Scholar 1923-4. He obtained his BSc with first class honours in 1923, taking first place in the examination, and was awarded the degree of MSc in December 1924, and the Associateship of the Royal Institute of Chemistry shortly afterwards.

In October 1925 he joined the staff of Thornley and Knight Ltd, a company he was to be associated with for over 50 years right up to the time of his death; he was appointed Technical Director in 1949.

During the whole of his time in the industry he was active in the education of technical staff. This included lecturing successively in Wolverhampton and South Staffordshire, Smethwick, and Birmingham Technical Colleges from 1925 to 1951 and subsequently acting as an examiner for the City and Guilds of London Institute. He also served on advisory committees of the Paint Research Association and on the British Standards Institute Committees.

Cyril Taylor was one of the founder members of the Birmingham Paint, Varnish and Lacquer Club, serving as President on two occasions, 1933-4 and 1943-4, and he was made an honorary member in 1968 in recognition of his outstanding service.

He became a member of the Oil & Colour Chemists' Association in 1925, was one of those instrumental in setting up the Midlands Section in 1948 and served on Council 1954-5 and 1964-5. He was a Vice-President in 1958-9 but will be remembered chiefly for his work on the Technical Education Committee for many years, and as editor-in-chief of the Paint Technology Manuals published by the Association. He also served on the Association's Publications Committee and the Professional Grade Committee.

Despite this record of academic and practical achievement, Cyril remained an approachable and modest man, ever delighting in the blossoming careers of "his" students. No mean sportsman in his young days, being particularly keen on tennis and athletics, he turned in later years to golf as his relaxation. This, together with his family, his rose growing and his continuing keen interest in all aspects of the latest technical innovations gave him a full and useful life up to the end.

He will be remembered with gratitude by all those in the industry who came under his influence.

B.J.A.

News of Members

Mr D. A. Bayliss, Chairman of the London Section, has been appointed to the Board of BIE Anti Corrosion Ltd as Technical Director.

Mr B. Cryer, an Ordinary Member attached to the Bristol Section, has been appointed Works Technical Manager in Berger's operations division in Hengrove.

Mr W. Grainger, an Ordinary Member attached to the Bristol Section, has retired as Hengrove Works Technical Manager after twenty-two years service.

Mr G. A. Cox, an Ordinary Member attached to the West Riding Section and an Associate in the Professional Grade, has been appointed as Technical Director of Ensecote Ltd after three years as Technical Manager.

Mr D. Sharpe, an Ordinary Member attached to the London Section and an Associate in the Professional Grade, has been appointed to lead short term research and development, and Mr P. Laybourn, also an Ordinary Member attached to the London Section, has been appointed Technical Service Manager of the Sericol Group Ltd.

Mr L. Tasker, an Associate Member attached to the Manchester Section, has been appointed Chief Chemist at R. J. Stokes & Co. Ltd.

Mr H. S. Parker, an Associate Member attached to the London Section, is to retire from his position at Leon Frenkel Ltd after over 48 years' service.

We were most interested to receive from an old and distinguished Member of the Association in Pakistan, Mr Rahim Bux Khan, who is now 83 years of age, a most interesting booklet which he has written entitled "Dynamism—An Elaboration".

Dr G. D. Parfitt, an Ordinary Member attached to the Newcastle Section and a Fellow in the Professional Grade, is to deliver a Plenary lecture on behalf of the Association at the XIVth FATIPEC Congress to be held in Budapest, Hungary on 4-9 June. The title of the lecture will be "Pigment dispersion—in principle and practice".



