Vol. 65 ~~No. 6~~ JOCCAB 65(6) 211-248 (1982) June 1982

OCCA CONFERENCE
"THE EFFICIENT USE OF
SURFACE COATINGS"

CALL FOR PAPERS

Next year's conference is to be held at

York, 15-18 June 1983

See the front inside cover for further details

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

*****WINNER OF THE 1980 ABC REED INTERNATIONAL AWARD**

Epoxy novolac esters
H. Panda and Rakhshinda

The testing of new products
L. Gall

Spectrophotometric studies on hard and soft lac resins
D. N. Goswami and N. Prasad

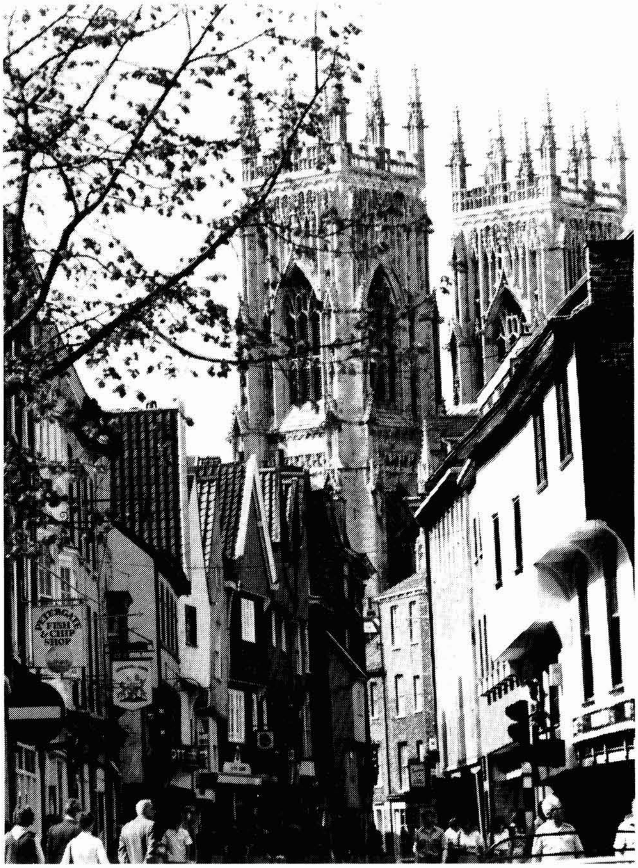
Aqueous gloss enamels
A. Mercurio, K. Kronberger and J. Friel

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

OCCA CONFERENCE 1983

YORK

15-18 JUNE
1983



CALL FOR PAPERS

THE EFFICIENT USE OF SURFACE COATINGS

The next OCCA Biennial Conference will be held at the Viking Hotel, York from 15-18 June 1983 with the theme "The efficient use of surface coatings".

There will be four technical sessions under the headings:

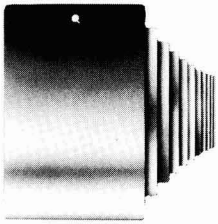
(I) Formulation including manufacturing etc.
(II) The efficient use of coatings application
(III) The use of computers in surface coatings
(IV) Maintenance of quality and prediction of performance

Following the innovation at the 1981 Conference there will be a discourse session included in the fourth technical session.

The Hon. Research & Development Officer now invites offers of papers for presentation at this Conference. Anyone wishing to submit a paper for consideration should notify his intention as soon as possible to: **The Director & Secretary, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF, England (Tel: 01-908 1086; Telex: 922670 OCCA G).**

Reader Enquiry Service No. 199


WORLD'S
No.1
PAINTS



natural and accelerated testing...

Most complete exposure testing service available. 3 locations nationwide—Miami, Phoenix, Chicago—offering Tropical, Desert and Light Industrial Atmosphere tests. Plus accelerated testing with Atlas Weather-Ometer®, Fade-Ometer® and UVCON™

Send for FREE full-color brochure.

South Florida Test Service INC.
A subsidiary of Atlas Electric Devices Co.
9200 N.W. 58 St. • Miami, FL 33178 U.S.A. • 305/592-3170

Reader Enquiry Service No. 192

Paint RESEARCH ASSOCIATION

Guide to Preservatives for Water-Borne Paints

Following the favourable response to the earlier Paint RA special publication, GUIDE TO PAINT FILM FUNGICIDES, published in July 1979, it became apparent that there was a need for a companion guide to the preservation of water-borne paints and paint components against microbial spoilage.

For this special publication, over five hundred potential suppliers of preservatives world-wide were contacted. As a result, comprehensive data, both chemical and toxicological, has been collected and tabulated on 130 commercially available products. The guide also includes a critical review of current literature on water-borne paint spoilage and preservation.

