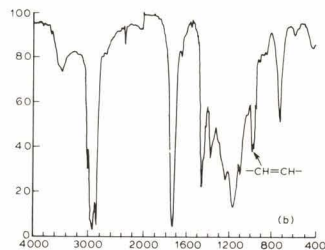




**J
O
C
C
A**

Instrumental Analysis in



"GO BACK TO CONVENTIONAL SPRAYING? - NOT ON YOUR LIFE"

(Say Management and operatives of leading furniture manufacturer in North of England).

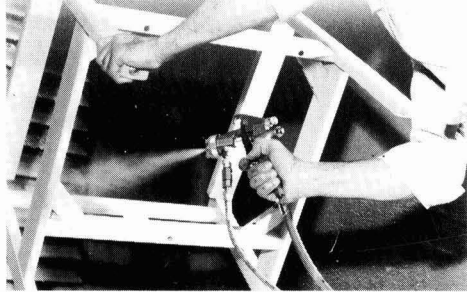
Quite simply, the Graco AA2000 Air-assisted airless finishing system represents the state-of-the-art in faultless furniture finishing techniques.

Unlike conventional airless spray, the Graco AA2000 uses air to shape and direct the fan pattern. It produces a precise, highly controllable spray fan which gives one-pass coverage with minimal material wastage and overspray. Designed for today's materials — lacquers, varnishes, stains, heavy substrates.

The Graco AA2000 is the most controllable system on the market!

*Can you afford to miss out on the savings — both in time and material?
Try the AA2000 for yourself!*

CALL GRACO — NOW!



GRACO U.K. LIMITED
Wednesfield Road,
Wolverhampton WV10 0DR
Telephone (0902) 51924
Telex 339237

Reader Enquiry Service No. 375

Atlas: Controlled Accelerated Weathering

All aspects of weathering, sunshine, rainfall, temperature, humidity and condensation can be tested in Atlas Weather-Ometers.[®]

Outdoor Weathering in

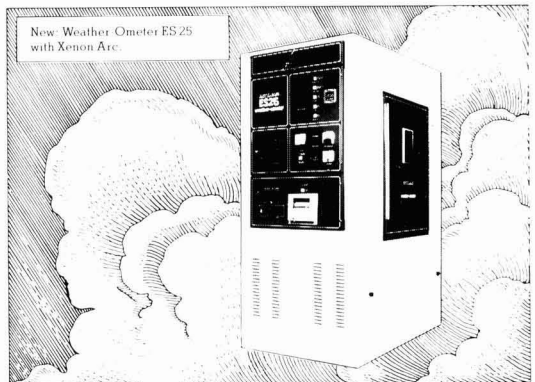
- Florida USA
- Chicago USA
- Arizona USA
- Lochem Netherlands

Accelerated Weathering And Light Fastness

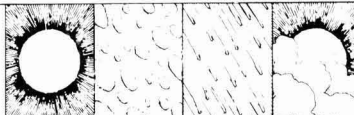
- By Weather-Ometer Fade-Ometer
- with Carbon Arc or Xenon Arc
- with Fluorescent Sunlamps

Sample Testing by Atlas SFTS

- Accelerated Weathering and Outdoor Weathering is possible at Atlas SFTS in Lochem.



ATLAS SFTS BV
P.O. Box 182
7240 AD LOCHEM
The Netherlands
Telephone (0)5730-6465 Telex 49036



**ATLAS
SFTS BV**
A Subsidiary of Atlas Electric Devices Co

Reader Enquiry Service No. 314



Formulating paint to get just the surface characteristics you want is no easy task. And once you have your formula, variations in the ingredients can't be tolerated.

As the world's largest supplier of diatomite functional fillers to the coating industry, Manville produces a number of Celite grades to give you the exact control you need.

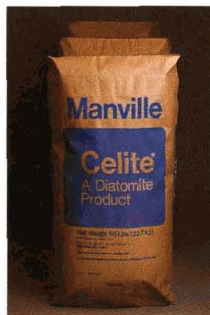
But we don't stop there. With over 50 years' experience in the industry, we know how important consistency is to you. So whether it's for emulsion or solvent coating, the Celite grade you call for is the Celite grade you get. Always consistent.

When you need high quality extenders and flattening agents for uniform gloss and sheen control in your coating formulation, specify Celite. It's consistent bag after bag.

Contact Manville (GB) Ltd.
Ryde House, 391 Richmond Road
Twickenham, Middlesex, TW1 2EQ
Tel: 01 891-0813. Telex: 928635

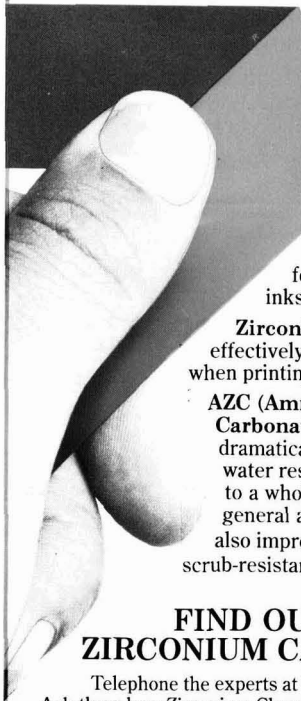
**Control
gloss and sheen
with Celite® fillers.**

**Consistent—
bag after bag after bag.**



Manville

WHAT TO DO TO IMPROVE PRINTING INKS, ADHESIVES AND PAINT



TELEPHONE
MAGNESIUM
ELEKTRON
AND ASK
ABOUT
ZIRCONIUM

Zirconium Chemicals are finding increasing benefits in the formulation of printing inks, adhesives and paint.

Zirconium Propionate effectively promotes adhesion when printing on polyolefin film.

AZC (Ammonium Zirconium Carbonate) in water based inks, dramatically improves heat and water resistance, and is leading to a whole new generation of general adhesives. Zirconium also improves the adhesion and scrub-resistance in paints.

FIND OUT HOW ZIRCONIUM CAN HELP YOU

Telephone the experts at Magnesium Elektron. Ask them how Zirconium Chemicals can help with your particular problem.



Magnesium Elektron

Magnesium Elektron Ltd
Regal House, London Road,
Twickenham TW1 3QA
ENGLAND
Telephone 01-892 4488
Telex 8811765

Magnesium Elektron Inc.
RD2 Box 251 Flemington,
New Jersey 08822 U.S.A.
Telephone 201. 782 5800
Telex 230 833310



CHLORINATED RUBBER TYPE

CHLORINATED POLYETHYLENE
CHLORINATED POLYPROPYLENE
CHLORINATED RUBBERS
etc.

APPLICATION

- Anticorrosive coatings
- Road marking paints
- Gravure printing inks
- Adhesives

**ADHESIVE GRADES
(Low Chlorine Type)**
CHLORINATED POLYPROPYLENE
CHLORINATED ETHYLENE VINYL
ACETATE COPOLYMERS
etc.

APPLICATION

- Gravure printing inks, adhesives for untreated Polypropylenes
- Primer for plastics



SANYO-KOKUSAKU PULP CO., LTD.
CHEMICALS TRADING DEPT.
1-4-5 Marunouchi, Chiyoda-ku, Tokyo, Japan
Telex: J 24279 SK PULP
Cable: SANKOKUPA TOKYO

Reader Enquiry Service No. 283

JOURNAL of the OIL AND COLOUR CHEMISTS' ASSOCIATION

Hon. Editor: D. S. Newton, AMCT
CGIA, FICorrST, FIMF, FTSC

Publications Committee:

D. B. Bannington, ATSC
P. A. Bentley, BSc
L. J. Brooke, ATSC
H. J. Clarke, FTSC
J. Coy, BA
R. E. Cromarty, BSc, ATSC
C. N. Finlay, ATSC
S. R. Finn, BSc, CChem, FRSC, FTSC
B. F. Gilliam, ATSC
J. A. Gant, LRSC
R. G. Handley
A. C. Jolly, BSc, FTSC
A. Macdonald, ANZIC, ATSC
P. Marr, PhD
J. S. Mason, BA
I. Maugham
B. E. Myatt
A. J. Newbould, BSc, CChem, MRSC
R. H. Philbrick
F. B. Redman, FTSC
R. C. Somerville
J. R. Taylor, BSc, CChem, FRSC, FICorrST,
FTSC
J. F. M. Van Campfort, NZCS
G. Warman, FPISA

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

General correspondence should be addressed to:

R. H. Hamblin, MA, FCIS,
Director & Secretary,
Oil and Colour Chemists' Association
at the address below.

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

Assistant Editor: J. C. Setton, BSc

Annual subscription to non-members:
£60.00 (\$120), post free by surface mail,
payable in advance.

Single copies £5.25 (\$12), post free by
surface mail, payable in advance.

Copyright 1985



Member of the
Audit Bureau of
Circulations

Contents

Vol. 68 No. 5

May 1985

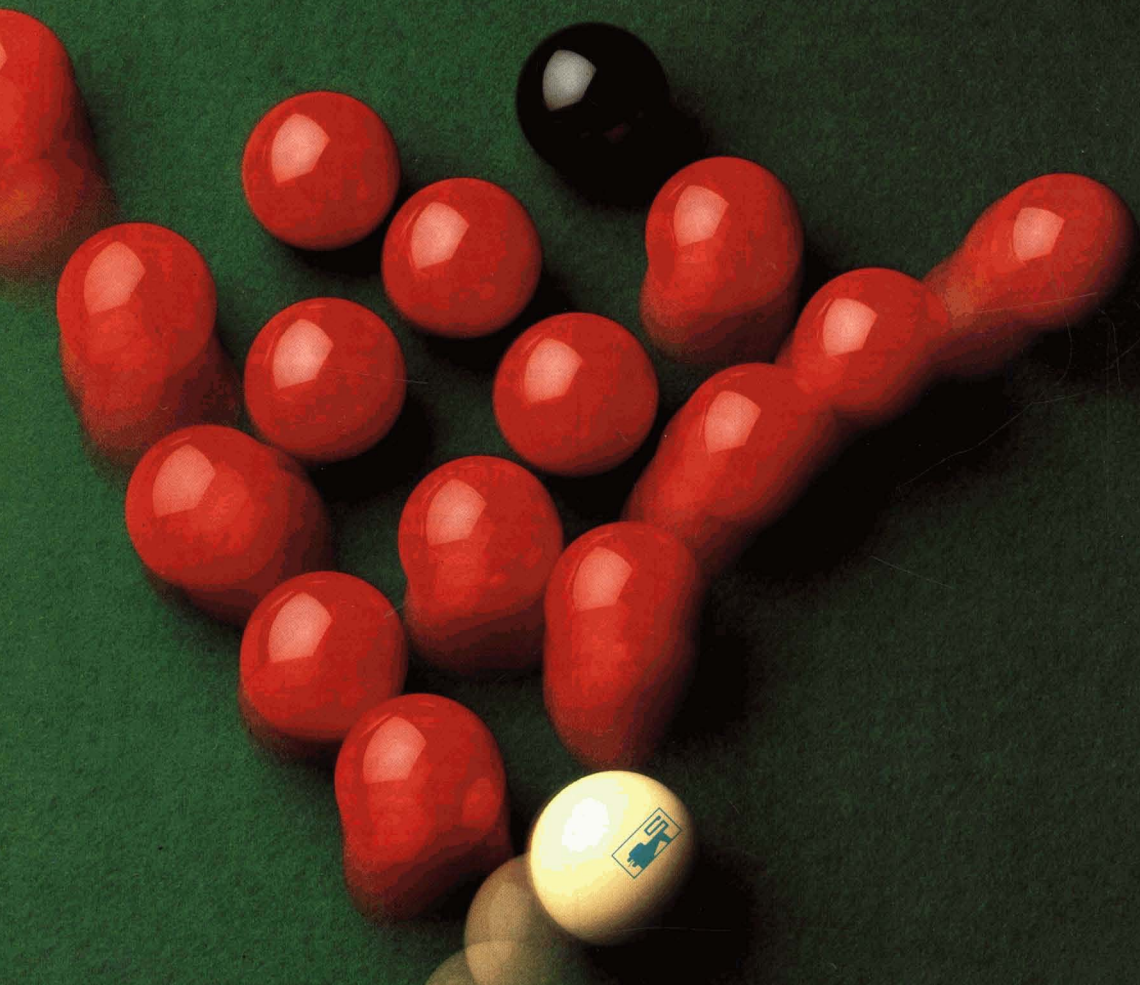
Transactions and Communications

| | |
|--|-----|
| Instrumental analysis in the coatings industry | 109 |
| <i>by I. L. Davies</i> | |
| The photochemistry of the peroxodisulphate ion in aqueous alcohol solutions | 116 |
| <i>by C. G. Roffey</i> | |
| Modification of shellac with some unsaturated acids | 120 |
| <i>by N. Prasad, S. C. Agarwal, P. C. Gupta and A. K. Dasgupta</i> | |
| Next month's issue | 122 |
| OCCA meetings | 123 |
| News | 126 |
| OCCA Conference 1985 | 115 |
| OCCA news | 129 |

OIL AND COLOUR CHEMISTS' ASSOCIATION
Priority House, 967 Harrow Road, Wembley, Middlesex, HA0 2SF England

The Honorary Editor, the Council and the Oil and Colour Chemists' Association do not accept responsibility for opinions expressed by correspondents or contributors to this Journal. The contents of the Journal are copyright and permission to reproduce Transactions and Communications, in full or in part, must first be obtained from the Honorary Editor

Blanc Fixe Micro.[®] Brilliant. And not just in its speed of dispersion.



The remarkable Blanc Fixe Micro, by Sachtleben, performs brilliantly to reduce costs and enhance the finished product.

This new micronised extender is unique in its speed and ease of dispersion. Excellent gloss values can be obtained at a low

binder requirement and at an average particle size of only $0.7\mu\text{m}$. It augments titanium dioxide, and other pigments, without the slightest reduction in opacity, gloss or covering power. And its weathering ability has been tested to the limit under widely-differing conditions worldwide.

Use Blanc Fixe Micro to save time and energy in the manufacture of primers, stoving enamels, gloss paints, powder coatings, artists' colours and printing inks. Just contact us for today's best cover story.



»SACHTLEBEN«

UK Sales Offices: 56 High Street, Edenbridge, Kent TN8 5AJ.
Tel: (0732) 863694 Telex: 95297

38A Bury Old Road, Whitefield, Manchester M25 5TL. Tel: (061) 773 2000

Sachtleben products include Hombitan[®] TiO₂, Sachtolith[®], Lithopone, Blanc Fixe, Albaryt[®] and EWO[®] grades of Barytes.

Instrumental analysis in the coatings industry

I. L. Davies

Group Analytical Laboratory, Plascon-Evans Paints (Natal) Ltd, PO Box 1386, Durban, 4000, South Africa

Summary

This paper describes the two instrumental techniques, namely infrared spectroscopy (IR) and gas chromatography (GC), commonly used to determine the composition of surface coating materials.

The basic theory underlying each technique is explained briefly and several examples are given to show their applications in the coatings industry.

The first section deals with the interpretation of infrared spectra and the use of surface techniques in infrared spectroscopy.