Photograph of guests at the Scottish Section annual dinner dance, a report of which appeared on page 137 of the April issue of JOCCA. (Left to right) Back row: Mr I. R. McCallum, Mr A. McKendrick, Mr M. Prigmore, Mr R. King. Second row: Mr J. Mitchell, Mr A. McLean, Mr J. D. W. Davidson, Mr T. Wilkinson. Third row: Mrs McCallum, Mrs Wilkinson, Mrs Prigmore, Mrs McKendrick. Front row: Mrs Mitchell, Mrs McLean, Mrs Davidson, Mrs King

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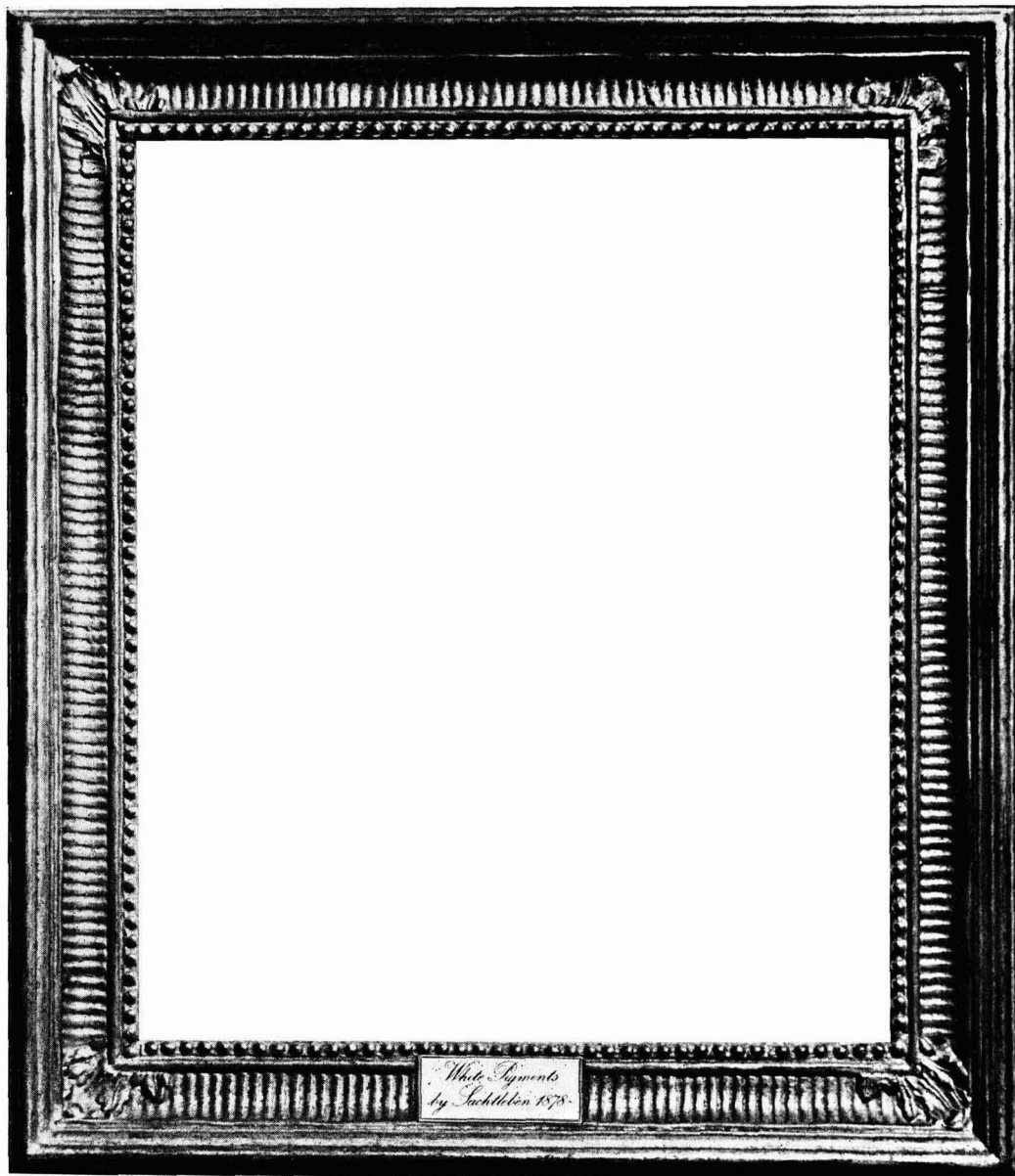
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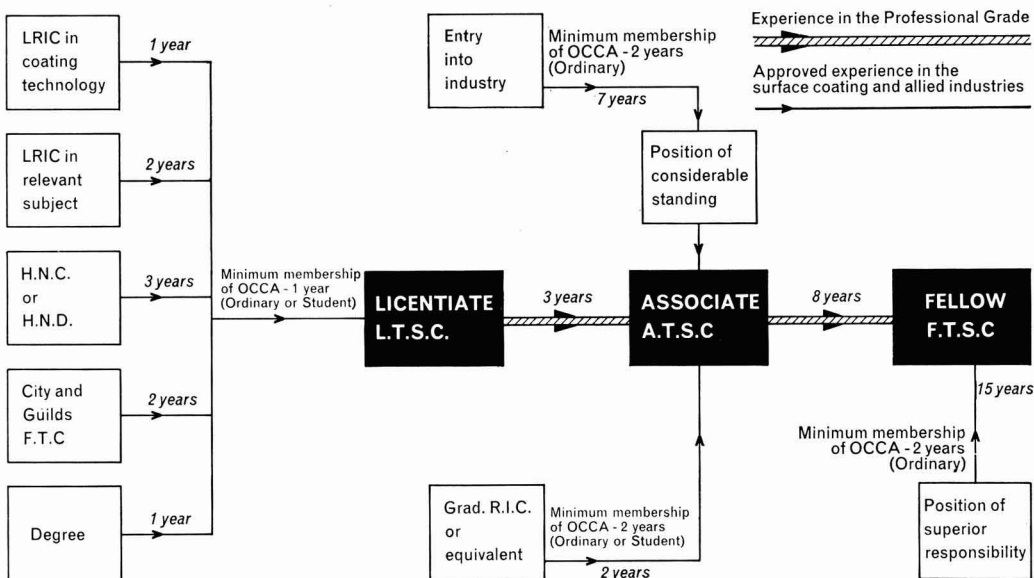
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Optional Professional Grade for Ordinary Members