It has not been possible to list preservative activity, even in comparative terms as this, to a great extent, is dependent upon the product in which the preservative is to be used. The aim of the guide is primarily to enable the user to select a short-list of potentially suitable products prior to further testing for compatibility and preservative activity.

It is hoped that this guide will be of use not only to the paint industry and its suppliers and customers, but also to many other industries handling water-based products.

Price: £25
(Paint R.A. Members – £10)

Available from:
Mrs Caroline Veitch
Publications Co-ordinator
Information Department
Paint Research Association
Waldegrave Road
Teddington, Middlesex TW11 8LD

Reader Enquiry Service No. 110

CHLORINATED POLYPROPYLENE
&
CHLORINATED POLYETHYLENE

SUPERCHLON
CPP, CPE

It'll help you in formulating **High build chlorinated rubber-type paints** for Ships, Tanks, Bridges, and other steel works, with outstanding exterior durability, storage stability and airless sprayability.

Try Superchlon for your formulations.

For detailed information, please contact:

Sanyo-Kokusaku Pulp Co.,Ltd.
CHEMICALS SALES DEPT.
1-4-5 Marunouchi, Chiyoda-ku, Tokyo, Japan
Telex: J24279 SKPULP
Cable: SANKOKUPA TOKYO

Reader Enquiry Service No. 112

JOURNAL of the OIL AND COLOUR CHEMISTS' ASSOCIATION

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: D. N. Buddles, BSc

Annual subscription to non-members: £50.00 ($100), post free by surface mail, payable in advance.

Single copies £5.00 ($10), post free by surface mail, payable in advance.

Contents **Vol. 65 No. 6** **June 1982**

Transactions and Communications

Epoxy novolac esters 211
H. Panda and Rakhshinda

The testing of new products 217
L. Gall

Spectrophotometric studies on hard and soft lac resins 223
D. N. Goswami and N. Prasad

Aqueous gloss enamels 227
A. Mercurio, K. Kronberger and J. Friel

Reviews 239

OCCA Meetings 239

News 244

OCCA News 246

New members 248

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

OIL & COLOUR CHEMISTS' ASSOCIATION—PUBLICATIONS

Fourth edition

The Introduction to Paint Technology, of which 20,000 copies have already been sold, forms an excellent introduction to the whole field of surface coatings and related technologies. The fourth edition contains the important addition of a glossary of the terms used in the book together with explanations of their derivations. The text has been thoroughly revised and updated to incorporate recent advances in technology of the surface coatings industries.

Price: £5.00 (US $12.00)

Paint Technology manuals

Works Practice

This publication, which is of great use both to the practical man within the industry and the student entering the industry, is concerned with the practical aspects of making paints. As very little has been published on this subject, a fairly broad coverage is attempted including factory layout and organisation, paint and media manufacturing processes, legal aspects and safety precautions.

Biennial Conference

PREPRINTS

The Association organises an international Conference every two years and preprints of the papers are prepared for delegates. A strictly limited number of the following are available to those who wish to have the complete bound sets of papers.

1981 Bath. *Alternative technologies in coatings.* Eleven papers presented. **Price £5.00. (US $12.00)**

1979 Stratford. *The challenge to coatings in a changing world.* Ten papers presented. **Price £5.00. (US $12.00)**

1977 Eastbourne *The conservation of energy, materials and other resources in the surface coatings industries.*
Fourteen papers presented. **Price £5.00 (US $12.00)**

1973 Eastbourne *Towards 2000.* Sixteen papers presented. **Price £5.00 (US $12.00)**

UV

Ultraviolet polymerisation and the surface coatings industries

The Association has published two volumes on this subject, comprising 23 papers presented at Symposia, forming an authoritative work of reference for this rapidly developing technology. **Special offer: £10.00 (US $24.00) for both volumes purchased together.**

UV2

Monograph No. 1. MARINE FINISHES. Now available **Price £2.00 (US$5.00)**

ORDER FORM

To: **Oil & Colour Chemists' Association,**
Priory House, 967 Harrow Road,
Wembley, Middlesex HA0 2SF, England.

Please send me copies of the books as listed below:

____

____

____

____

____

I enclose remittance of £ (US $)......to cover the cost.

Name: ____________________

Address to which books should be sent:

____

____

____

____

Prices include postage and packing (surface mail).

J. Oil Col. Chem. Assoc., 1982, 65, 211-216

Transactions and Communications

Epoxy novolac esters

By H. Panda and Rakhshinda

Research & Development Section, The Indian Turpentine & Rosin Co. Ltd, PO Clutterbuck Ganj-243 502, Bareilly (UP), India

Summary

Epoxy novolac resins were synthesised by condensing epichlorohydrin with novolacs based on different types of phenols. The novolac resins were prepared in the usual manner. The rosin was treated as an acid and reacted with the epoxy novolac resins at high temperatures thereby forming the epoxy novolac esters.