The second section deals with the identification of paint solvents and the use of pyrolysis and headspace techniques in gas chromatography.

Introduction

"Analysis" may be defined as the resolution of a substance into its simple components.

Using this definition as a guideline, the analytical chemist in the coatings industry can be described as a person who makes use of various techniques to determine the composition of surface coatings.

Many years ago, the analysis of a paint, consisting only of an oil, pigments and thinners, was a relatively easy task. Although paint formulations today are more complex, the analytical chemist now has the help of sophisticated instruments such as computerised infrared spectrophotometers and gas chromatographs.

In a discussion of the analysis of paint, it is first necessary to define the term "paint". (Figure 1).

Paint consists of pigment and varnish. The pigment may be divided into two categories, namely:

1. Prime pigments which provide colour, hiding power and protection of the substrate by corrosion inhibition.
2. Extenders which are cheaper than prime pigments and are used as fillers.

The varnish consists of thinning liquid, binder and various additives:

1. The liquid portion is often a mixture of solvents and diluents;
2. The binder is the film former and is usually a polymer, or reactive components that form a polymer on curing;
3. Additives may be thickeners, matting agents, catalysts, wetting agents or plasticizers.

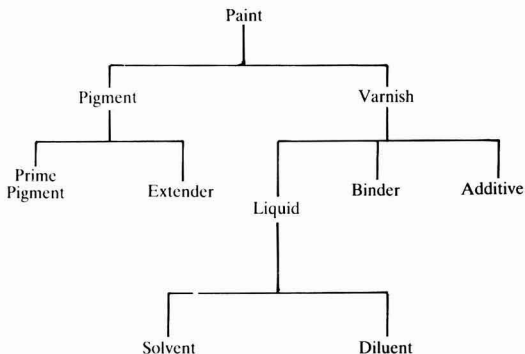


Figure 1. What is paint?

Surface coatings may require analysis for the following reasons:

1. Research and development:
Laboratory research and development is often aided by easy access to an analytical service within the same company.
2. Competitor product evaluation:
It is always informative to evaluate new products brought onto the market by competitors.
3. Customer complaints:
Products rejected by the customer may require analysis to locate the source of the problem.
4. Factory production troubleshooting:
Analysis of a faulty batch provides the information needed in order to take corrective action.
5. Quality control monitoring:
Routine quality control checks on raw materials and final products are an essential part of a paint laboratory.

There are many instrumental techniques that can be used to analyse surface coatings:

1. Infrared spectroscopy (IR)
2. Gas chromatography (GC)
3. Gel permeation chromatography (GPC)
4. Atomic absorption spectroscopy (AA)

5. Nuclear magnetic resonance spectroscopy (NMR)
6. X-ray spectroscopy (XRS)
7. Thermal analysis (TA)
8. Microscopy.

The two techniques most commonly used in an industrial paint laboratory are infrared spectroscopy (IR) and gas chromatography (GC).

IR is a universal technique used to analyse many organic and inorganic compounds. In the coatings industry, its most useful application is the qualitative and quantitative identification of binders. However, it is also used to analyse solvents, waxes, surfactants and pigments.

GC is a separation technique used to determine the composition of volatile components. Its most useful application is in the quantitative analysis of paint solvents.

The basic theory and applications of each technique will be covered in the following sections.

Infrared spectroscopy

Spectroscopy may be defined as the study of the interaction of electromagnetic radiation with matter. Infrared spectroscopy is the study of transitions within the energy levels of a molecule due to the absorption of radiation in the infrared region of the electromagnetic spectrum.

Two types of infrared spectrophotometers are currently available. Dispersive instruments pass infrared radiation of varying frequencies through a sample and record any absorptions which occur. Fourier-Transform instruments (FT-IR) pass the entire infrared spectrum simultaneously through a sample by means of a Michelson Interferometer.

Qualitative Analysis

The infrared spectral data obtained from a spectrophotometer is in the form of an infrared spectrum. This is a plot of frequency (cm^{-1}) versus percentage transmittance (%T).

The frequencies at which a molecule absorbs infrared radiation are a characteristic property of its structure. For example, a compound may contain organic groups such as $-\text{OH}$, $=\text{NH}$, $-\text{CH}_3$, $=\text{CO}$, $-\text{CN}$ or $-\text{COOH}$. Each of these structural units has its own characteristic absorption frequency in the infrared region. Identification of the compound is possible by observing the presence (or absence) of absorption frequencies associated with such groups. This is known as possible structural unit (PSU) analysis of infrared spectra.

The interpretation of infrared spectra is aided by structure frequency correlation tables and reference spectra libraries^{1,2,3}. An experienced analytical chemist can often identify a compound by merely glancing at its infrared spectrum. This is referred to as the finger-print analysis of infrared spectra.

The following two examples show the use of PSU analysis in the identification of unknown samples.

Example 1: Poly (vinyl acetate)

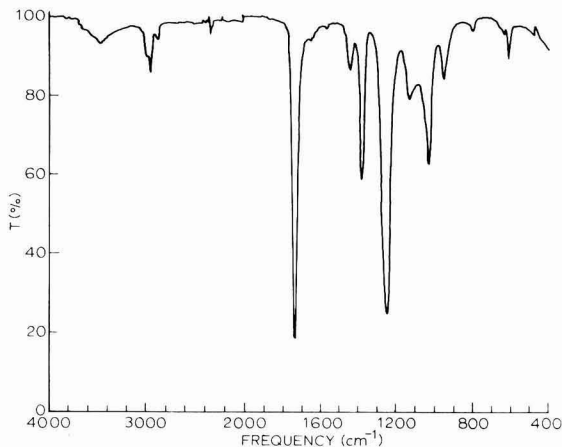
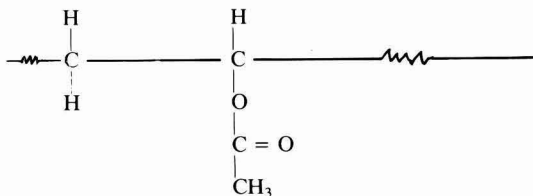


Figure 2. Poly (vinyl acetate)

Figure 2 shows an IR spectrum of a poly (vinyl acetate) (PVA) homopolymer. The absorption frequencies below indicate the following structural units:

| Frequency (cm^{-1}) | Indication |
|--------------------------------|---|
| 3000 | C-H short chain aliphatic |
| 1730 | C=O ester carbonyl |
| 1430 | CH_2 aliphatic methylene |
| 1370 | CH_3 aliphatic methyl |
| 1240 | C-O ester |
| 1020 | C-O ether |
| 600 | $\text{CH}_3-\text{C}-\text{O}$ acetate ester <div style="margin-left: 100px;"> $\begin{array}{c} \\ \text{O} \end{array}$ </div> |

This information can be used to construct the PVA structural unit:



Obviously, qualitative analysis becomes more difficult as the number of components in the sample increases. PVA is often plasticized to increase flexibility by copolymerization or by the addition of external plasticizers. These modifications to the PVA chemical structure are reflected in its infrared spectra (Figures 3-6).

The ethylene modification of PVA may be identified by the increased absorption at the 3000 cm^{-1} and 1040 cm^{-1} regions (Figure 3).

The vinyl versatate modification is seen by the difference in the absorption band at 3000 cm^{-1} and by the occurrence of a shoulder at 1460 cm^{-1} (Figure 4).

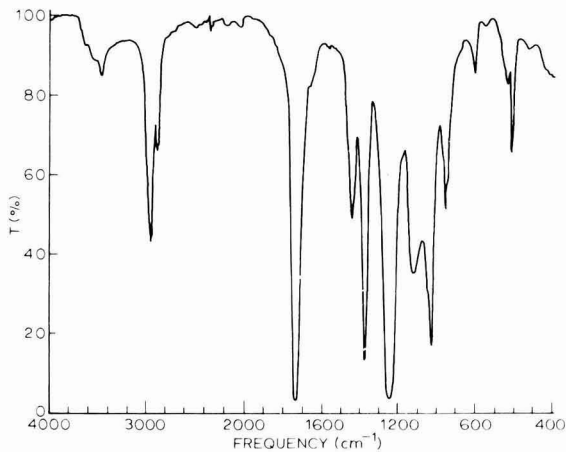


Figure 3. Vinyl acetate—ethylene copolymer

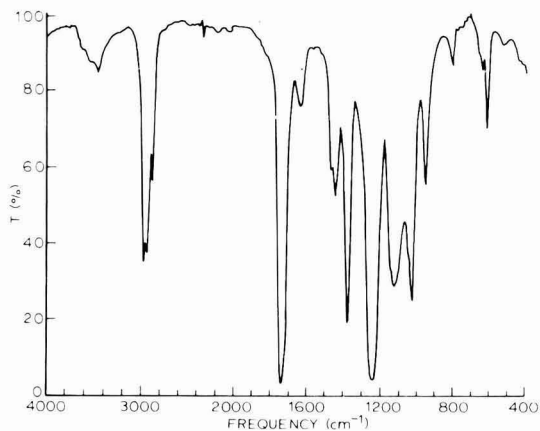


Figure 4. Vinyl acetate—vinyl versatate copolymer

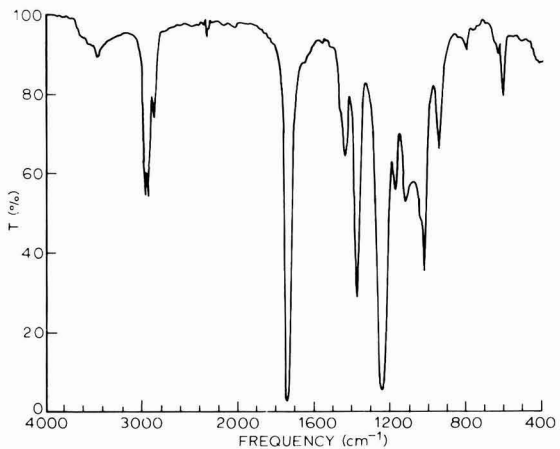


Figure 5. Vinyl acetate—acrylate copolymer

Vinyl acetate-acrylate and vinyl acetate-dibutyl maleate copolymers give identical infrared spectra. Both modifica-

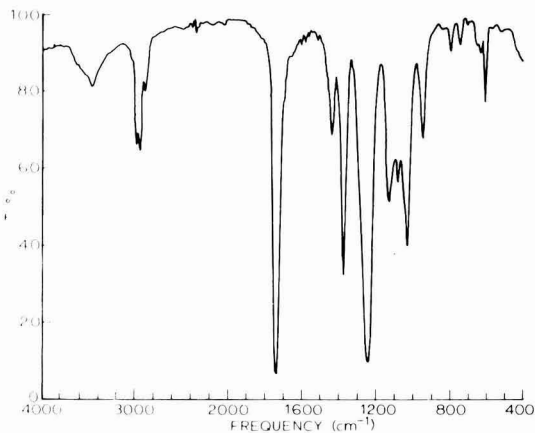


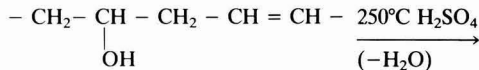
Figure 6. Phthalate plasticized pva

tions have an additional peak at 1170 cm^{-1} , a shoulder at 1460 cm^{-1} and a change in the 3000 cm^{-1} region (Figure 5).

The presence of phthalate plasticizers in PVA emulsions can be recognized by the appearance of a third peak in the centre of the "fish-tail" at 1080 cm^{-1} (Figure 6).

Example 2: Castor oil

The dehydration of castor oil is a good example of the influence of chemical structure on the infrared spectrum. On heating with sulphuric acid, castor oil is dehydrated by the loss of an hydroxyl ($-\text{OH}$) group and the gain of a carbon-carbon double bond ($-\text{CH}=\text{CH}-$).



Castor Oil

$-\text{CH}=\text{CH}-\text{CH}_2-\text{CH}=\text{CH}-$
70% dehydrated castor oil (non conjugated)

+

$-\text{CH}_2-\text{CH}=\text{CH}-\text{CH}=\text{CH}-$
30% dehydrated castor oil (conjugated)

The $-\text{OH}$ infrared absorption usually occurs at 3400 cm^{-1} and the $-\text{CH}=\text{CH}-$ at 975 cm^{-1} . Figure 7 shows that the castor oil spectrum has a more intense absorption at 3400 cm^{-1} and a less intense absorption at 975 cm^{-1} .

Quantitative Analysis

Another important feature of infrared spectroscopy is that it is a quantitative technique. The Beer-Lambert Law states that:

$$A = -\log \frac{I}{I_0} = \log \frac{I_0}{I} = abc$$

where A = absorbance observed from the spectrum
 I = intensity of transmitted radiation
 I_0 = intensity of incident radiation
 a = absorptivity
 b = cell path length (cm)
 c = concentration (g/l)
 T = transmittance observed from the spectrum

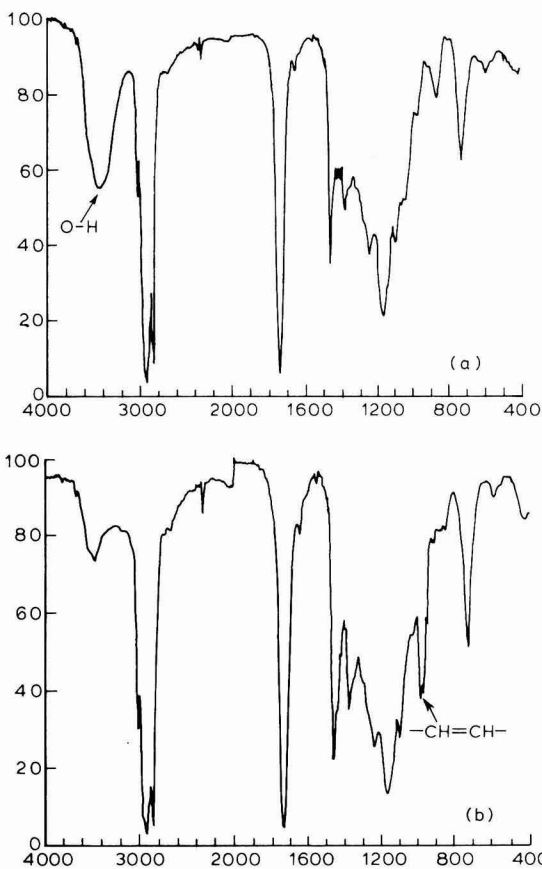


Figure 7. (a) Castor oil; (b) Dehydrated castor oil

The absorbance of infrared radiation at a specific wavelength is directly proportional to the concentration of the component which absorbs at that wavelength.

Standard samples of known concentration are usually used to obtain a working curve of absorbance versus concentration. The concentration of an unknown sample is then read from this curve.

IR applications

Several methods can be used to obtain an infrared spectrum of a sample. These may be divided into solid phase techniques, in which the infrared radiation passes through the sample, and surface techniques, in which the infrared radiation reflects off the surface of the sample.

Solid phase techniques

A binder is solvent extracted from its matrix and cast onto a clear potassium bromide disc. The disc is then placed in the path of the infrared light from the spectrophotometer.

Pigments are either ground up with a little potassium bromide powder and pressed into a disc or mixed with Nujol and squeezed between two discs.