The innovation of the Professional Grade has proved to be most successful, as evidenced by the impressive list of names in the December 1977 issue of the *Journal*. For the convenience of potential applicants, a chart indicating different routes to the various grades is shown below.

Routes to the Professional Grades



Note: At present there is no restriction on Students up to 21; between 21 and 25 a certificate from the employer or college confirming the course being taken is required.

Regulations for admission to the Professional Grade

Note: For the sake of simplicity, reference is made only to UK examinations etc., but equivalent qualifications overseas will naturally be accepted.

A. Licentiate

1. Shall be an Ordinary Member of the Association and have been an Ordinary Member or Student of the Association for not less than one year.

2. Shall have attained the age of 22.

3. (a) Shall be a Licentiate of the Royal Institute of Chemistry in Coatings Technology (*viz.* Higher National Certificate + Endorsement in coatings technology + 1 year approved experience in the science or technology of coatings after passing the endorsement examination).

OR (b) Shall be a Licentiate of the Royal Institute of Chemistry in another relevant subject such as advanced analytical chemistry, colour chemistry or polymer science, and shall

have two years' approved experience of coatings since so qualifying.

OR (c) Shall hold the Full Technological Certificate of the City and Guilds of London Institute in a relevant subject as approved by the Professional Grade Committee and shall have two years' approved experience in the science or technology of coatings since gaining the FTC.

OR (d) Shall have passed Higher National Certificate or Higher National Diploma with three years' approved experience in the science or technology of coatings since qualifying, but two years' approved pre-qualification experience shall be deemed equivalent to the third post-qualification year.

OR (e) Shall be graduate in relevant subject with not less than 1 year's approved experience.

OR (f) Shall have passed such other qualifications as approved by the Professional Grade Committee from time to time.

4. Shall be required to satisfy the Professional Grade Committee, or some other body approved by the Professional Grade Committee in a *viva voce* examination and submit a dissertation on a topic previously approved by the Professional Grade Committee.

5. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

6. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

B. Associate, being already a Licentiate

1. Shall, since his election to the Licentiate-ship, have practised the science or technology of coatings for not less than three years.
2. Shall provide evidence acceptable to the Professional Grade Committee of his superior professional skill and maturity.
3. Shall hold the City & Guilds of London Institute Insignia Award OR shall submit a thesis or dissertation of comparable level on a topic previously approved by the Professional Grade Committee OR shall have published work which, in the opinion of the Professional Grade Committee, is of comparable merit.
4. MAY be required to satisfy the Professional Grade Committee or some other body as approved by the Professional Grade Committee in a *viva voce* examination.
5. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.
6. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

C. Associate, not already a Licentiate**EITHER**

1. Shall be not less than 24 years of age.
2. Shall be an Ordinary Member of the Association and have been an Ordinary Member or Student of the Association for not less than two years.
3. Shall hold the Graduateship of the Royal Institute of Chemistry or Council of Physics or a University or Council of National Academic Awards degree recognised by the Royal Institute of Chemistry or Institute of Physics as giving full exemption from the Graduateship examination.
4. Shall have not less than two years' approved post-graduate experience in the science or technology of coatings.
5. Shall normally be required to satisfy the Professional Grade Committee or some other body approved by the Professional Grade Committee, at a *viva voce* examination.
6. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.
7. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

OR

8. Shall be not less than 30 years of age.
9. Shall be an Ordinary Member of the Association and have been an Ordinary Member of the Association for not less than two years.

10 Shall have been engaged in practising the science or technology of coatings for not less than seven years and shall have attained a position of considerable standing in the industry.

11 Shall normally be required to satisfy the Professional Grade Committee in *viva voce* examination of his professional competence.

12 Shall normally be sponsored by three Ordinary Members of the Association in the professional grade (either Associate or Fellow) at least one of whom must be a Fellow.

13 Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

D. Fellow

Note: This is the senior award of the professional grade and signifies that the holder has made outstanding contributions to the science or technology of coatings or has reached a position of eminence in the industry through the practice thereof. The Professional Grade Committee will require substantial evidence of professional maturity in the science or technology of coatings although commercial experience will be taken into account in assessing the merits of candidates.

1. Shall be not less than 33 years of age.
2. Shall have been an Ordinary Member of the Association for not less than two years.
3. Shall be engaged in a position of superior responsibility in the coatings industry.
4. EITHER (a) shall have been an Associate of the professional grade for at least eight years;
OR (b) shall have not less than fifteen years' experience of the science or technology of coatings in a position of superior responsibility.
5. Shall submit, with his application, an account of his experience, with due reference to scientific and technological interests, achievements and publications.
6. Shall normally be sponsored by three Ordinary Members of the Association in the professional grade, all of whom must be Fellows.
7. Shall have paid the fee stipulated by the Council and have paid the current subscription payable by an Ordinary Member.

The fees payable with applications are as follows:

Fellow—£10.00	Associate—£6.00
Licentiate—£3.00	
(Plus VAT at standard rate)	

Application

Completed application forms should be returned, together with the appropriate remittance, to the Director & Secretary at the Association's offices (except in the case of those Members attached to the Auckland,

South African and Wellington Sections who should address their forms to their Section Hon. Secretaries).

The Committee wishes it to be known that Members rejoining the Association after a period in other industries may include length of service as an Ordinary Member before their resignation as part of the qualifying periods for entry into the Grade.

Students wishing to apply for entry into the Professional Grade must first make application in writing for upgrading to Ordinary Membership, giving the reasons for their eligibility for such regrading. Applications, together with the appropriate remittance, should be addressed as for application for admission to the Professional Grade.

Potential applicants are recommended to give the fullest possible details of their appointments, including the number and type of staff under their control, and indicating to whom the applicant is responsible, as this aids the committee considerably in its deliberations.

It is felt that applicants for admission to the Licentiate grade might wish to have further information on the pattern which it is suggested should be followed for dissertations, the subjects of which have first to be approved by the Professional Grade Committee.

The dissertation should be preceded by a short summary and commence with a brief introduction and some account of the current state of knowledge. Where practicable it should follow the general format of a paper in *JOCCA*.