Methods of preparation of novolac resins, epoxy novolac resins and rosin modified epoxy novolac resins, i.e. epoxy novolac esters, have been given and their properties have been studied. These esters had satisfactory melting points, solubilities and compatibilities with various drying oils. By virtue of their properties, these rosin modified epoxy novolac resins can be used in high quality varnishes and other surface coatings.

Keywords

Raw materials for coatings

binders (resins, etc.)

epoxy resin
phenolic resin

Processes and methods primarily associated with manufacturing or synthesis

esterification

Raw materials for coatings

used in manufacture or synthesis of ingredients for coatings

epichlorohydrin
phenol
rosin

Les esters abiétiques de novolaques époxydées

Résumé

Des résines novolaques epoxydées ont été synthésisées en faisant la condensation d'épichlorhydrin avec les résines novolaques dérivées de divers phénols. Les résines novolaques étaient préparées par la méthode classique, et la colophane en vue de ses caractéristiques acides, était mise à réagir aux températures élevées avec les résines novolaques époxydées afin de produire les esters abiétiques de novolaques époxydées.

On a donné les méthodes de préparation des résines novolaques, des résines novolaques époxydées et des résines novolaques époxydées et modifiées par colophane, c'est-à-dire, des esters de novolaques époxydées, et l'on a étudié leurs caractéristiques. Ces esters possèdent un point de fusion acceptable et de bonnes caractéristiques de solubilité et de compatibilité aux diverses huiles siccatives. En vertu de leures caractéristiques, ces résines novolaques époxydées et modifiées par colophane peuvent être utilsées en vernis clairs de haute qualité et par les industries de peintures.

Epoxidierte Novolakharzester

Zusammenfassung

Epoxidierte Novolakharzester wurden bei der Kondensation von Epichlorhydrin mit Novolaken der verschiedenen Phenole. Die Novolakharze wurden normalerweise hergestellt, und Kolophonium sich als Säure mit den epoxidierten Novolakharzen reagieren liessen um die von Kolophonium modifizierten epoxidierten Novolakharzester herzustellen.

Die Methode zur Herstellung der Novolakharze, epoxidierten Novolakharze und von Kolophonium modifizierten Novolakharze, d.h. die epoxidierten Novolakharzester wurden festgestellt und ihre Eigenschaften untersucht. Diese Ester besitzen einen annehmbaren Schmelzpunkt und gute Löslichkeits- und Verträglichkeitseigenschaften hinsichtlich verschiedener trocknenden öle. Auf Grunde ihrer Eigenschaften können diese von Kolophonium modifizierten epoxidierten Novolakharze in Hochqualitätsklarlacke angewandt und von den Lackindustrien benützt werden.

Introduction and discussion

Refs, 1-9

Epoxide resins[1-4] may be used instead of hexamethylene tetramine (HMTA) to crosslink phenolic resins if the splitting off of volatile compounds must be avoided.

Epoxidised novolacs combine the reactivity of the epoxy group and thermal resistance of the phenolic backbone. They may be obtained as solid grindable resins by the reaction of novolac resins with epichlorohydrin, depending on the molecular weight of novolac resin used (Figure 1).

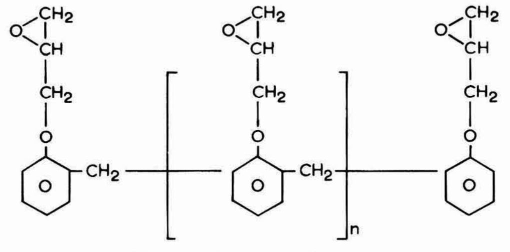


Figure 1. Epoxy novolac resin

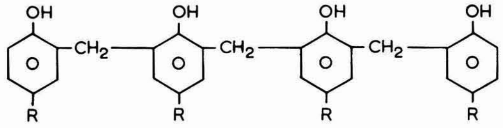


Figure 2. Novolac resin.

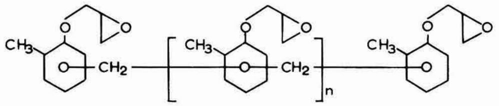


Figure 3. Epoxy cresol novolac resin

Both novolacs and resols may be employed. The products obtained exhibit high strength, high adhesion, excellent dielectric properties and improved oxidation resistance, but show somewhat reduced thermal resistance. Additional crosslinking, which increases thermal stability, may be effected by the addition of small amounts of HMTA.

The ring opening of epoxides is an SN^2 type reaction which is catalysed by inorganic and organic bases, preferably tertiary amines[5]. Similar products may be obtained if the phenolic resins are treated with epichlorohydrin and sodium hydroxide. The chlorohydrin group obtained in the first step is converted to a second epoxide group which then further reacts with a phenolic hydroxyl group.