Solvents are injected into a liquid cell, which consists of

two potassium bromide windows separated by a thin teflon spacer and secured with two demountable metal plates.

Surface techniques

The ability to study the surface composition of a material is obviously important in the coatings field. The three techniques available are multiple internal reflectance (MIR), specular reflectance (SR) and diffuse reflectance (DR). All three have the advantage of being rapid, non-destructive and require little sample preparation.

1. Multiple internal reflection (MIR)

The MIR technique is based on the theory of attenuated total reflection (Figure 8).

If the angle of incidence of infrared radiation entering a prism is at an angle greater than the critical angle, then total internal reflection will occur. Placing the sample to be analysed against the surface of the prism causes the infrared radiation to penetrate the sample to a depth of a few microns and absorption by the sample occurs. However, as the energy absorbed from a single reflection is normally weak, an optical crystal plate may be used to give multiple internal reflections (MIR). The MIR accessory containing the crystal is mounted in the spectrophotometer sample compartment.

MIR is particularly useful in the analysis of paint flakes, adhesives, rubbers, plastics, fabrics, foams and insoluble materials. One disadvantage of this technique is that the whole sample is scanned, and absorption due to pigments may mask the polymer spectrum.

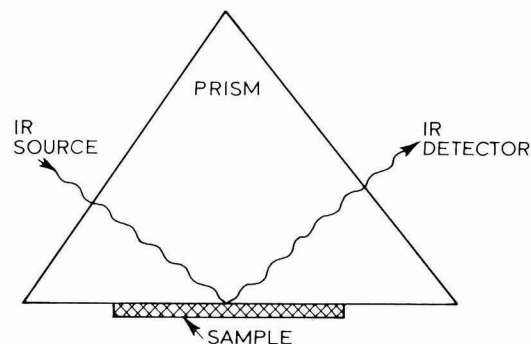


Figure 8. Attenuated total reflection (ATR)

2. Specular reflectance (SR)

Specular reflectance is used to obtain spectra of clear coatings on highly reflective substrates.

The specular reflectance accessory is mounted in the sample compartment and consists of two plane mirrors angled so that the infrared radiation penetrates the sample layer, but is reflected off the substrate. (Figure 9).

This non-destructive technique is used mainly for the analysis of can linings.

Computers

Modern infrared spectrophotometers can be linked to

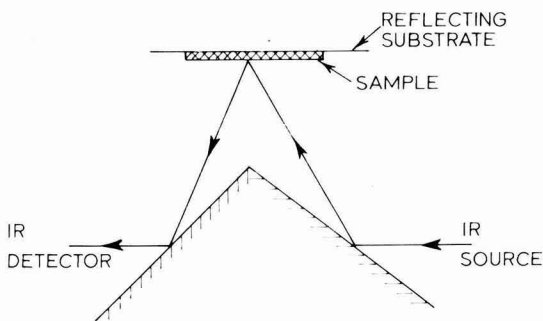


Figure 9. Specular reflectance

computers and software is available which allows the chemist to perform any of the following tasks: Store spectra on floppy discs; interpret spectra by PSU analysis; search libraries of standard spectra to find the closest match; build up a library of standard spectra; perform quantitative calculations; manipulate the spectra to simulate larger sample sizes; add or subtract two spectra; and accumulate the data from successive scans.

Figure 10 is an example of computer enhancement of a weak spectrum. This feature is particularly useful when only a small amount of sample is available.

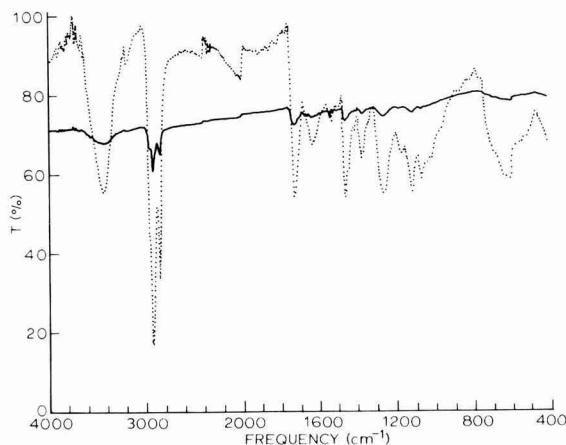


Figure 10. Spectrum enhancement

Gas chromatography

Chromatography is a separation technique used to determine the composition of volatile mixtures.

This separation takes place within a column which is usually made from stainless steel, glass or fused silica. A stationary liquid phase is either coated directly onto the internal walls of the column or coated onto a solid inert support in the column.

The column is situated in the heated oven of a gas chromatograph (Figure 11). The sample is injected with a liquid syringe into the heated injection port and carried

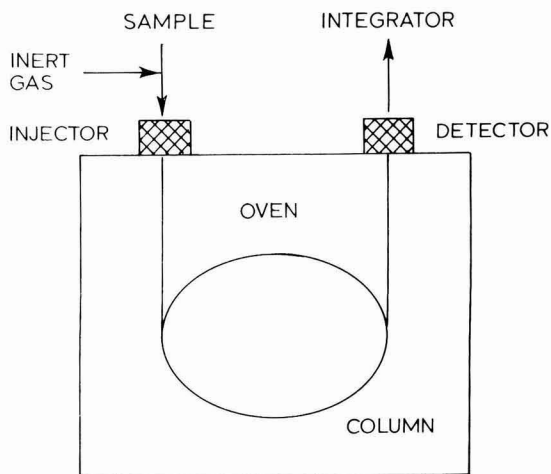


Figure 11. Gas chromatograph

through the column in the vapour state by an inert gas, such as helium or nitrogen. During this period, the sample component molecules spend some of their time in the stationary liquid phase and the rest of their time moving through the column in the gaseous phase. A component which is highly soluble in the stationary liquid phase will be retained longer in the column than a less soluble component. The relative retardation of the two components will differ according to their different solubilities and they will eventually emerge separately at the end of the column. The detector will respond to each component eluted over a period of time and a chromatogram will be obtained. Each component is identified by its characteristic retention time in the column, which is a constant under constant conditions. Compilations of chromatographic relative retention time data are obtainable⁸.

The underlying principle behind the above explanation is the Partition Law, which states that the partition coefficient K , is a fundamental value for a given solute, where

$$K = \frac{\text{concentration of solute in phase 1}}{\text{concentration of solute in phase 2}}$$

The chromatogram also provides quantitative data on the components identified, since the area under the chromatogram can be used to quantify the component causing that peak.

Gas chromatographs are all essentially sophisticated ovens, but the accessories and conditions used may be varied to suit the sample analysed. For example, the detector may be flame-ionization, thermal conductivity, electron-capture or mass spectroscopic; the column may be packed, capillary polar or non-polar; the oven, detector and injector temperatures are variable and the injectors may be automatic or manual, split, direct or splitless. All these factors influence the quality of the separation and detection of the sample components.

GC applications

Solvent analysis

The single most important use of a gas chromatograph in

| Component | Material analysed | Method used |
|----------------------|--------------------------------------|----------------------|
| 1. Solvent | Paints, Resins, Inks | Direct GLC |
| 2. Monomers | Acrylic polymers, Emulsions | Pyrolysis - GLC |
| 3. Retained solvents | Paint flakes, Packaging, Can linings | Headspace - GLC |
| 4. Fatty acids | Oil modified resins | Saponification - GLC |
| 5. Organic vapours | Atmosphere | Charcoal tubes - GLC |

Figure 12. GC applications in the coatings industry

the coatings industry is in the analysis of solvent compositions. The paint, resin or ink is thinned with an appropriate solvent and then injected directly into the column. A pre-column (flash vapouriser) is usually used to trap the involatile components and to avoid column contamination.

For example, Figure 13 is the chromatogram obtained when a mixture of the following five commonly used solvents were injected into a non-polar fused silica capillary column: acetone, toluene, ethylene glycol monoethyl ether (Union Carbide "Cellosolve"), methyl isobutyl ketone (4-methyl-pentan-2-one) and an aliphatic distillate cut.

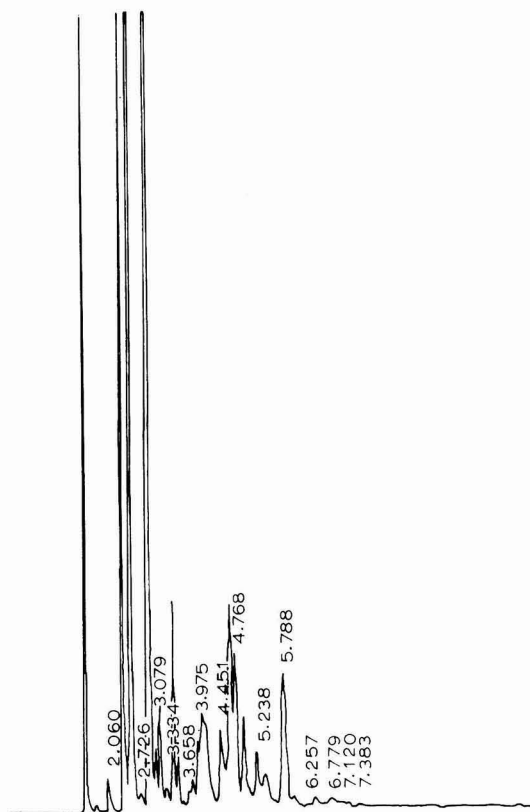


Figure 13. GC chromatogram of solvent mixture

The oxygenated components were removed from the solvent mixture by the addition of a blend of concentrated

sulphuric and phosphoric acids. Figure 14 is the chromatogram of the hydrocarbons (aliphatic and aromatic) remaining after the acid treatment.

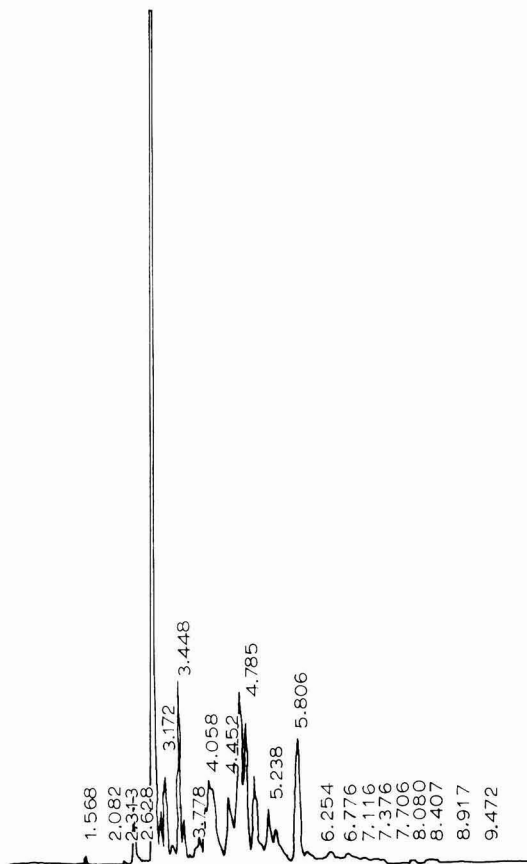
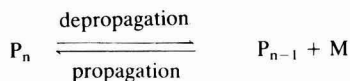


Figure 14. GC chromatogram of the hydrocarbons

The aromatic component, toluene, was removed from the hydro-carbon mixture by nitrating with concentrated nitric acid and the aliphatic residue was extracted in dimethyl sulphate (Figure 15).

Monomer analysis

The monomer composition of acrylic and methacrylic polymers is determined by pyrolysis-gas chromatography (P-GC). A thin film of the polymer is deposited onto the platinum filament of a pyrolysis probe. The probe is placed in a heated chamber connected to the chromatograph injection port. The sample is pyrolysed by rapidly heating the filament to approximately 600°C and the pyrolysates are swept onto the column in the stream of carrier gas. Most acrylic polymers undergo radical depolymerization, producing monomer as the predominant volatile product:



where P_n is the polymer containing n monomer units (M)

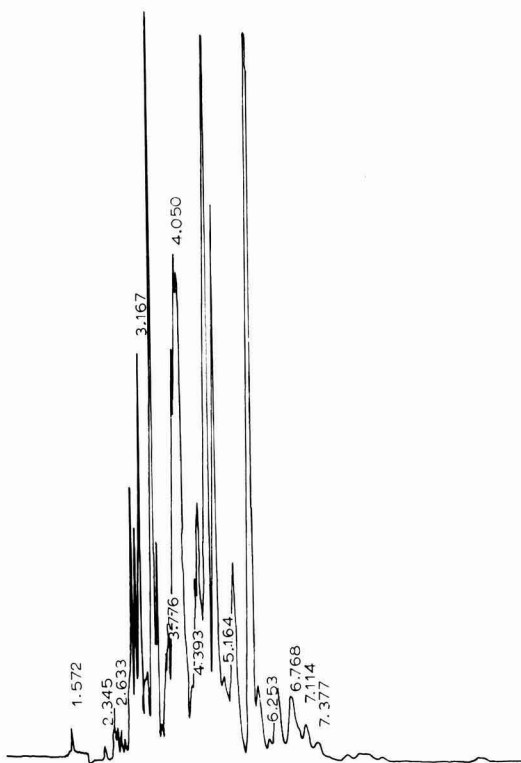


Figure 15. GC chromatogram of aliphatics

Although other polymers do not unzip into monomer units as readily as acrylics, most of them give characteristic fingerprint pyrograms which may be for identification purposes.

Retained solvent analysis

Residual solvents retained in packaging materials and can linings are known to cause product off-flavour and off-taste. These retained solvents may be detected at parts per million levels by headspace-gas chromatography (H-GC).

This technique involves heating the coating material in a closed container to vapourize the retained solvents into the headspace. A volume of the headspace vapour is withdrawn by a gas tight syringe and injected into the gas chromatograph for analysis.

Fatty acid analysis

Fatty acids may be converted to their volatile methyl esters and directly analysed by gas chromatography.

Organic vapour monitoring

Personal monitoring of the industrial atmosphere for organic vapours involves pumping a known volume of air (usually 10-20 litres) through a charcoal tube over an eight hour shift. The solvents adsorbed onto the charcoal are later desorbed with carbon disulphide and analysed by gas chromatography.

Discussion

This paper has attempted to give the reader an idea of the type of work done by the analytical chemist in the coatings industry. In particular, the importance of instrumental techniques has been stressed, but it must not be forgotten that wet chemistry still has a place in the laboratory.

Instrumental analysis is a fast-moving and highly sophisticated field which has made a great contribution to the analysis of surface coatings.

References

1. "An Infrared Spectroscopy Atlas for the Coatings Industry", Federation of Societies for Coatings Technology, Pennsylvania, USA, 1980.
2. "The Coblenz Society Desk Book of Infrared Spectra", Craver, C. D. (Ed), Second Edition, April 1982.
3. "Atlas of Polymer and Plastics Analysis", Hummel, D. O. and Scholl, F., Verlag Chemie, Weinheim, Second Edition, 1982.
4. "Compilation of Chromatographic Data", ASTM AMD 25A S-1.