The dissertation may be a review of a subject, a theoretical treatment, descriptive of practical work or a combination of these. It must indicate that the candidate has a reasonably wide and up-to-date knowledge of his chosen subject and understands the basic scientific principles underlying it, and that he is able to think critically and constructively.

Where practical work is described, some attempts should be made to draw theoretical conclusions or to form some provisional hypothesis, together with an outline of what further work would be required to confirm the views put forward or further to advance the knowledge of the subject.

Where the dissertation is a review or a theoretical treatment, there should be an attempt to contrast and compare any opposing views expressed in earlier works, to examine the views critically, to suggest any compromise interpretation, to account for all the known facts and to outline any further work by which the opposing views could be tested.

Where applicable, references should be given to published work, graphs, diagrams etc. to be appended.

Length: Text should be approximately 5 000 words.

Applicants should refer to the paper by Moss which appeared in the January 1973 issue; the Professional Grade Committee feels that candidates for the Licentiate grade could with advantage use this paper as a model for their dissertations.



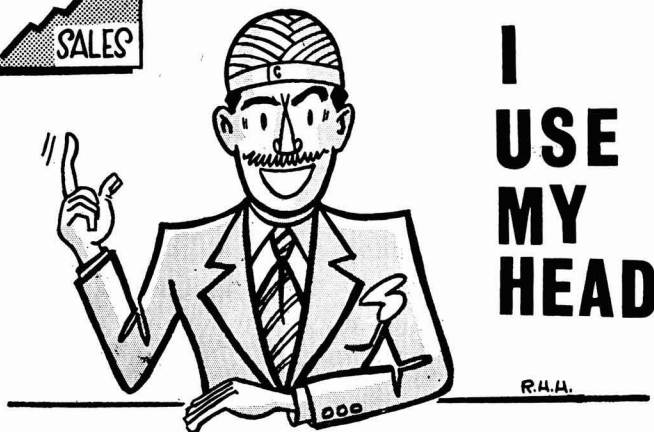
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Jordan Award

This award was instituted by the late Mrs M. R. Jordan in memory of her husband Dr L. A. Jordan, who was President of the Association 1947-49 and an Honorary Member, and who died in December 1964. The Committee invites applications for the fifth award of £100.

The rules of the Award are:

1. The Award will be made for the best contribution to the science or technology of surface coating by a Member of any nationality working in either the academic or industrial field who is under the age

of 35 at the date of application.

2. The final date for submission of applications will on this occasion be 31 December 1978 and it is hoped to present the award at the Stratford-upon-Avon conference in the following June.

3. The selection of the recipient of the Award will be made by a Committee under the chairmanship of the Association's Hon. Research and Development Officer.

4. There will be two methods of application. First, by the submission of a paper describing original work by the candidate which is offered for publication in the

Journal or has been so published during application. The alternative method will be by recommendation by a superior for work which for reasons of commercial secrecy cannot be published; in this case the candidate will be expected to submit a dissertation on a topic relating to his work and demonstrating his superior knowledge of the principles thereof. The Award is for individual merit and clear evidence of the candidate's own contribution will be required if a paper is offered under joint authorship.

5. Applications should be addressed to the Director & Secretary at the Association's offices.

Forthcoming Events

Details are given of Association meetings in the United Kingdom and Ireland up to the end of the month following publication and in other parts of the world up to the end of the second following publication.

May

Thursday 11 May

London Section: Afternoon visit to the Paint Research Association, Waldegrave Road, Teddington, Middlesex TW11 8LD commencing at 2.30 p.m.

June

June

Irish Section: Golf outing—details and date to be announced.

Friday 2 June

Scottish Section—Eastern Branch: Car rally and barbeque—details to be announced

Wednesday 21 June

Association Annual General Meeting

The Annual General Meeting for 1978 will be held on Wednesday 21 June at the Piccadilly Hotel, London W1, commencing at 12.30 p.m. with a reception in the Edward Room. This will be followed at 1.00 p.m. by luncheon.

At 2.15 p.m., in the Margaret Room, Dr

D. Davies, Chief Scientist of the Department of Industry, will deliver a lecture entitled "Resource problems in the downstream chemical industries".