The novolacs of pure phenol react with epichlorohydrin (see Equation 1, Figure 4) in the same way as other novolacs based on bisphenol A and other substituted phenols.

In this study, epoxy novolac resins were prepared from a range of different phenol derivatives. These epoxy novolac resins were further esterified with rosin to obtain the corresponding esters.

The reaction conditions, properties, etc. of novolac resins, epoxy novolac resins and their corresponding rosin esters are shown in tables 1-3.

The non-reactive[6] novolac resin consists of a molecular chain of varying lengths and may be represented as shown in Figure 2.

Rosin modified phenolic[6] resins are used almost exclusively in the manufacture of oleoresinous varnishes and binders. Different grades of the basic resin type may be made by varying (a) the type of phenol derivative and the ratio of "phenol" to formaldehyde used, (b) the ratio of phenolic resin to rosin used, or (c) the type of alcohol used and the extent of esterification.

A general division into hard and soft types of resin may be made. The former have melting points no higher than 105°C and form a clear solution in white spirits at 20 per cent solids content. The latter have melting points in the region of 130°C, and only give clear solutions in white spirits at concentrations higher than 40 per cent by weight. Varnish making is always simpler with the soft resins.

The proportion of rosin component in these resins is always high, often as much as 80-90 per cent.

Binders based on rosin modified phenolic resins are not distinguished by high exterior durability as they embrittle quite rapidly, a property which is common to all binders based on rosin or its derivatives.

Resorcinol can be used in the preparation of epoxy resins, but these resins are not normally used alone as the

Table 1A

Reaction conditions and properties of novolac resins

Sample No.	Type of phenol derivative	Weight (g)	HCHO 37% (aq/ml)	Catalyst	rt (min.)	Temp. (°C)	mp (cap) (°C)	sp (R&B) (°C)	IR values (cm^{-1})	Compatibility with oils LO	CO	TO	DBL
1.	Phenol	130	95	2[a]	90	100	80	100	3, 6.2, 12.2, 13.3	f	f	f	ex
2.	o-cresol	149	100	2[a]	120	100	60	75	3, 6.2, 3.4, 12.2, 13.3	ex	ex	ex	ex
3.	m-cresol	108	60	2[a]	90	100	138-40	160	3, 3.4, 6.2, 12.2, 13.3	f	f	f	ex
4.	p-cresol	298	200	3.5[a]	120	100	65	80	3, 3.4, 6.2, 11.45, 13.3	ex	ex	ex	ex
5.	p-tert-butyl phenol	207	100	4[a]	90	100	101	120	2.9, 3.4, 4.40, 6.2, 11.4, 13.3	ex	ex	ex	ex
6.	Bisphenol A	315	100	2[a]	90	100	101	122	2.95, 6.2, 11.4, 13.3	f	f	ex	ex
7.	2,4-dimethyl phenol	169	92	2[b]	90	100	45	60	3, 3.4, 2.9, 6.2, 11.40 13.3	ex	ex	ex	ex
8.	2,6-dimethyl phenol	169	92	2[b]	120	100	170	181	3, 2.9, 3.4, 6.2	ex	ex	ex	ex

rt = reaction time, LO = linseed oil, CO = castor oil, TO = tung oil, DBL = double boiled linseed, f = fail, ex = excellent, a = oxalic acid in g, b = hydrochloric acid (36%) in ml

Table 1B
Colour and solubility characteristics of novolac resins

Sample No.	Colour (gardner scale)	Acetone	Turpentine	Petroleum spirit	Benzene	MEK	Toluene	Carbon tetra-chloride	Ethyl acetate	Chloroform	Ethyl alcohol
1.	7	ex	i	i	i	ex	i	i	ex	i	ex
2.	7	ex	i	i	i	ex	i	i	ex	ex	ex
3.	12	ex	i	i	i	ex	i	i	ex	i	ex
4.	7	ex	ex	i	ex	ex	ex	ex	ex	ex	s
5.	6	ex	ex	ex	ex	s	ex	ex	ex	ex	ex
6.	7	ex	ss	i	ss	ex	ss	ss	ex	i	ex
7.	12	ex	ex	i	ex	ex	ex	ex	ex	ex	ex
8.	12	ex	ps	i	ps	ex	ps	ex	ex	ex	ex

MEK = methyl ethyl ketone, ex = excellent, i = insoluble, ps = partially soluble, ss = slightly soluble, s = soluble

resin part of a binder due to their brittleness. The amount used with bisphenol A epoxy resins should not be higher than 5-15 per cent. This is sufficient to considerably increase the reactivity and the resistance against organic acids.