Bibliography

1. Lear, J. B., "Analysis of Paint", *JCT*, March 1981, Vol. 53, No. 674, 51.
2. Lear, J. B., "Analysis of Paint Films", *JCT*, April 1983, Vol. 53, No. 675, 63.
3. Haslam, J. and Willis, H. A., "Identification and Analysis of Plastics", *Iliffe Books Ltd, London*, 1965.
4. McNair, H. M. and Bonelli, E. J., "Basic Gas Chromatography", *Consolidated Printers, California, USA*, Fifth Edition, March 1969.
5. Haken, J. K., "Gas Chromatography of Coating Materials", *Marcel Dekker, New York*, 1974.

OCCA conference

OCCA Conference 1985 – New substrates, new materials, new problems?

As already announced the Association's Biennial Conference will take place in Edinburgh 26-29 June 1985. Twenty-three papers on the theme will be presented in four sessions in the mornings and afternoons of Thursday 27 June and Friday 28 June. Details of both the technical programme and the social programme were given in the registration form, a copy of which was enclosed with each issue of the February *JOCCA*. In addition the technical programme appeared on page 53. Further copies can be obtained from Priory House; photostats of the registration form will be acceptable but all applications must be accompanied by the appropriate remittance.

The photochemistry of the peroxodisulphate ion in aqueous alcohol solutions

C. G. Roffey

National Printing Ink Co. Ltd, Industrial Estate, Chichester, Sussex PO19 2UE

Summary

A survey of peroxodisulphate ion photochemistry is presented, including original unpublished material by the author. Knowledge of this system is important with respect to emulsion polymerization for the surface coatings industry.

The photolysis of this ion in water and interpretation is described together with that in the presence of scavengers, both organic and inorganic. Quantum yields found are reported and mechanisms proposed for the free radical species participating, an observed chain reaction with alcohols, metal ion photo-oxidation and ionic strength effect. A model based upon a solvent cage effect is outlined.

Introduction

There appears to be a paucity of data in the published literature concerning the photochemistry of the peroxodisulphate anion, compared to its analogue hydrogen peroxide.

Interest has centred however on the former species owing to the emulsion polymerization studies of McGinniss and Kah^{1,2}.

These researchers have used both peroxodisulphate and peroxomonosulphate as generating systems for the sulphate ion radical. Their studies involved the quenching of sulphate anion radicals by vinyl monomers in emulsion polymerization systems. Interpretation is made of the competition between this and associated colloid stabilizers or surfactants. The latter materials may be compounds with scavengeable hydrogen atoms such as alcohols, polyethers, mcecaptans etc, thereby leading overall to a lowering of the polymerization rate efficiency and decrease in polymer solution viscosity.

A short summary concerning peroxodisulphate photochemistry appears in the work of Ogata *et al*³, which is a useful supplement to the present report.

The absorption process and fates of the excited state

The absorption of a 253.7 nanometer quantum by the peroxodisulphate ion leads to an electronically excited state. The electronic transition is probably one involving the transfer of a non-bonding electron on one of the oxygen atoms to the anti-bonding sigma orbital localised in the O-O region. Such an $n \rightarrow \sigma^*$ transition would lead to a weakening and possible rupture of the O-O bond. Both the peroxodisulphate ($S_2O_8^{2-}$) and peroxomonosulphate ions are reported to be cleaved at this bond to form the sulphate anion radical¹⁻⁶ ($SO_4^{\cdot-}$).

The energy of a 253.7 nanometer quantum is 112 Kcal mole⁻¹ which is in excess of that required for the dissociation. Thermally the energy needed to dissociate the

O-O bond is about 51 Kcal mole⁻¹, whereas photochemically it is of the order 80 Kcal mole⁻¹.

Quenching of the excited state, ($S_2O_8^{2-*}$) \rightarrow $S_2O_8^{2-}$ by collision with solvent molecules is another possible fate. This is only possible if the process of collision is rapid compared with the time of vibration of the O-O bond. If it isn't then photo-dissociation is the more probable fate of ($S_2O_8^{2-*}$). The asterisk denotes excitation energy.

The excited peroxodisulphate ion ($S_2O_8^{2-*}$), would be produced in a solvent cage, (denoted by round brackets). The likely dissociation products are either the formation of two sulphate ion radicals in the cage by homolytic cleavage:-



or the formation by heterolytic cleavage of sulphur tetroxide, SO_4 and a sulphate ion

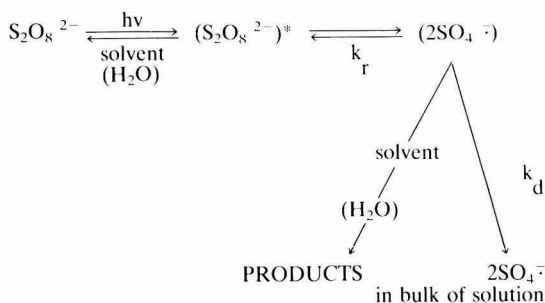


The species SO_4 is dubious in this case owing to its lack of free radical characteristics and instability above 73°C.

Once these two sulphate ion radicals have been produced in the solvent cage they can undergo any combination of the four processes below:

1. Recombination in the cage as the activation energy for recombination is low.
2. Diffusive transfer to the bulk solution, each radical then possessing its own solvent cage.
3. A concerted reaction of the two radicals with the cage if the cage consists of scavenger molecules.
4. Reaction with the solvent cage.

The processes described can be summarised as:



where k_r represents recombination and k_d diffusion rate constants.

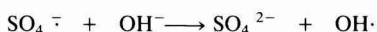
Evidence for the photochemical production of free radicals was afforded from the formation of acrylonitrile polymer observed in this work.

Properties of the sulphate radical anion

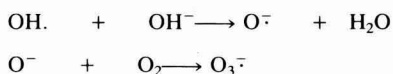
Dogliotti and Hayon⁶ using the technique of flash photolysis have found that the sulphate anion radical has an absorption maximum at 455nm ($\epsilon = 460 \text{ M}^{-1} \text{ cm}^{-1}$) and a half life of 300 μs .

Dimerisation of the sulphate ion radicals occurs in the absence of inorganic and organic scavengers. This reforms $\text{S}_2\text{O}_8^{2-}$ with the second order rate constant of $3.7 \times 10^8 \text{ M}^{-1} \text{ s}^{-1}$.

The radical is stable under acid and neutral conditions but very unstable in alkaline solution, having a large rate constant of $7.3 \times 10^7 \text{ M}^{-1} \text{ s}^{-1}$ because of its fast reaction with hydrogen ions.



These workers found a transient spectrum assigned to the ozonide ion, $\text{O}_3^{\cdot -}$ on the flash photolysis of aerated alkaline peroxodisulphate, probably from the reaction



This transient has a maximum absorption at 430nm and its decay is pH dependent and concentration dependent on $\text{O}_3^{\cdot -}$ ions

Much evidence for the existence and properties of the $\text{SO}_4^{\cdot -}$ anion radical is reviewed in the thermal properties of the peroxodisulphate ion by House¹⁵.

Photolysis in water

The photochemical decomposition of this ion in water has been investigated several times at 253.7 nm, but the overall quantum yield, has been found to have several different values. Crist⁸ obtained the value of 1.0 in neutral and alkaline solutions.

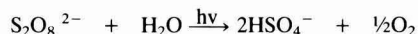
Heidt⁹ found a value of 0.58 ± 0.03 and later¹⁰ he found a value of 0.567 ± 0.005 .

Tsao and Wilmarth¹¹ obtained a value of 0.79 ± 0.07 .

The contributing factor to these discrepancies was thought by Barrett and Roffey to be attributed to the presence of inorganic and organic impurities.

Roffey¹² therefore has redetermined this value using purified water, low in both of these. He found the value to be 0.58 ± 0.04 in accordance with the original of Heidt.

Morgan and Crist^{13,14} found the photochemical decomposition to be represented by

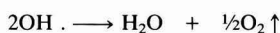
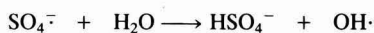


with no evidence of any intermediate reaction.

Currently the proposed mechanism for this aqueous photolysis is thought to proceed via the intermediate hydroxyl ($\cdot\text{OH}$) radical and sulphate anion ($\text{SO}_4^{\cdot -}$) radical. It may be postulated as



1985(5)



Effect of Alcohols

When an organic substrate is present there is the possibility of hydrogen abstraction from the substrate by the sulphate ion radical,



and this reaction would be expected to compete with that of the sulphate ion radical and water,



or in alkaline solution,

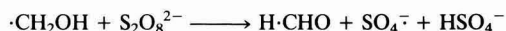


There is considerable evidence against the hydrolysis reaction (1) occurring in the presence of a scavenger. The flash photolysis work of Dogliotti and Hayon suggests that the sulphate ion radical is acid stable but decays at a $\text{pH} > 8.5$ to produce hydroxyl free radicals as in reaction (2). In the present treatise the solutions used were all found to be acidic. Further evidence for the absence of this hydrolysis reaction was afforded by an ionic strength effect found in the photochemical oxidation of chromium ions (see later) of this work, for which a charged species reacting is necessary to cause the effect, (ie not $\text{OH}\cdot$ which is neutral but probably, $\text{SO}_4^{\cdot -}$, which is charged). In addition, Bhat the reacting radical species is other than $\text{OH}\cdot$ was indicated from a comparison of experimentally determined relative rate constants in the Cr (III) system with those previously determined for hydroxyl radicals by other researchers.

In the presence of methanol, ethanol and iso-propanol, a chain mechanism was found by the author^{12, 14, 16} to be induced, which for methanol can be postulated as



followed by,



An initial increase in ϕ_T has been shown to occur for all three alcohols at low concentration, thereafter decreasing to a limiting ϕ_T value at high alcohol concentration, see figures 1, 2 and 3. This tended to approach the value for the primary quantum yield, ϕ_p which was measured for methanol using allyl alcohol as a scavenger. The primary quantum yield ϕ_p , may be described in terms of the molecules of peroxodisulphate decomposed in the photoact per quantum of radiation absorbed. This in effect represents the fraction of the peroxodisulphate molecules, which after absorbing quanta decompose to give two sulphate ion free radicals that take part in secondary reactions. The other absorbing molecules must either be involved in electronic deactivation before decomposition can occur, or in recombination processes which do not lead to net decomposition.

The results for all the alcohols gave an overall quantum

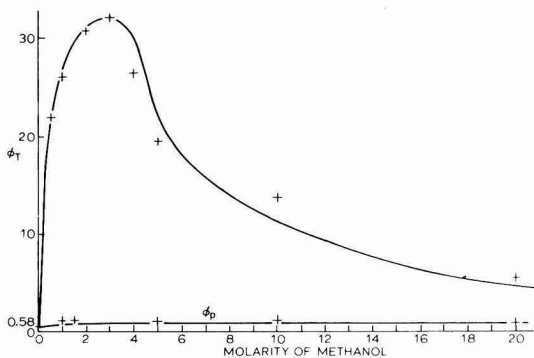


Figure 1. Quantum yield for peroxodisulphate decomposition in aqueous methanol. From: *Photopolymerization of Surface Coatings* (1982) by C. G. Roffey, ISBN 0471 10063 reprinted by kind permission of John Wiley & Sons Ltd.

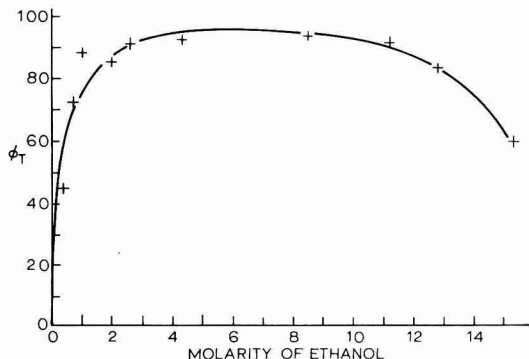
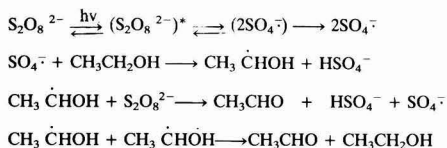


Figure 2. Quantum yield for peroxodisulphate decomposition in aqueous ethanol.

yield greater than unity over a large range of alcohol concentrations. This led to the conclusion that this was the result of a chain reaction. A photo-chemically initiated chain reaction which is terminated by dimerisation of the chain carriers would in fact occur when the quantum yield for the disappearance of the absorbing molecule is inversely proportional to the square root of the intensity of the absorbed radiation. This was observed for this system.

The kinetic proposed schemes for ethanol and 2-propanol are similar.

For ethanol the mechanism postulated is,



Whilst for 2-propanol it is postulated as,

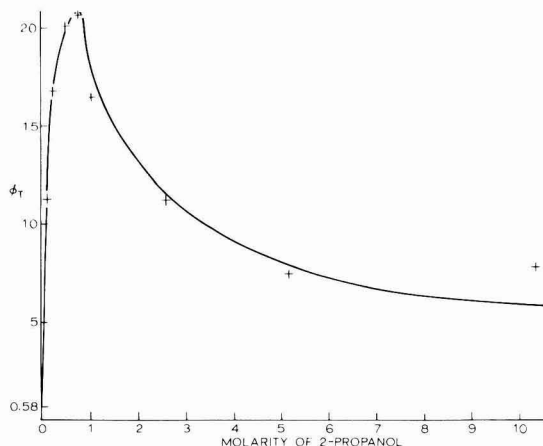
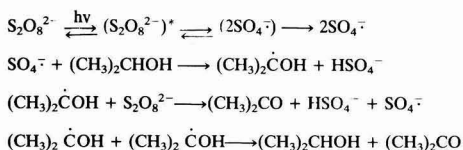


Figure 3. Quantum yield for peroxodisulphate decomposition in aqueous 2-propanol

It is interesting to consider the scavenging of the hydrogen atoms in the three alcohols. Scavenger would be expected to occur on the α -carbon atoms, owing to the weakening of the CH-bond by the electron donating effect of the alkyl groups. Also, oxygen being more electronegative in character than carbon will attract the bonding electrons between itself and the carbon atom and hence induce a partial positive charge on the carbon atom. From these considerations the hydrogen atom abstraction reaction rates with the sulphate ion radical would be expected to be in the order, $(\text{CH}_3)_2\text{CH}(\text{OH}) > \text{CH}_3\text{CH}_2\text{OH} > \text{CH}_3\text{OH}$

This was not found to be so in practice, the order being: $\text{CH}_3\text{CH}_2\text{OH} > (\text{CH}_3)_2\text{CH}(\text{OH}) > \text{CH}_3\text{OH}$, and may be due to steric and statistical factors. The steric factor can be interpreted in terms of the bulky methyl group hindering access of the sulphate ion radical to the α -carbon atom hydrogen atom. This would tend to reverse the expected order depicted above.

The statistical factor can be understood in terms of the number of α -carbon hydrogen atoms available to sulphate ion radical attack, methanol having three, ethanol two, and 2-propanol one. This would tend to reverse the expected order.

In the presence of allyl alcohol a value of about unity was determined for ϕ_p in water with no methanol present and was found to be maintained at around this value over the whole range of methanol concentrations used. This is in agreement with the value of unity to be expected as a maximum value for ϕ_p . This maximum of unity is a theoretical value resulting from one peroxodisulphate molecule disappearing for one quantum absorbed, assuming no recombination or deactivation occurs.