At 3.00 p.m., or as soon thereafter as the lecture shall have terminated, the Annual General Meeting will be held in the Margaret Room. Members not wishing to attend the luncheon and lecture will, of course, be able to participate in the Annual General meeting.

Tickets for the luncheon/lecture are priced at £6.48 each, inclusive of wine and VAT, and are available from the Association's offices at the address on the Contents page.

Register of Members

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

Ordinary Members

AHERN, PETER ANTHONY, BSc, 36 Brook Courts, Monkstown, Co. Dublin. (*Irish*)

CHAN, DOMINGO, 192 Timberbank Blvd, Agincourt, Ontario M1W 2A3, Canada. (*Ontario*)

CLARKE, JAMES ANTHONY, BSc, PhD, 35 Beverley Avenue, Leigh, Lancs. (*Manchester*)

COTTON, LES, Punch & Co, Bluebell Industrial Estate, Dublin 12. (*Irish*)

DOBELL, ROBERT KEITH, 52 Lucerne Way, Harold Hill, Romford, Essex. (*London*)

FALDER, JOHN STUART, BSc, H Marcel Guest Ltd, Collyhurst Road, Manchester M10 7RU. (*Manchester*)

GRIMES, ANTONY THOMAS, Ciba-Geigy (PAC) Ltd, Simonsway, Manchester 22. (*Manchester*)

HUTCHINSON, VINCENT GERARD, BSc, 149 Corbawn Wood, Shawhill, Co Dublin. (*Irish*)

KELLY, JEFFREY, 47 Patch Croft Road, Peel Hall, Wythenshawe, Manchester M22 5JR. (*Manchester*)

LAUDER, HUNTLY ST. JOHN, PhD, Dunfirth, Butterfield Avenue, Dublin 14. (*Irish*)

LONG, PETER, 38 Eksteen Avenue, Bergvliet 7800, South Africa. (*Cape*)

MCMAUS, LAURENCE DONALD, PhD, MA, Anchor Chemical Co Ltd, Clayton Lane, Manchester M11 4SR. (*Manchester*)

MANNING, CECIL RICHARD, Site 388, Clonburris, Clondalkin, Co Dublin. (*Irish*)

MOREBY, FREDA HELEN, 105 Culford Road, Toronto, Ontario M6M 4K4, Canada. (*Ontario*)

RIGBY, FREDERICK JOHN, BSc, Nairn Floors, PO Box 1, Kirkcaldy KY1 2SB. (*Scottish—Eastern Branch*)

RILEIGH, ALBERT KENNETH, MSc, 21 Harley Crescent, Eastwood, NSW 2122, Australia. (*General Overseas*)

ROBINSON, PETER WILLIAM ARTHUR, 85 Ashley Road, Walton-on-Thames, Surrey KT12 1HH. (*London*)

UPTON, MICHAEL, BSc, GRIC, 99 Woolwich Road, Bexleyheath, Kent DA7 4LP. (*London*)

VORSTER, JAMES DENIS MONTGOMERY, BSc, 115 Crabtree Lane, Hemel Hempstead, Herts, HP3 9EL. (*London*)

WASTELL, JOHN, 12 Elm Street, Little Harwood, Blackburn, Lancs. (*Manchester*)

WEIR, LEONARD, 7 Twinburn Hill, Monkstown, Newtownabbey, Co Antrim, N Ireland. (*Irish*)

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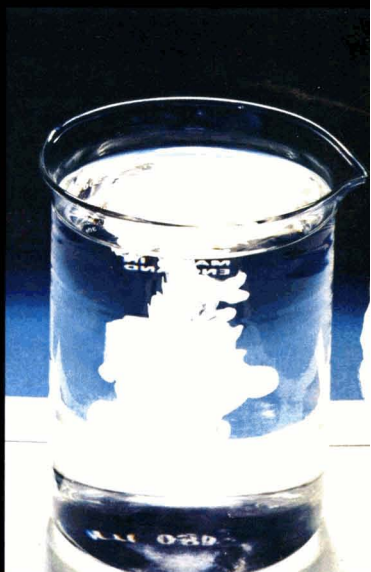
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