Epoxide resins[7] are essentially long chain polyhydric alcohols which, as such, will react with carboxylic acids to form esters. Reaction takes place between the carboxyl group of the acid and both the epoxy group and the hydroxyl group of the epoxide, as can be seen in Equation 2, Figure 4. This hydroxyl group can then react normally with another carboxyl group with the elimination of one molecule of water. The equivalent weights which are quoted in the trade literature for these resins take this fact into account, and by using the quoted equivalent of the epoxide resin, complete esterification of the epoxy and hydroxyl groups can be achieved. In practice, however, this is seldom the case, usually between 40 and 80 per cent of the available groups being esterified; the degree of esterification depending upon the type of coating which it is desired to produce.

Cresol based epoxy novolac resins[8,9] are multifunctional solid polymers, characterised by low ionic and hydrolysable chloride impurities, high chemical resistance and thermal performance. ECN (epoxy cresol novolac) resins are widely used as base components in high performance electronic and structural compounds, high temperature adhesives, structural moulding powders, castings and laminating systems, tooling applications, powder coatings etc. (Figure 3).

Experimental

Material used

1. Phenol derivatives:

   (a) pure phenol – commercial grade
   (b) o-cresol BU Co. India
   (c) m-cresol BU Co. India
   (d) p-cresol BU Co. India
   (e) p-tert-butyl phenol – AIP India
   (f) bisphenol A– AIP India
   (g) 2,4-dimethyl phenol – Fluka AG, West Germany
   (h) 2,6-dimethyl phenol – Fluka AG, West Germany

2. Formaldehyde – LR/SM

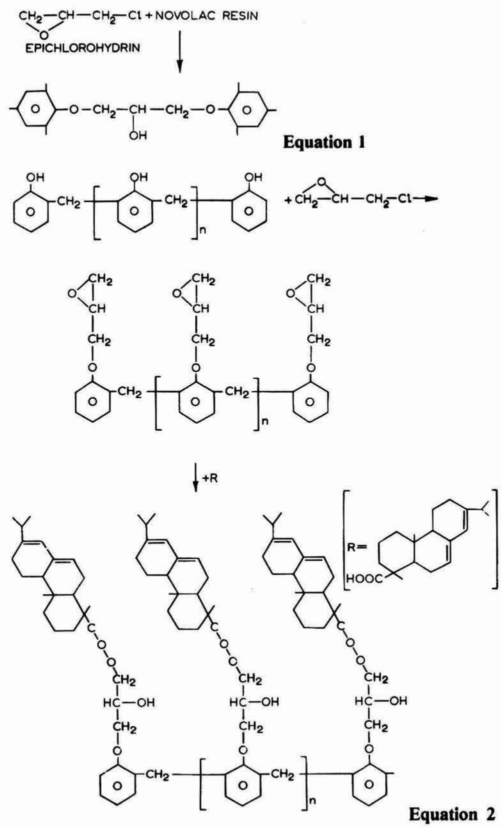


Figure 4.

3. Epichlorohydrin, LR/SM

4. Sodium hydroxide – GR/SM

5. Gum rosin – WG/ITR Co. Ltd

Table 2A

Reaction conditions and compatibilities of epoxy novolac resins

Sample No.	Novolac based on	Novolac resin (g)	Epichlo-rohydrin (ml)	Sodium hydroxide (g)	Time (h)	Temp. (°C)	Compatibility with oils at 250 ± 5°C			
							Tung oil	Castor oil	Linseed oil	DBL
9.	Phenol	40	80	4.0	2.5	110 ± 5	g	g	g	g
10.	o-cresol	35	70	3.5	2.5	110 ± 5	g	ex	p	p
11.	m-cresol	35	105	3.5	3.0	110 ± 5	f	p	f	ex
12.	p-cresol	45	90	4.5	2.5	110 ± 5	ex	ex	ex	ex
13.	p-tert-butyl phenol	90	80	4.0	2.5	110 ± 5	ex	ex	ex	ex
14.	Bisphenol A	45	90	4.5	2.5	110 ± 5	g	g	g	g
15.	2,4-dimethyl phenol	35	70	3.5	3.5	120 ± 5	ex	ex	ex	ex
16.	2,6-dimethyl phenol	35	70	3.5	3.5	120 ± 5	ex	ex	ex	ex