Hence, $\phi_p^{\text{MAX}} = 1$ with respect to disappearance of peroxodisulphate.

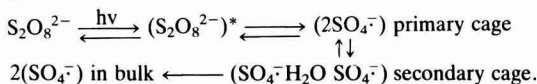
With respect to sulphate ion radical production this maximum value would be 2, ie $\phi_p^{\text{MAX}} = 2$.

The value obtained for a primary yield will depend on its method of measurement. In this work it was measured with respect to peroxodisulphate decomposition and hence any recombination of sulphate ion radicals will lower this value from the theoretical maximum value. This is demonstrated by the apparent overall quantum yield obtained for peroxodisulphate decomposition in water, which was found to be 0.58. It is probably a result of cage recombination of sulphate ion radicals, diffusion from the cage and to reaction of the caged sulphate ion radicals with water. It is necessary therefore to define two more primary quantum yields as well as $\phi_{-S_2O_8^{2-}}^{MAX} = 1$, one in the presence of water alone,

(no scavenger), $\phi_{-S_2O_8^{2-}}^{H_2O} = 0.58$ and one in the presence

of a scavenger, $\phi_{-S_2O_8^{2-}}^{SCAVENGER} \approx 1$

This value of about unity in the presence of a scavenger can be interpreted in terms of the existence of a secondary cage, resulting from a diffusive displacement of one of the sulphate ion radicals over about a molecular diameter, allowing a molecule of water to come between the two sulphate ion radicals. This configuration may be regarded as a secondary cage in which the two sulphate ion radicals still have a possibility of again becoming adjacent or of diffusing further away from each other in the bulk of the solution where they would possess their own solvent cages, i.e.



In this situation, recombination is competing with diffusion and scavenger molecules and therefore once this secondary cage has been formed recombination is difficult owing to the presence of a water molecule in the cage. A scavenger would not be expected to compete with primary recombination unless the scavenger is at a mole fraction of unity but in this secondary cage the scavenger can compete with the recombination at any concentration.

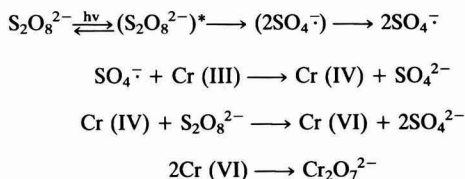
The variation of the primary yield for the peroxodisulphate system is interesting as it differs from that of the hydrogen-peroxide system studied by Barrett *et al*¹⁷.

Owing to the presence of this secondary cage, the $(2SO_4^{\cdot-})$ recombination was found to be slower than $(2OH^{\cdot})$ recombination. The main reason for this is probably because of the two negatively charged species in the solvent cage of the former.

Effect of metal ions

In the same work, a photo-oxidation has been found to occur at 253.7 nanometers between peroxodisulphate and the metal ions chromium (III) and cerium (III). It was ensured that the peroxodisulphate ion was predominantly absorbing the incident ultra-violet radiation in preference to the metal ions. Electron transfer reactions have been observed from metallic ions such as Ruthenium and Cerium to peroxodisulphate by other workers when the converse

occurs^{18,19}. For chromium (III), it is suggested from an observed ionic strength effect found in this work that the sulphate ion radical is the oxidising species. The proposed mechanism is:

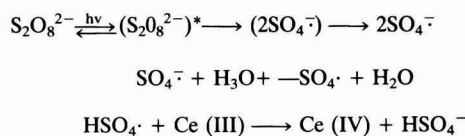


The quantum yield for dichromate formation was determined as 0.18 ± 0.2 . This reaction for the oxidation of Cr (III) is known to occur thermally, i.e.



but slowly, and is generally catalysed by the presence of silver ions. The photo-oxidation could provide a simple, analytical method for the determination of such ions, without the complications of contaminating metal catalytic ions.

In the acid Ce (III) system, HSO_3^+ is the postulated oxidising species, the proposed mechanism being,



The quantum yield for cerium (IV) formation was determined as 1.05 ± 14 .

The introduction of various oxidisable substances into both metallic ion systems thereby depressed the quantum yields for dichromate and cerium (IV) ion formation. This can be interpreted in terms of the extra species competing with chromium (III) or cerium (III) ions for the sulphate anion radical. This permitted a series of relative rate constants to be obtained for each oxidizing radical species.

A comparison was made of the peroxodisulphate system at equivalent light intensity with the hydrogen peroxide system of Barrett *et al*¹⁵ for methanol in the maximum region of the chain reaction. It was found that the chain length of the peroxodisulphate reaction was approximately an order higher than that of the hydrogen peroxide system.

This tends to suggest that for the species of free radicals studied, the oxidising power is in the sequence



It appears that this observation is in contrast to some data found by other workers²⁰.

An extension of this work to include studies of the peroxidiphosphite ion under similar conditions could prove fruitful to elucidate properties of the PO_4^{2-} and $HPO_4^{\cdot-}$ anion radicals. Some work has been performed in this area by Lussier²¹ who found the quantum yield for $P_2O_8^{4-}$ decomposition in water at 253.7 nanometers to be 0.8. In the presence of 2-propanol and ethanol in basic solution the values found were 1.7 and 1.05 respectively,

although under acid conditions these quantum yields rose to a maximum value of about 46 for 2-propanol and 27 for ethanol.

Acknowledgements

The original work contained in this survey was in part that undertaken by the author for his doctorate work (PhD Thesis 1970) at Chelsea College, London University. This work was in collaboration with Dr J. Barrett, lecturer in chemistry at the above college, to whom the author is indebted.

References

1. McGinniss, V. D. and Kah, F. A., *J. Coat. Technology*, 1977, **49** (634) 61.
2. McGinniss, V. D. and Kah, F. A., *J. Coat. Technology*, 1979, **51** (654), 81, *Chem Abstr*, 1979, 91, 124047u.
3. Yoshiro, O., Kohtaro, T. and Kyoji F., "The chemistry of functional groups, peroxides", *John Wiley & Sons Ltd*, Edited by Patai, S., 1983, 757.
4. Chawla, O.P. and Fessenden R. W., *J. Phys. Chem.*, 1975, **79**, 2693.

5. Norman, R. O. C., Story, P. M. and West, P. R., *J. Chem. Soc (B)*, 1970, 1087.
6. Dogliotti, L. and Hayon, E., *J. Phys Chem.*, 1967, **71** 2511.
7. Dorfman, L. M. and Taub, *J. Am. Chem. Soc.*, 1963, **85**, 2370.
8. Crist, R. H., *J. Am. Chem. Soc.*, 1932, **54**, 3939.
9. Heidt, L. J., *J. Chem. Physics*, 1942, **10** 297.
10. Heidt, L. J., Mann, J. B. and Scheider, H. R., *J. Am. Chem. Soc.*, 1948, **70** 3011.
11. Tsao, Maak-Sang and Wilmarth, W. K., *J. Phys. Chem.*, 1959, **63**, 346.
12. Roffey, C. G., Ph.D. thesis, Chelsea College, London University, 1970.
13. Morgan and Crist, *J. Am. Chem. Soc.*, Part 1, 1927, **49**, 16.
14. Morgan and Crist, *J. Am. Chem. Soc.*, Part 2, 1927, **49**, 338.
15. House, D. A., *Chem. Rev.*, 1962, **62**, 185.
16. Roffey, C. G., "Photopolymerization of surface coatings", *John Wiley & Sons Ltd*, ISBN 0471 10063, 1982, 3.
17. Barrett, J., Mansell, A. L. and Ratcliffe, R. J. M., *Chemical Communications*, No. 1194, 1968, 48.
18. Bolletta, F., Juris, A., Maestri, and Sandrini, D., *Inorganic Chimica Acta* **44**, 1980, L 175-L 176.
19. Mathews, R. W. and Sworski, T. J., *J. Phys. Chem.*, 1975, **79**, 684.
20. Maruthamuthu, P. and Neta, P., *Phys. Chem.*, 1978, **82**, 710.
21. Lussier, R. J., *Diss Abstr. Int B* **31**, 1970, 103, *Chem. Abstr.*, 1971, **74**, 106669t.

Modification of shellac with some unsaturated acids

Prasad, N., Agarwal, S.C., Dasgupta, A.K., and Gupta, P.C.

Indian Lac Research Institute, Namkum, Ranchi-834010

Summary

Modification of shellac in combination with unsaturated acids namely maleic, crotonic and acrylic in the presence of benzoyl peroxide as an initiator, improves adhesion strength and also film properties such as hardness, flexibility and impact resistance. The maximum bond strength on iron surface is 0.35, 0.28 and 0.23 ton/sq. inch respectively for compositions based on the above unsaturated acids whereas plain shellac gave only 0.08 ton/sq. inch.

Introduction

Shellac, one of the most valuable natural resins of animal origin possess a good number of desirable properties for its use in various industries. But due to certain inherent drawbacks such as low softening and melting points, poor water, alkali and solvent resistances, etc., it is facing hard competitions from tailor made synthetics. In order to overcome this challenge attempts have been made in the past by several workers¹⁻³ to make suitable modifications in shellac so as to upgrade its properties in specified directions. A critical appraisal of the past work revealed that an average molecule of lac, which contains five hydroxyl groups, one free carboxyl group (acid value \approx 74), an aldehyde group

(partly free and partly combined), an unsaturation (iodine value \approx 16.4) and molecular weight (\approx 1000), had been esterified in two ways; (a) either by esterifying the free carboxyl with mono or polyhydric alcohols^{2,5}; or (b) by esterifying the hydroxyls with saturated mono or polybasic acids⁴. Some casual attempts have, however, been reported with unsaturated acids also^{5,6}. The ultimate products obtained so far, have limited industrial applications. The present paper describes a study on the modification of shellac with unsaturated acids namely maleic, crotonic and acrylic, and the possible utilisation of these products in various fields.

The modification of shellac with unsaturated acid (using their different percentages on the weight of shellac), was attempted by adopting different methods: (a) fusion of mixture at 150°C; (b) dissolution of mixture in an inert solvent (tetrahydrofuran) or (c) in a common solvent (alcohol) and carrying out the reaction at reflux temperature in the presence of an initiator. It has been observed that the desired modification only takes place using the last method (c) judging by the performance of the properties investigated. Only two compositions from each set were chosen on the basis of their properties, for the pre-

Table 1

Preparation and properties of shellac-esters modified with unsaturated acids (shellac 40.0g, benzoyl peroxide 1% in each composition)

| Composition No. | Unsaturated acids (percentage) | Chemical constants | | |
|-----------------|--------------------------------|--------------------|----------------|--------------|
| | | Acid value | Hydroxyl value | Iodine value |
| A | Maleic acid (4%) | 50.4 | 175.4 | 24.7 |
| B | Maleic acid (5%) | 48.6 | 145.7 | 24.2 |
| C | Crotonic acid (2%) | 48.0 | 136.8 | 24.9 |
| D | Crotonic acid (5%) | 51.0 | 142.5 | 25.4 |
| E | Acrylic acid (5%) | 52.3 | 159.3 | 25.0 |
| F | Acrylic acid (6%) | 53.8 | 160.5 | 25.2 |
| Control | | 74.7 | 239.0 | 16.4 |

paration and are reported in the experimental section (see Table 1).

Experimental

Materials

Shellac: Used; dewaxed or waxy shellac.

Chemicals: Used; maleic acid, crotonic acid, acrylic acid, benzoyl peroxide, alcohol (methylated spirit distilled), tetrahydrofuran, chloroform, acetone, hydrochloric acid and trichloroethylene. (All the reagents used throughout the investigation were of pure grade).

Methods and techniques

Shellac (40g, dissolved in 100ml alcohol), unsaturated acids (maleic, crotonic and acrylic acids taken in different percentages on the weight of shellac) and benzoyl peroxide (1 per cent) were placed separately in a round-bottom flask. The mixture was refluxed at water bath temperature for seven hours) whereupon a uniform solution resulted. The excess of the solvent was distilled off from each composition. An alcoholic solution (30 per cent) of each composition was prepared for testing film properties in one set and in another set, alcoholic solution (10 per cent) of each composition was prepared for testing the adhesive properties.

Panels of iron (7.5 x 2.5 cm) with cleaned and polished surfaces were used for the adhesive test. The panels were finally cleaned with trichloroethylene in order to remove oily and greasy impurities from the surface. The prepared solution of each composition (1 ml) was spread over the surface of the panel so as to cover 1 sq. inch area and allowed to dry overnight at room temperature. The panel was then baked at $95 \pm 1^\circ\text{C}$ in a steam oven for one hour. Two such panels were placed one upon the other, overlapping the coated area (1 sq. inch) and were then hot

pressed for one hour in a carver press at 150°C and 200 lbs/sq. inch pressure, after which they were allowed to cool to room temperature.

Methods of analysis and evaluation

The acid, hydroxyl and iodine values of the various compositions (see Table 1) were determined according to the standard methods⁷ adopted for shellac.

Resistance of the films on glass slides to water, hydrochloric acid, sodium hydroxide solution (1 per cent each) and organic solvents (ethyl acetate, acetone and chloroform) were tested by dipping them for 48 hours at room temperature and the extent of damage, such as blushing or film failure noted. The scratch hardness, flexibility and impact resistance of the films on tin panels were tested by an automatic scratch hardness tester (1mm steel ball), a conical mendral (3 mm) and a falling block instrument respectively. The properties are recorded in Table 2.

In the adhesive test, the bond strength of each pair of panels was determined by Hounsfield Tensometer. Five pairs of bonded panels were tested for each experiment and the mean value was taken. The values are recorded in Table 3.

The infrared analysis of the samples was carried out on Perkin-Elmer IR spectrophotometer Model 137 (neat KBr disc) at CDRI, Lucknow, India.

Results and Discussion

It is evident from Table 1 that the hydroxyl value decreases (a drop of ≈ 100 units) and iodine value increases during the course of reaction indicating desired esterification of hydroxyl groups of shellac with carboxyl group of unsaturated acid has taken place primarily. This is further evidenced by IR spectra of all the compositions having sharp absorption band for $\text{CH} = \text{CH}$ at 1640 and 760 cm^{-1} and reduced intensity peak for OH at $3200\text{-}3300 \text{ cm}^{-1}$ as compared to control. The drop in acid value, however, reveals that some esterification between the carboxyl group of shellac and the hydroxyl group of solvent alcohol might have inevitably occurred as a secondary reaction.

It is worth mentioning that the softening point of the above compositions improved considerably as compared to the control.

Film properties

Finish

The air dried and baked (one hour at 150°C) films of all the compositions on glass and tin surfaces were smooth, glossy and non-tacky.

Hardness

There was appreciable improvement in the hardness of film for all the compositions other than the control.

Flexibility and impact resistance

All the air-dried and baked films passed these tests.