DBL = double boiled linseed oil, ex = excellent, f = fail, g = good, p = poor

Table 2B

Properties of epoxy novolac resins

Sample No.	Colour (gardner scale)	mp (cap) (°C)	sp (R & B) (°C)	IR values (cm^{-1})	State	Solubility in								
						Benzene	Diox-ane	Turp-entine	MEK	CTC	E Al	E Ac	Chloro-form	Pet. spirit
9.	8	60	75	2.95, 3.4, 7.44 9.05, 8.1, 10.8, 9.7, 13.3	solid	i	s	i	ex	i	i	ex	i	i
10.	9-10	55	73	2.95, 3.4, 7.44. 8.1 9.05, 9.7, 10.95, 12.1 13.3	solid	ps	ex	i	ex	i	ex	ex	i	i
11.	10-11	125	140	2.95, 3.4, 7.44, 8.1 9.05, 9.7, 10.95, 12.1, 13.3	solid	i	ps	s	ex	i	ps	ex	s	i
12.	7	65	85	2.95, 3.4, 7.44, 8.1 9.05, 9.7, 10.95, 11.5, 12.1, 13.2	solid	ex	ex	i	ex	i	ex	ex	ex	i
13.	7	57	75	2.95, 3.4, 7.44, 8.1 9.05, 9.5, 10.95, 12.1, 13.2	solid	ex	ex	ex	ex	ex	ex	ex	ex	i
14.	6	53	60	2.95, 3.4, 7.44, 8.1 9.05, 9.5, 10.95, 12.1 13.2, 17.5	solid	i	s	i	ex	i	ss	ss	i	i
15.	12	60	80	2.95, 3.4, 7.44, 8.1 9.05, 9.5, 10.95, 12.1, 13.2	solid	ex	ex	ex	ex	ex	ex	ex	ex	i
16.	12	170	185	2.95, 3.4, 7.44, 8.1, 9.05, 9.5, 10.95, 12.1	solid	ex	ex	ex	ex	ex	ex	ex		i

MEK = methyl ethyl ketone, CTC = carbon tetrachloride, E Al = ethyl alcohol, E Ac = ethyl acetate, ex = excellent, i = insoluble, ps = partially soluble, ss = slightly soluble, s = soluble

Procedure

1. Preparation of novolac resins

Novolac samples of different functionalities were prepared firstly by mixing various quantities of phenol or its derivatives with water in a three necked flask. The pH was adjusted to 0.5-2 with oxalic acid or with hydrochloric acid and the contents heated to 80°C with constant stirring. Various quantities of formaldehyde were added over a period of half an hour after which water was removed under vacuum. Various novolac resins produced by this method were used in the preparation of corresponding epoxy novolac resins. Experimental data are shown in Table 1A and B.

2. Preparation of epoxy novolac resins

Novolac samples prepared as above were reacted with epichlorohydrin at 102-105°C. Quantities of 10 per cent sodium hydroxide solution were added gradually to the reactants over periods ranging from 2.5 to 4.5 hours. The contents were then washed with hot water to completely remove the salt formed. The water was removed under vacuum t 80°C (Table 2A and B).

3. Preparation of rosin modified epoxy novolac resins (epoxy novolac esters)

The modified resins were prepared by placing the high molecular weight epoxy novolac resins and colophony

Table 3A

Reaction conditions and properties of rosin modified epoxy novolac resins (epoxy novolac esters)

Sample No.	ENR based on	Rosin (g)	ENR (g)	Time (h)	Temp. (°C)	mp (cap) (°C)	sp (R & B) (°C)	av mg KOH/ g	Colour (gardner scale)	IR values (cm⁻¹)
17.	Phenol	100	10	3	270 ± 5	100	119	68	10	2.95, 3.4, 5.8, 6.62, 7.44, 8.1, 9.05, 9.7, 12.1, 13.5
18.	o-cresol	100	10	3	270 ± 5	75	90	55	9-10	2.95, 3.4, 5.8, 6.62, 7.44, 8.1, 9.05, 9.7, 12.1, 13.5
19.	m-cresol	100	10	3	270 ± 5	85	100	130	12	2.95, 3.4, 5.8, 6.62, 7.44, 8.1, 9.05, 9.7, 12.1, 13.3
20.	p-cresol	100	10	3	270 ± 5	80	95	60	18	2.95, 3.4, 5.8, 6.62, 7.44, 8.1, 9.05, 9.7, 11.5, 12.1, 13.2
21.	p-tert-butyl phenol	100	10	3	270 ± 5	75	92	112	9	2.95, 3.4, 5.8, 6.6, 7.44, 8.1, 9.05, 9.5, 12.1, 13.2
22.	Bisphenol A	100	10	3	270 ± 5	85	105	93	12	2.95, 3.4, 5.8, 6.62, 7.44, 8.1, 9.05, 12.1, 13.2, 17.5
23.	2,4-dimethyl phenol	100	10	3	270 ± 5	65	85	20	12	2.95, 3.4, 5.8, 6.62, 7.44, 8.1, 9.05, 9.5, 12.1, 13.2
24.	2,6-dimethyl phenol	100	10	3	270 ± 5	65	80	112	12	2.95, 3.4, 5.8, 6.62, 7.44, 8.1, 9.05, 9.5