Table 2
Film properties of compositions

| Composition No. | Film appearance (air-dried) | Water resistance | | Acid resistance (1%) | | Scratch hardness (g) | | Flexibility | | Impact resistance | |
|-----------------|------------------------------|------------------|---------|----------------------|---------|----------------------|---------|-------------|----------|-------------------|----------|
| | | A.D. 3 | Baked 4 | A.D. 5 | Baked 6 | A.D. 7 | Baked 8 | A.D. 9 | Baked 10 | A.D. 11 | Baked 12 |
| 1 | 2 | | | | | | | | | | |
| A | Smooth, glossy and non-tacky | B | NB | NB | NB | 800 | 900 | P | P | P | P |
| B | Smooth, glossy and non-tacky | B | NB | B | NB | 900 | 900 | P | P | P | P |
| C | Smooth, glossy and non-tacky | B | NB | B | NB | 1000 | 1000 | P | P | P | P |
| D | Smooth, glossy and non-tacky | B | NB | B | NB | 1000 | 1000 | P | P | P | P |
| E | Smooth, glossy and non-tacky | B | NB | B | NB | 900 | 900 | P | P | P | P |
| F | Smooth, glossy and non-tacky | B | NB | B | NB | 900 | 1000 | P | P | P | P |
| | Control (Lac) | B | B | B | B | 300 | 600 | F | F | F | F |

B = bluish; N.B. = no bluish; A.D. = air dried; Baked = baked at 150°C for 1 hour; P = passed; F = failed.

Table 3
Adhesive strength of compositions (Iron surface)

| Comp No. | Bond strength (ton/sq. inch) |
|---------------|------------------------------|
| A | 0.35 |
| B | 0.29 |
| C | 0.24 |
| D | 0.28 |
| E | 0.23 |
| F | 0.21 |
| Control (Lac) | 0.08 |

Water, acid and alkali resistance

All the air-dried films blushed within 30 minutes while the baked films were resistant to acid and water (seven days). The alkali resistance, however, remained unsatisfactory as both types of film blushed within a short period.

Solvent resistance

With regard to the resistance of films to solvents such as ethyl acetate, chloroform and acetone, some improvement was noted as compared to the control.

The foregoing results reveal that the modification of shellac with maleic, crotonic and acrylic acids improves most of the film properties and the present findings are in close agreement with the results of Venugopalan⁵.

Adhesion properties

From the results given in Table 3, it will be found that incorporation of unsaturated acids into shellac, increases the bond strength as compared to earlier reports^{6,8} on iron surface. The maximum bond strength was 0.35, 0.28 and

0.23 ton/sq. inch respectively for the compositions prepared by maleic, crotonic and acrylic acids modifications. These values, are two-three times higher than the value obtained for control but at par with the results of Islam *et al*⁶.

Possible uses

Thus it appears that the composition based on shellac modified with unsaturated acids (maleic, crotonic and acrylic acids) may find applications in the field of surface coatings, plasticizers and adhesives.

Acknowledgement

The authors are thankful to Dr. B. B. Khanna, Director, Indian Lac Research Institute for his keen interest in the work. The technical assistance of P. Devi, T. K. Saha, M. Ekka and K. K. Prasad is also acknowledged.

Received 20 August 1984

References

1. Khanna, B. B. and Tripathi, S. K. M., *Chemicals and Petro-Chemicals Journal*, 1979, Oct.
2. Sankarnarayanan, Y., Indian Lac Res. Inst., A.R., (1949-50) 18-19, (1950-51), 17-18.
3. Sengupta, S.C., Ghosh, D., Das, R. K. and Sinha, A. R., *JOCCA*, (1980), 63, 15.
4. Bhattacharya, R. and Gidwani, B. S., *London Shellac Res., Bur. Tech Paper No. 15*.
5. Venugopalan, M., *Current Sci.* (India), (1939), 814; Indian Lac Res. Inst., A.R., (1936-37) 9; (1937-38) 9; (1938-39), 9-10; (1949-50), 17.
6. Islam, M., Ghose, P. K., and Gupta, P. C., *Res and Ind.*, 27 (1982), 178.
7. "A Handbook of Shellac Analysis", *Indian Lac Research Inst.*, 2nd Edition, 1952.
8. Prasad, N., Ghose, P. K. and Bhomwick, T., *Res. and Ind.* (1972) 17 98.

next month's issue

The Honorary Editor has accepted the following papers for publication in the April issue.

- Corrosion monitoring of coated steel using a "Multi-layer sandwich" cell by S. Feliu, J. M. Bastidas and M. Morcillo
Rheological parameters of red iron oxide-alkyd paints: Their dependence on pigment content and on solubility parameters of solvents by K. M. A. Shareef and M. Yaseen
Maleinization of rosin and isolation of maleopimaric acid by M. C. Shukla, V. S. Gandotra, K. L. Bedi and A. K. Vasishtha

Thames Valley Section

Concrete coatings in hostile environments

The meeting was held at the Crest Motel, Beaconsfield on Thursday, 17 January, 1985.

The speaker Mr E. French, of Taylor Woodrow Ltd, gave in his introduction the details of the involvement of his company in a project to design and test coatings suitable for concrete structures to be erected in the Barents Straights of the Arctic. It was envisaged that four structures were to be built each having a minimum radius of 965 metres built to a conical design with wall angles varying from 17° to 60°—the area of each construction to be coated in the region of 10,000 sq. m.

The major design consideration is the effect of ice on the structure and how to minimise the various ice forces collectively known as "ad freeze".

Firstly it was necessary to establish whether standards existed covering the profile required for the concrete surface prior to coating, but their literature search revealed a limited source of information.

To begin the project it was necessary to establish the effects that ice will have on such a substrate. The speaker went on to list them as follows:

1. Frictional effects due to thawing
2. Freeze thaw cycle
3. Hydrostatic pressure
4. Impact testing
5. Seabed abrasion

For each of the above, a detailed account was given of the individual test methods devised by the project team and with the aid of slides the speaker was able to demonstrate the effect of anticipated environmental effects on a wide range of coatings and how individual coatings varied in their performance.

To conclude, it was in the speakers' opinion, that the major contributing factor to coating failure was the inability of the coating to match as near as possible the thermal coefficient of expansion of concrete.

An interesting discussion period followed the talk and the vote of thanks was proposed by Mr Alan Fell.

The trouble with bridges

The above meeting was held at the Crest Motel, Beaconsfield, on Thursday, 21 February, 1985. The speaker, Mr G. Hill, of ITI Consultancy Services, commenced his talk by describing the major problems encountered when engaged in the inspection and detailing of a suitable specification for both pretreatment and painting of bridges. This subject was

well illustrated with excellent slides, much of which covered the speaker's involvement last year with the repainting of Tower Bridge.

The evening closed with a lively question time and a vote of thanks was proposed by Mr. P. Watson.

J. A. Gant

Midlands section

Table-Top Exhibition—Trent Valley Branch

The Trent Valley Branch held a "Table-Top" Exhibition on Thursday, 22 November, at the British Rail's School of Transport, on London Road, Derby.

Elcometer provided the subject of the exhibition of their well known film thickness measuring instruments. Dr P. E. Doherty and Mr J. Podvoiskis gave information and advice to visitors during the hours of 12 noon to 5 p.m. when several visitors brought along samples for evaluation.

In the evening session, Dr Doherty gave a technical presentation in which he outlined many of the practical solutions to problems of film thickness measurement and analysis associated with such coatings as micaceous iron oxides, chlorinated rubber and rough surface profiles.

Various forms of instrumentation available were explained, with the help of a slide show.

A lively discussion period followed a break for refreshments.

Approximately 30 people attended and amongst the paint users were people from Rolls Royce, Raleigh Cycles and British Rail.

A vote of thanks was proposed by Mr J. P. Bourne.

J. R. Küchen

Manchester section

The conservation of paintings

On Friday, 11 January, 1985, a Ladies' Evening Lecture and Buffet was held at the Georgian House, Blackrod, near Chorley. Eighty members and guests heard a lecture by Mr B. Cardy of the Manchester City Art Gallery entitled "The Conservation of Paintings".

Following the lecture an excellent buffet was enjoyed by all present, making the evening a very pleasant social occasion.

Mr Cardy gave a well illustrated talk, detailing the various techniques used by artists of yesteryear and the many problems associated with the restoration of these pictures. A sobering thought for modern paintmakers is the fact that egg tempera, an old formulation using egg as the binding medium, is one of the most durable paint systems. The task of the restorer is complicated by both the techniques of the original artist, the environments in which

Pearson Panke Test Instruments for

| | | |
|----------------|---------------|---------------------------------|
| Film thickness | Gloss | Heat stability |
| Hardness | Colour | Gelation properties |
| Adhesion | Salt Spray | Minimum film forming properties |
| Abrasion | Cupping tests | |

And full range of equipment from
ERICHSEN - LIST MAGNETIK - TOYOSEIKI - COESFELD - GEL

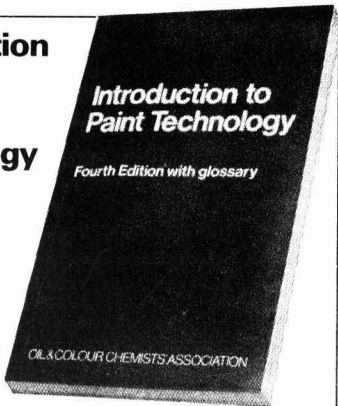
Consult the experts

For further information please contact



Reader Enquiry Service No. 377

Introduction to Paint Technology



The Introduction to Paint Technology, of which over 25,000 copies have been sold, forms an excellent introduction to the whole field of surface coatings and related technologies.

To: OCCA, 967 Harrow Road, Wembley,
Middlesex HA0 2SF, UK

Please send copy/ies of Introduction to Paint
Technology at £7.50 each (inc. p&p) to the following
address (*BLOCK CAPITALS*):

.....
.....
.....

I enclose remittance (payable to OCCA) for £

occa meetings

the painting has been stored over the years and the efforts of previous restorers, which in many cases seem to do more harm than good.

Cleaning is the initial step and must proceed with great caution as it is quite possible to remove the painting as well as the dirt. Following cleaning repairs to tears, scuffs and missing paint areas are made. The supporting structure of the canvas or wooden panel is also given attention and the canvas is relined on the back using fibreglass and hot melt adhesive.

This most interesting lecture was followed by a period of questions and the vote of thanks was offered by Mr Graham Fielding.

R. G. Handley

Robotics

Monday, 11 February, at 6.30 p.m. was the time when 25 OCCA members represented the human race in meeting the robots manufactured by Dainichi-Sykes Ltd., at the (ironic?) location of the Walton Summit Employment Centre, Bamber Bridge, Preston, Lancashire.

Mike Podmore, Technical Sales Engineer of the Company, extended a warm welcome to us and described the history of his Company. Brief details included the following facts:

1. Dainichi-Sykes have three factories at the Walton Summit area.
2. They have 16 robots in their range.
3. Of the original 50 companies involved in robotic manufacture, there are 25 remaining.
4. Dainichi-Sykes was formed in 1981 with technical collaboration between Sykes Group, Huddersfield, and Dainichi Kiko, Japan. the joint Company has a Japanese president supported by two British and two Japanese directors.
5. Present business operations rely on importation of sub-assemblies but plans have been made for a manufacturing facility on the present site.

Mike Podmore went on to describe the Company's approach to potential markets:

1. Sales marketing demonstration administration and training sections.
2. Feasibility studies and assessment trials.
3. Joint ventures, e.g. Jaguar Cars Ltd and Dainichi-Sykes; £50m over five years.

It was stated that the ideal situation was the following sequence: Site survey/total manufacturing process/islands of automation/successful automation. This system gives a total systems approach.

occa meetings

Of the 120 employees, 95 have an industrial engineering bias. We were then shown a 20 minute video film which clearly illustrated the benefits of industrial robotics in relation to this:

1. Assembly of shower base units.
2. Bottle manufacture.
3. Welded assembly process for hospital beds.

We then visited the factory demonstration area where robotic machinery was working, unattended, of course, and the tasks being performed included:

- Vacuum blasting of aircraft parts.
- Adhesive application to the underside of Jaguar bonnets.
- Loading computer discs into a polishing machine.
- Cartons containing firelighters being transferred/stacked on pallets.
- Deburring of castings.

Paint spraying robotics for the car industry were discussed along with teach boxes identified as the main method of programming. It was stated that 4,500 pro-

grammes may be required to produce 450 steps in a robot movement sequence.

A discussion period followed and Mike Podmore answered many questions on a variety of subjects which included:

1. Robotic benefits—pay back 18 months/two years but improved quality/no breaks/no fatigue/24 hour working.
2. The "psychological effect"—effect on employment levels.
3. Accuracy—stationery robots—two thou; rail mounted robots less accurate.
4. Technological advances—effect on existing robots—it was stated that it was the software that was easily replaced.

Dainichi-Sykes were proud of their technical director, John Rhodes, who has been appointed to the chair in Advanced Manufacturing Technology at Salford University.

This very interesting visit was concluded on behalf of the visiting humanoids by our Chairman, Gordon Robson, who proposed a vote of thanks which was warmly applauded in non-robotic fashion.

F. B. Windsor

BASILDON CHEMICALS: SILICONES FOR SOLVING PROBLEMS

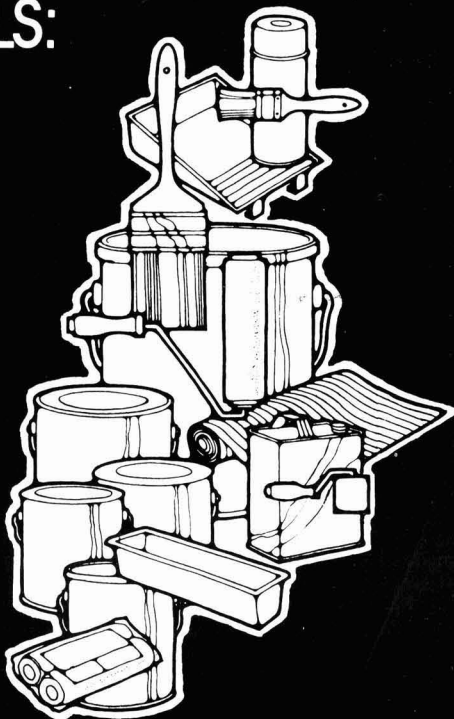
Antifoams
Release Agents
Fluids
Greases
Paint Additives
Polish Additives
Antistats

For further information contact

Basildon Chemical Company Limited

Kimber Road, Abingdon, Oxon OX14 1RZ

Telephone: (0235) 26677



Surge in chemical investment

Capital spending in the chemical industry is expected to rise sharply in 1985 with investment topping £1,400m by the year's end. Allowing for inflation this is nearly 30 per cent up on the estimated final figure for 1984. And, according to the Chemical Industries Association's annual investment intentions survey published on 2 April, 1985, the spending surge is likely to continue into 1986, although at a slightly diminished rate, whilst a significant decline in spending is expected in 1987.

Commenting on the figures, Martin Trowbridge, CIA's Director General, said: "To some extent the apparent strength of the upturn has been exaggerated by projects brought forward in response to the Government's new regional policy and tax legislation. Companies want to get through the gate before it closes. At the same time this helps to explain some of the decline predicted for 1987".

Reader Enquiry Service No. 30

UK distributor appointed by Du Pont

Du Pont de Nemours International SA of Geneva, Switzerland, has announced the appointment of Charles Tennants & Co (London) Ltd as distributor for its range of "Elvacite" acrylic resin products. Tennants will have primary responsibility for the United Kingdom and the Republic of Ireland with effect from April, 1985.