ENR = epoxy novolac resin, mp (cap) = melting point by capillary method, sp (R & B) = softening point by ring and ball method, av = acid value

Table 3B

Compatibility and solubility of epoxy novolac esters

Sample No.	Compatibility with				Solubility in								
	TO	LO	CO	DBL	Dioxane	MTO	E A1	MEK	Benzene	Turp-entine	CTC	Chloroform	Ethyl acetate
17.	ex	ex	ex	ex	ex	ps	s	ex	s	ps	s	s	ex
18.	ex	ex	ex	ex	ex	ps	s	ex	ex	s	ex		ex
19.	ex	ex	ex	ex	ex	ss	ex	ex	ex	s	ex	ex	ex
20.	ex	ex	ex	ex	ex	ps	s	ex	ex	ex	ex	ex	ex
21.	ex	ex	ex	ex	ex	ex	ex	ex	ex	ex	ex	ex	ex
22.	ex	ex	ex	ex	ex	ps	ps	ex	ex	ex	ex	ex	ex
23.	p	p	ex	ex	ex	ex	s	ex	ex	ex	ex	ex	ex
24.	ex	ex	ex	ex	ex	ex	s	ex	ex	ex	ex	ex	ex

TO = tung oil, LO = linseed oil, CO = castor oil, DBL = double boiled linseed oil, MEK = methyl ethyl ketone, E A1 = ethyl alcohol, CTC = carbon tetrachloride, MTO = mineral turpentine oil, ex = excellent, ps = partially soluble, s = soluble, ss = slightly soluble, p = poor

(rosin) in a 250 ml four necked reaction flask equipped with an efficient stirrer, thermometer, nitrogen inlet and siphoning device to withdraw samples during the esterification. The mixture was heated gradually to 260-270°C and maintained until low acid value products were obtained. The course of the esterification was followed by determining the acid value of samples removed periodically from the reaction mixture. For properties see Table 3A and B.

Conclusion

Refs, 10,11

Epoxy novolac esters (ENE) show better properties than either epoxy esters of rosin[10], or epoxy esters of styrenated rosin[11].

These esters have satisfactory softening points and better solubilities and compatibilities with different drying oils. ENE resins can be used for high quality varnishes, wax modifications to improve clarity, strength and heat sealability, in cable oils as co-constituents with dielectric oils to control the viscosity and to improve impregnating properties, as corrosion inhibitors against hydrochloric and other acids. Above all, the preparation of these ENE resins is simple and the products are economically competitive. They should, therefore, prove a useful addition to the present range of solid resins.

Acknowledgements

This communication is a contribution by the Research & Development Section of ITR Co. Ltd and is published with the permission of the managing director to whom the authors express their gratitude. The authors are also indebted to Dr A. Knop, GmbH (Germany), Dr I. H. Updegraff, Conn. (USA), Dr H. A. Newey, Calif. (USA), Dr J. Longreen, Stockholm (Sweden), Dr Y. Tanaka, (Japan), C. A. May, Lockheed Missiles & Space Company, Calif. (USA) for their valuable discussions, criticisms and suggestions for the improvement of this paper.

[Received 5 October 1981

References

1. Knop, A. and Scheib, W., "Chemistry and application of phenolic resins", *Springer – Verlag*, Berlin, Heidelberg, New York, 1979, p. 55.
2. Lee, H. and Neville, K., "Handbook of Epoxy Resins", *Mcgraw-Hill*, London, 1967.
3. Potter, W. G., "Epoxide Resins", *Illiffe Books*, London, 1970.
4. Singh, G. P., Kapoor, B. M., Saksena, S. C., Uddin, A., Panda, H. and Rakhshinda, "Epoxy Resins – A review", *J. Col. Soc.*, 1981, **20**(1), 21.
5. Ishii, Y. and Sakai, S., 1,2-Epoxides in Frish, K. C., Reegen, S. L., – "Ring Opening Polymerisation", vol. 2, *Marcel – Dekker Inc*, New York, 1967.
6. Nylen, P. and Sunderland, E., "Modern Surface Coatings", *John Wiley and Sons Ltd*, New York, 1965, pp. 184-185.
7. Chattfield, H. W., *"Paint & Varnish Manufacture"*, 1956, p. 86.
8. Kirk, Othmer, "Encyclopedia of Chemical Technology", 3rd ed., *John Wiley & Sons*, New York, 1980, vol. 9, p. 267.
9. US pat 2, 456, 408, 1948, 14 Dec. S. O. Greenlee.
10. Hingorani, B. H., Uddin, A., Panda, H., Saksena, S. C. and Rakhshinda, *JOCCA*, 1980, **63**, 407.
11. Ahisanuddin, Saksena, S. C., Panda, H. and Rakhshinda, Epoxy Esters of Styrenated Rosin, *Paint India*, 1981, **XXXI** (8), 3.