"Elvacite" acrylic resin products are methacrylates, and are used in a wide range of applications such as printing inks, plastic coatings, adhesives, marine paints, hot stamp foils, aerosols and toners. They have been sold in Europe for over 15 years.

Reader Enquiry Service No. 31

Graco UK rationalise distributor network

Graco UK Ltd, the Wolverhampton based company specialising in fluids handling and product finishing equipment, has rationalised its UK distributor network.

Five major distributors now cover the UK. They supply Graco finishing equipment: air and airless, manual and electrostatic spraying equipment plus the latest air-assisted airless spray equipment, as well as coating machines and plural component material application systems.

Graco's major distributors are: Inmar Ltd, Spraon International Ltd, Spray Equipment Services Ltd, Tri-Spray (UK) Ltd and Wilson Supply Ltd.

Commenting on the new distributor network, Graco UK's Chief Executive, Mike Cowley said, "We maintain a direct sales operation for high technology equipment—including robots, fluids handling and specialised pumping—and for the motor industry. But we rely heavily on efficient, well-managed distribution to make sure we reach all areas of the market place. This new network should, therefore, act as the springboard for even more expansion".

Present at the Wolverhampton headquarters when the new distributor agreements were signed was Mr Barry Calhoun, Vice President, International Operations of Graco Inc of Minneapolis, USA.

Reader Enquiry Service No. 32

Th. Goldschmidt's new subsidiary

The West German speciality chemical manufacturer, Th. Goldschmidt AG of Essen, has recently formed a new subsidiary, Tego Chemie Service GmbH. This subsidiary has the task of expanding the already significant market that Goldschmidt has for its products in the surface coating industries, and particularly, paint and printing inks. Tego Chemie Service will be operating on a world-wide basis.

In the UK, Tego Chemie Service will be operating as a division of Th. Goldschmidt Ltd under the control of Angus Smith. The company believes that by creating this new venture, and concentrating resources it will be able to satisfy far more effectively the demanding needs of this technically oriented market. At present, the range of products being marketed include a wide number of surface active additives in addition to a range of silicon resins. However, the main emphasis will be on problem solving and customer service, utilising the knowledge and abilities of the extensive research facilities in Essen.

Reader Enquiry Service No. 33

New company uses BP Chemicals Rigipore

A new company, Wellfill Insulation Ltd, has been formed to handle French Kier's insulation business. The company, which was formerly known as French Kier Insulation Ltd, has been created as a result of a management buy-out.

Wellfill Insulation Ltd, is said to have had a considerable amount of experience working with a number of boroughs, county councils and development councils,

all very much concerned with energy conservation. Buildings treated range from private homes to hospitals.

Under an agreement signed in 1983 between BP Chemicals and French Kier Insulation Ltd, the terms of which still stand, Rigipore expandable polystyrene made at BP Chemicals plant at Wingles in France is supplied to an expander in Essex. From there the expanded bead is delivered in purpose-built articulated lorries, either to Wellfill's depots or direct to larger contracts. It is then blown with a bonding agent through pre-drilled holes into cavities using special lorry-mounted equipment.

Reader Enquiry Service No. 34

Bradite Paints receive ministry approval

Bradite Paints Ltd has been awarded official recognition of the quality of its paints and lacquer by receiving Ministry of Defence Standard 05/24 approval. The approval calls for a high standard of quality control through all stages of manufacture, with the majority of the work being entrusted to Bradite.

As manufacturers of an extremely wide selection of paints and lacquer, from NATO green to standard household emulsions, Bradite Paints claim to specialise in being able to supply small quantities at very short notice to meet users' demands.

This service, it is claimed, will be of great use to a wide range of MoD approved suppliers, from heavy equipment manufacturers wishing to provide matching paint to touch-up equipment after transit or installation, to companies supplying small run items.

Said Bradite general manager Jim Dean, "We are naturally very pleased to have been awarded official Ministry of Defence approval as this shows that we can and do meet the highest possible standards of production and quality control. We look forward to further developing our business in all areas as all our products for both civil and military applications are produced to the same high standard".

Reader Enquiry Service No. 35

Paint sales improving

The encouraging news that volume sales of paint rose by 4.7 per cent in the last three months of 1984, was announced by the Paintmakers' Association of Great Britain who issued their quarterly sales bulletin in March. Exports were particularly strong and building paint sales also improved

compared with the same period in 1983. The good news was tempered by the sharp drop in industrial coatings sales over the same quarter.

Exports of building and industrial paints remain buoyant and sales increased by an impressive 12.1 per cent in the fourth quarter. These figures are in line with the trend of generally improved sales performance and a comparison with equivalent figures for 1982 shows an overall increase of 30 per cent in export volume sold during the last two years.

The 9.0 per cent rise in building paints represents the third consecutive quarterly improvement in sales volume. A comparison with 1983 figures shows volume to have increased by 3.7 per cent in total. Sales of building paints have shown a steady improvement for some 3 years, and are now significantly higher than at their most depressed period during 1981.

In contrast with export and building paints markets, industrial coatings were disappointing during November and December, with a drop in sales volume of 5.0 per cent for this quarter. The only sector showing improvement was vehicle refinishing—where sales rose slightly. Marine and automotive however, fell sharply. The average selling prices for paint during the quarter showed an increase of 3.1 per cent—a similar increase to that for the year as a whole. Raw material costs, however, which account for approximately half the industry's total manufacturing costs, increased by 8.6 per cent over a comparable period.

Michael Levene, director of the Paint-makers' Association, was concerned that margins were still being squeezed by ever higher raw material costs and severe price competition in the market place.

Reader Enquiry Service No. 36

products

Mebon introduce two new floor coatings

Mebon plc, the Nottinghamshire based high technology industrial coating manufacturers, have introduced two new floor coatings systems; Mebaqua and Mebothane. These are said to be suitable for the majority of possible general industrial applications.

Mebaqua is a non-toxic, water solvated coating which can be applied by plant owners' existing labour force. Drying

rapidly to a low sheen finish and available in a limited range of colours, Mebaqua is said to be both easy to use and repair. It is particularly suitable for the light industrial pedestrian traffic to be found in laboratories, canteens, corridors, offices, etc.

Mebothane is a moisture-cured urethane coating, available in a range of six standard colours and is suitable for the general purpose industrial floors to be found in warehouses, production areas, etc. Mebothane resists damage from fork trucks, knocks, scrapes and other abuse.

Designed for the protection of concrete and similar substrates they are claimed to be hardwearing, durable and resistant to a multitude of chemicals. They are fast curing and therefore create the minimum of disruption during application. Both coatings can be applied by brush or roller, airless spray or conventional spray.

Reader Enquiry Service No. 37

Paint line efficiency

Obtaining information on the temperature profile of a component as it passes through the paint curing oven is crucial to calibrating the production line to provide optimal line speed, minimal rejects and reduced energy usage.

A new British invention, the Datapaq Tracker, is said to provide a quick and easy in-situ analysis both of how well the curing process is performing, and how its efficiency can be maximised.

This development is the brainchild of scientist-turned-businessman, John Bates, who has formed his Cambridge-based Datapaq company to manufacture and market the product.

The microprocessor-driven Datapaq Tracker is said to provide an independent, modular, battery-powered data acquisition, processing and storage system which will analyse the curing profile of a component at up to four separate points on its surface, and give an instant comparison of actual performance against the specified curing schedule. This previously unobtainable information allows for immediate alteration of line speed or oven temperature to maximise curing performance efficiency, and points the way to substantial productivity gains, or savings in energy and reject costs.

One of the Datapaq Tracker's advantages, is that the complete recording and analysis system (combining both hardware and software) is totally portable, which permits a technical service engineer to carry and operate it in-situ when visiting



The portable Datapaq Tracker

customers. The Datapaq Tracker, in this form, contains the information gathering and storage modules, an insulated container, and a specially-programmed NEC notebook computer with four colour mini-printer.

Datapaq is confident that users of its Tracker innovation will achieve a minimum improvement of 3-5 per cent in paint line efficiency within the first three months of regular use, and confidently expects 90 per cent of its customers to recoup the system's £4,150 cost within the first six months of operation.

Reader Enquiry Service No. 38

Esso Chemical's new low odour high boiling solvent

Esso Chemical have announced the addition of a new high boiling solvent, Exxsol D190-10, to its low odour, low aromatically Exxsol range.

The new product is the ninth member of the range. It is expected to find wide application in paints, reaction diluents and similar areas, where low odour and low reactivity are vital.

Exxsol D 170-190 is said to have a particularly low aromatics content and when used as a reaction diluent gives improved product yields in cases where aromatic molecules interfere with the process. Also the good evaporation characteristics resulting from its narrow boiling range facilitate recovery and recycling.

Tony Cross, Esso Chemical Solvents marketing manager, commented, "So far

there has been a great deal of interest in other solvents in the range for specialised applications. Recently for example ICI Paints Division has begun to use Exxsol D 150-200 for a low odour reformulation of their eggshell paint. The low odour avoids the pungent, irritating and persistent smell that is the problem with conventional eggshell paints and makes the paint ideal for situations where people must occupy the area concerned either whilst it is being painted or very soon after. During the development stages we worked closely with ICI to ensure that the use of this especially pure solvent did not detract from the normal performance of the paint. We are expecting that similar novel uses will be found for the new Exxsol D170-190 and are looking forward to working with other customers in their development".

Reader Enquiry Service No. 39

Hoechst announce that Reflex Blue production will meet market needs

Due to the increase in popularity in recent years of offset printing (in many cases at the expense of letterpress), Hoechst has recently announced that its existing capacity for Reflex Blue production can meet demands for this growing market both in Europe and overseas.

Reflex Blue is the designation used by Hoechst for its alkali blue pigments. In order to compensate for the yellow undertone of carbon black pigments, black inks are often toned with alkali blue, either in flushed form or as a powder. Alkali blue pigments have therefore become firmly established in the market and their use will continue to grow with offset printing.

Steps have also been taken by Hoechst to enable production to be increased by about 30 per cent to keep pace with the demand.

Reader Enquiry Service No. 40

Aluminium window finish lasts twice as long

The decorative appeal of aluminium window frames is claimed to have received a boost following recent independent tests to BS.3900, Part F3 performed on the Valtec powder coating for metallic substrates. Carried out by Tioxide Central Laboratory, the MARR tests show only slight loss of gloss and virtually no chalking at 4,000 hours, which is double the recommended British Standard test procedure time scale for weatherability.

The weatherability standard is a measure of the coating's exterior performance, par-

ticularly in the areas of gloss retention and chalking, and is usually based on accelerated tests over 2,000 hours. At this point most coatings display gloss reduction and chalking—except it is claimed Valtec.

This degree of longevity for a polyester dry powder coating is believed to be a significant breakthrough in the decorative and protective coating of metallic substrates, especially externally exposed aluminium panels, profiles and window frames.

The tests were carried out on aluminium panels coated in white Valtec with a gloss finish of at least 65 per cent reflectance measured at 60 per cent incidence to DIN 67530. After 4,000 hours the coating was examined for signs of chalking. The surface showed no chalking in excess of the minimum illustrated in the photographic standards of ASTM-D 659:1980. The coating was then washed with a one per cent aqueous solution of mild detergent, to examine it for checking, cracking, flaking and loss of gloss. No such deterioration was visible and the gloss level was 55 per cent—a loss of just ten units.

Aluminium is an increasingly popular building material because of its lightness and ease of maintenance. But with more architects specifying coloured finishes, the surface coatings industry has had to develop coatings equally maintenance free and capable of withstanding the rigours of the weather and environmental pollution. correctly applied, Valtec is said to perform well in excess of a British Standard which other systems strive hard to meet.

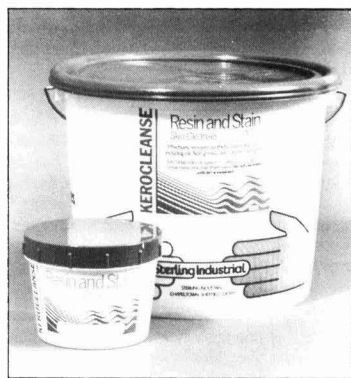
Recently introduced, Valtec is manufactured in the UK by The Valentine Varnish and Lacquer Company which was recently acquired by Glasurit Beck, a wholly owned member of the BASF Group. It is available from stock in 24 selected RAL and BS 4800 colours.

Reader Enquiry Service No. 41

Resin and stain skin cleanser

An alternative cleanser for the removal of resins and stains from the skin has been added to the range available from skincare specialists, Sterling Industrial. Said to be adept at the removal of resinous substances, the additional cleanser is claimed to be particularly effective at cleansing skin of persistent staining agents—certain inks, dyes and other compounds.

Kerocleanse resin and stain skin cleanser can be used in conjunction with a conventional cleanser or simply rinsed with water. A sufficient amount of resin and stain skin cleanser is worked into the skin until it can be seen that the staining agent is



Kerocleanse Resin and Stain Skin Cleanser

loosened. A conventional cleanser may then be applied in the normal way and washed off with clean water.

Where the skin is likely to be contaminated with either resins or stains it is advantageous in assisting the skin cleansing process to apply a barrier cream before commencement of work. Sterling Industrial's Kerodex Anti-Solv. barrier cream is said to be specially formulated for this purpose.

Reader Enquiry Service No. 42

New masonry coating

Incorporating the very latest advances in masonry coating technology, an entirely new type of textured coating named System 7 has been launched by the Worldwide Protective Coatings Division of Camrex Ltd.

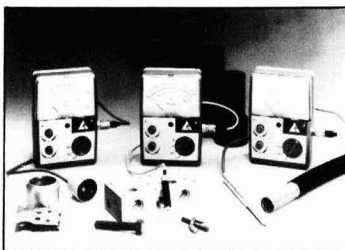
System 7 is said to have an attractively textured surface appearance and is available in 16 carefully selected architectural colours. Designed primarily for external use, it is said to have excellent weathering properties and, in contrast to most conventional masonry coatings, to have a life expectancy of at least 10-15 years. The use of System 7 can, it is claimed, therefore lead to significant savings in the cost of regular coating maintenance and, as importantly, minimise the disruption and inconvenience that this can cause.

System 7 is said to be resistant to environmental attack by acid rain and alkali and entirely successful in combating the unsightly effects of mould growth and retained dirt. About 20 times thicker than conventional coatings, its textured finish is said to be suitable for disguising hair-line cracks, patch rendering and other substrate defects. The special formulation of System 7 also provides a very effective barrier against carbon dioxide penetration. Known as carbonation, this is a major

cause of rebar corrosion and subsequent concrete failure.

Due to its advantages, System 7 will replace Camrex's existing Tex-Gard masonry coating system which has been used extensively throughout the UK for over ten years.

Reader Enquiry Service No. 43



Sheen's SM range of analogue coating thickness gauges.

Thickness gauges

To complement their popular SS range of digital thickness instruments, Sheen Instruments Ltd have introduced their new offer the SM range of analogue coating thickness gauges, which is to be low cost.

Features include very long battery life (up to 1000 hours—1.5V battery) and a selection of probes to carry out standard measurements and more difficult job such as measurement of coatings on small surface area, the inside of pipes, tubes, cross sections, and also curved surfaces. All probes are compensated against temperature effect and are, within limits, interchangeable.

The SM range measures non-magnetic coatings (including electroplate) on steel.

Reader Enquiry Service No. 44

both workers and the general public by requiring dangerous substances including preparations and other mixtures to be adequately packaged and suitably labelled, come into full effect on 1 January, 1986, or for smaller packages, on 1 January, 1987.

The guidance booklet provides general advice on the CPL Regulations themselves, and on their supporting documents—the Authorised and Approved List and the two Approved Codes of Practice on classification and labelling of substances for supply and/or conveyance by road, and on packaging of dangerous substances for conveyance by road.

Reader Enquiry Service No. 45

literature

Guide to classification, packaging and labelling of dangerous substances regulations published

A guide to the classification, packaging and labelling of dangerous substances regulations 1984 has been published by the Health & Safety Executive, and costs £5.00

The new regulations, which are designed to increase health and safety protection for

New publication describes Binks new Taiver airless spray systems

A new booklet from Binks-Bullows Ltd of Brownhills, Walsall, describes the new range of Taiver portable airless spray systems, which is designed for painting furniture, ships, aircraft, large steel structures and buildings and many other industrial applications.

Reader Enquiry Service No. 46

OCCA NEWS

News of members

Mr W. F. McDonnell

Mr W. F. (Bill) McDonnell, works director of Goodlass Wall & Co. Ltd, the Liverpool based paint manufacturers, has retired after 41 years with the company.

Commencing as a laboratory assistant, Bill McDonnell was chief chemist before taking responsibility for the factory as works manager in 1965. He was appointed director in 1973.

Well known in the paint industry, Bill McDonnell has served as vice president of the Oil & Colour Chemists' Association, and as a past chairman of Manchester section. He has also been a member of the National Joint Industry Council (Paint, Colour and Lacquer), and was inaugural chairman of the N.W. Cheshire & Clwyd branch of the Institute of British Management.

Mr A. Sheath

Mr Alan Sheath has been appointed chief chemist of Lusteroid Paints Ltd, the largest New Zealand-owned manufacturer of industrial coatings.

Mr Sheath, 36, joined Lusteroid from the United Kingdom in April 1981 as an industrial chemist. In 1982 he was appointed deputy chief chemist, and in 1983, works chemist with responsibility for quality control.

He is a participating member of the Auckland section of the Oil & Colour Chemists' Association.

professional grade

Professional Grade

At a meeting of the Professional Grade Committee held on 10 April 1985 the following admissions were made:

Transferred from Associateship to Fellowship

van Londen, Anton Matthijs (*General Overseas - Netherlands*)

Admitted to Associateship

Greeff, Mattheus Philip (*Transvaal*)

Admitted to Licentiatehip

Pratt, Douglas Edward (*Ontario*)
Shroff, Farrokh (*Ontario*)

new members

Ordinary Members

Barnes, J. C., MSc (Auckland)
 Burgess, S. (Manchester)
 Burrell, S. W., BSc (Midlands)
 Ham, L. T. (Ontario)
 Ireland, A. J., BSc (Auckland)
 Jonas, D. M. (London)
 Krajic, C. F. J. (Manchester)
 McLean, S. (Ontario)
 Melville, I., BSc (Newcastle)
 Moulton, D. V. (Newcastle)
 Naidoo, T. (Natal)

Reilly, A. H. (London)
 Smith A. K. A., MSc, PhD (Thames Valley)
 Smith, A. L., BSc (Thames Valley)
 Tanipanichkul, S., BSc (General Overseas - Thailand)
 Usher, R. D., BSc (London)
 Walton, S. (Newcastle)
 Weal, D. J. (Wellington)
 Williamson, L. F. (Transvaal)
 Wothington, S. M. (Midlands)

Associate Members

Campbell, B. I. (Transvaal)

Crous, G. (Transvaal)
 Lambert, L. H. (Transvaal)
 Lembeck, B. K. P. (Transvaal)
 Niederheitmann, K. J. (Transvaal)
 Woolley, N. J. (Transvaal)
 Wrigley, K. J. (Auckland)

Registered Students

Payne, P. B. (London)
 Sharples, J. (Manchester)
 Tomlinson, P. B. (Manchester)

CLASSIFIED ADVERTISEMENTS

APPOINTMENTS

Expansion of Car Refinish Activities

PPG Industries (UK) Limited

PPG Industries have recently acquired the Car Refinish Paint interests of International Paint, based in Ladywood, Birmingham.

We are now looking to recruit people to strengthen our Refinish team in the following areas:

- Field Sales Manager (Birmingham based)**
- Sales Representatives**
- Experienced Development Chemists**
- Colour Management**
- Technical Service Representatives**
- Demonstrators**

If you are interested in joining the largest paint company in the World, and becoming part of our expansion plans,

contact:—**Mr. K. I. Moulder,**
Personnel Manager, P.P.G.
Industries (UK) Ltd., Rotton Park
Street, Birmingham B16 0AD.
 Tel: 021-455 9866.

Applications invited from both men and women



WORLD LEADERS IN
AUTOMOTIVE
FINISHES



Professional & Executive Recruitment

PIGMENT SALES REPRESENTATIVE

Ciba-Geigy have for many years held a dominant position in the U.K. pigment market and have built-up a first class reputation for quality and service.

We have a vacancy for a young, enthusiastic person to sell our products, initially to the printing ink industry in the South East of England.

The principal industries we supply are the paint, plastics and ink industries. They are technically complex and our sales force require the ability to talk to customers in their own language. The person we are looking for will be highly motivated, between the ages of 25 and 30 and will ideally be a science graduate. Candidates without a science degree but with relevant experience in the pigments or a related industry will be considered where they match the requirements in other respects. He or she must be prepared to live anywhere in the U.K. in line with the pigments business' future needs and as part of his or her career development, which will not necessarily be confined to the Marketing function.

Conditions of employment are those you would associate with a major company in the chemical industry. A starting salary within the range £10,000-£12,000 is likely.

A company car is provided and relocation expenses are available where appropriate.

If you feel you meet the requirements and are interested, please write or ring for an Application Form or send full c.v. to: T. C. Graham, Personnel Manager, Ciba-Geigy Pigments, Hawkhead Road, Paisley PA2 7BG.
 Tel: 041-887 1144.

CIBA-GEIGY

MISCELLANEOUS

FOR SALE: Small technical library comprising many surface coatings and other reference books. Includes bound issues of *JOCCA* from 1932 through to 1972 and a full set of the OCCA Paint Technology Manuals. Full list available on receipt of a stamped addressed envelope.—Reply **Box 553.**

SERVICES



SHELLACS NATURAL RESINS GUMS & WAXES

A. F. SUTER & CO. LTD.

**Swan Wharf 60 Dace Road
 London E3 2NQ**

Tel 01-986 8218 Gram Suterresin London E3
 Contact B. A. Slater



Dow Chemical is a large and successful international chemical company, with European Headquarters in Horgen near Zurich. In Europe, the Middle East and Africa we employ 12,000 people and operate 60 Sales Offices, 26 Manufacturing Locations and 9 Research Centers. The products we manufacture and sell vary

from base chemicals to plastics, speciality and agriculture chemicals as well as pharmaceutical products.

In our Technical Service & Development Center in Horgen, Switzerland, where we employ 200 people, we have an opening for a

CHEMIST WITH COATING EXPERIENCE

You will be a member of our international chemicals development team and have responsibility for the application development and technical service of DOWANOL* P-series glycolethers solvents.

Dow is one of the major producers of these propylene oxide based glycolethers which are finding increasing uses in the paint, detergent, cosmetic and a variety of other industries.

Frequent contacts with our customers will give you the opportunity to assess the technical feasibility of new products and their application in the market place.

- We require
- ★ A degree in Chemistry
 - ★ Minimum three years of experience in one of the forementioned application areas, preferably in coatings
 - ★ Working knowledge of the English and another European language
 - ★ Willingness to travel internationally
 - ★ Approximately 30 years of age

- We offer
- ★ A job where you will work in close liaison with other functions such as research, manufacturing, sales and marketing
 - ★ A stimulating working environment, where you can apply creativity in solving technical problems
 - ★ Career opportunities based on your performance
 - ★ Attractive employment conditions in a young international environment

If you are interested please call or write to us for an application form.

Mrs. M. E. Kaelin, Dow Chemical Europe, Bachtobelstr. 3, CH-8810 Horgen. Tel. 01041-/728.21.11)

* TRADEMARK OF THE DOW CHEMICAL COMPANY



Tailor-made preservation with MERGAL®

for example: in-can preservation

- MERGAL® K6N suitable for the preservation of all types of paint and glue
- MERGAL® K7 for products which must comply with the BGA XIV recommendations or which must be formaldehyde-free
- MERGAL® K8 preserving agent with enhanced fungicidal effect
- MERGAL® KM 206 highly concentrated preserving agent for use in very small quantities
- MERGAL® AF powdered product. Particularly suitable for solid mixes, e.g. for mould prevention treatment

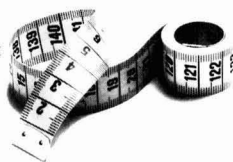
for example: fungicidal protection

- MERGAL® S88 fungicide and algicide for darker colours. Can be supplied in powder or paste form
- MERGAL® S89 universal fungicidal and algicidal powder
- MERGAL® S95 universal liquid fungicide and algicide
- MERGAL® S96 fungicide and algicide for solvent-containing systems

Take advantage of the extensive experience of our laboratories. Our technical service department will be pleased to advise on the benefits of preservation of your products with MERGAL®.

Coupon

Use the coupon to request a consultation and information material. You will get a reward.



Name _____

Position, Department _____

Address _____

JO 1/85

Riedel-de Haën

Aktiengesellschaft
Wunstorfer Str. 40, D-3016 Seelze 1/Hannover
Federal Republic of Germany
Telephone (51 37) 7 07-216, Telefax (51 37) 9 1979
Telex 9 21 295 rdhs d

In other countries we are represented by the sales organization of the Hoechst AG.

Reader Enquiry Service No. 308

JOCCA

AT YOUR SERVICE to develop finer, more durable products

The fast, economical weather tests of
SUB-TROPICAL TESTING SERVICE, INC

established 1929

Sub-Tropical Testing Service is known and utilised world-wide

Our 55th Year



Send for free
 Sub-Tropical colour
 brochure

**Sub-Tropical Testing gives fastest Natural Weather tests available for ★ paints ★
 chemical coatings ★ plastics ★ textiles ★ fabrics ★ related products ★**

Wir waren die ersten, welche einen Prüfdienst dieser Art für Lacke, Kunststoffe, chemische Beschichtungen, Stoffe und Gewebe aller Art, Farbstoffe und verwandte Erzeugnisse in den U.S. möglich machten.

Mr C. Hubbard Davis, Founder, Sub-Tropical Testing Service, Inc. (1904-1977)
 PO Box 560876, 8290 S.W. 120th Street, Miami, Florida, U.S.A. 33156

Phones: (305) 233-5341
 Cable: SUBTROPIC, Miami

Reader Enquiry Service No. 309

JOCCA

**READER ENQUIRY SERVICE
 MAY 1985**

Name

Job Title

Company

Address

.....

Country

Telephone

For further information on adverts or editorials enter the
 Reader Enquiry Service Number/s in the boxes below:

This enquiry will be forwarded to the company/ies
 concerned.

JOCCA

**Priory House, 967 Harrow Road, Wembley,
 Middx., HA0 2SF, England**

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

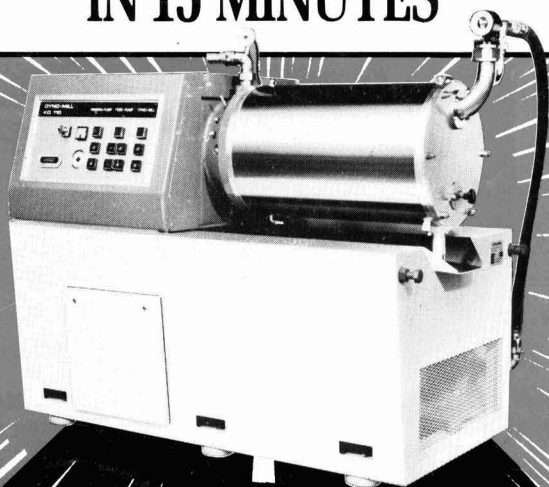
Photocopy or cut out form and send to above address:

INDEX TO ADVERTISERS

Further information on any of the products advertised in this Journal may be obtained
 by completing the Reader Enquiry Service form. The Reader Enquiry Service numbers
 are shown in brackets below.

| | | |
|--|----------|-------|
| | A | |
| Atlas SFTS | (314) | cover |
| | B | |
| Basildon Chemical Co. Ltd | (373) | 125 |
| | G | |
| Glen Creston Ltd | (374) | cover |
| Graco UK Ltd | (375) | cover |
| | M | |
| Magnesium Elektron Ltd | (376) | ii |
| Manville (GB) Ltd | (219) | i |
| | P | |
| Pearson Panke Equipment Ltd | (377) | 124 |
| | R | |
| Riedel-De-Haen AG | (308) | v |
| | S | |
| Sachtleben Chemie GmbH | (364) | iv |
| Sanyo-Kokusaku Pulp Co. Ltd | (283) | ii |
| Sub-Tropical Testing Service Inc | (309) | cover |

OUT OF THE RED AND INTO THE BLACK IN 15 MINUTES



The DYNO-MILL was the major breakthrough in milling and dispersing paints that turned the industry around. It enables the production of paints of superior gloss, transparency and colour development at lower cost.

Instead of the usual screens which get blocked and wear quickly, the DYNO-MILL utilizes a **self-cleaning** patented Dynamic Separator. Made of tough tungsten carbide it consists of a rotor/stator with an adjustable gap to suit beads of any size enabling the DYNO-MILL TO WORK EFFICIENTLY EVEN WITH VISCOUS AND THIXOTROPIC MATERIALS.

Enquiries outside the UK and Eire please contact:

WILLY A BACHOFEN AG,
Utengasse 15/17, CH-4005 Basel,
Switzerland. Tel. 061-33 55 55.
Telex 62564

Cleaning or product changes generally take only 15 minutes for minimal downtime. All of which adds up to more operating time, longer mill life and greater profit.

And if those benefits aren't enough to convince you

LOOK AT THESE:

SAFETY: The DYNO-MILL'S chamber and motor are well separated; a much safer design than some **COMPETITIVE** models with their motor flush-mounted to the milling chamber with its flammable solvents.

VARIABLE SPEED: For better control of particle size and a wider range of products.

INTERNAL CHANGES recently introduced to enable the DYNO-MILL to handle high viscosity millbase at higher throughput and lower temperature.

SUPERB ENGINEERING guarantees trouble-free operation and enables the use of high density media (zirconium oxide and steel) without excessive wear of machine parts.

FIRST-CLASS SERVICE if and when required.

DYNO MILL



For complete DYNO-MILL information call our technical director Ian Kerr.

GLEN CRESTON LIMITED
16 Dalston Gardens, Stanmore,
Middlesex, HA7 1DA, England
Tel. 01-206 0123 Telex 925791