SURVIVAL KIT.
THE CORROSION FILE.
crown
Protective Coatings

OIL & COLOUR CHEMISTS' ASSOCIATION

AUSTRALIA

publishes monthly

PROCEEDINGS AND NEWS

Advertising and subscription rates obtainable from:

Geoff Goullet
Advertising Manager
Suite 18, 65 Queens Road,
Melbourne, Australia 3004

Annual subscription rate $A30.00

Editorial contributions welcomed

L. A. HILL
Honorary Editor
13 Melby Avenue, Balaclava,
Victoria, Australia 3183

Reader Enquiry Service No. 129

OIL & COLOUR CHEMISTS'

ASSOCIATION

Marine Finishes

Reprints are now available of the OCCA Monograph number 1 on Marine Finishes by Dr T. A. Banfield from the Association's offices. These bound copies reprinted from the February and March issues of the *Journal* cost £2 including post and packaging.

J. Oil Col. Chem. Assoc., 1982, 65, 217-222

The testing of new products

By L. Gall

BASF Aktiengesellschaft, Ludwigshafen, West Germany

Keywords

Processes and methods primarily associated with

analysis, measurement or testing

test procedure

manufacturing or synthesis

shading

service or utility

weathering

Properties, characteristics and conditions primarily associated with

materials in general

opacity

dried cured films

fade resistance
gloss

Miscellaneous terms

mill base

Introduction

Enormous efforts are made by chemical researchers to develop new pigments, that is, pigments with assets that would recommend them to any paint or ink manufacturer. Yet the low ratio of hits to misses suggests that the application technologist is trying to test the development chemist's product out of existence.

It is not proposed to discuss the coarse screening tests that simply determine whether a pigment is of colouristic interest and satisfies basic demands of fastness etc. The main purpose is to present a few examples of how, with modern systematic methods, it is possible to determine which pigments stand a chance of being accepted by the paint technician.

To simplify a procedure which is often quite complicated, a flow chart is shown (Figure 1) in which the various sections to be discussed are detailed. The route begins at exactly the point where every pigment must, that is, in dispersion, and ends, often literally, on the exposure racks. In section number 1, for determining the dispersibility and flow properties of a mill base, the term "binder demand" is examined. In section number 2, "binder demand" is expressed in terms of the dry coating; the gloss being at a minimum specified level but the pigment concentration a maximum.

Section number 3 concerns the optical properties of the pigment. The transparency and hiding power of the dispersed pigment either at a defined concentration or at the maximum concentration. The aim is to find the maximum hiding power obtainable with a pigment.

Section 4 is the determination of colour strength, that is, the quantitative evaluation of light absorption. For this purpose, specimen coatings are prepared in various depths of shade. It is necessary to know the reduction ratio, RR, at the standard depth of shade that is laid down. The colour strength is thus obtained automatically, even as a function of the depth of shade.

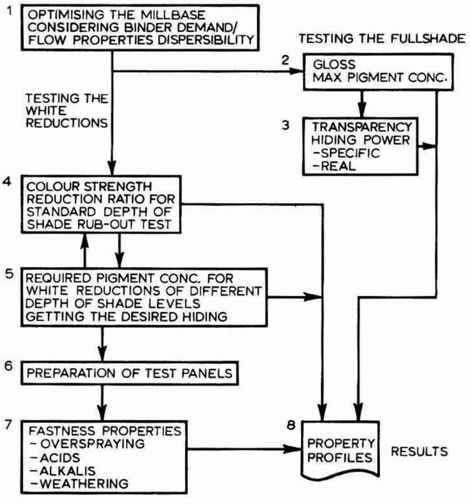


Figure 1.

From section number 5 an arrow also points backwards to section number 4. The reason for this is that the colour strength clearly depends upon the total pigment concentration, but this has to be adjusted in the light of the hiding power of the pigment blend. The concentration of pigment in a paint should not be any higher than that required to give good contrast hiding at normal thicknesses.

After having followed this complicated route, section

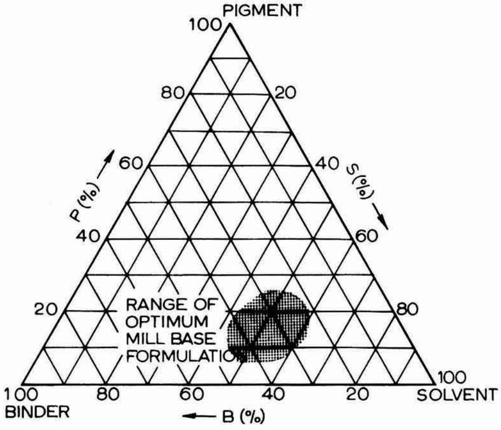


Figure 2.

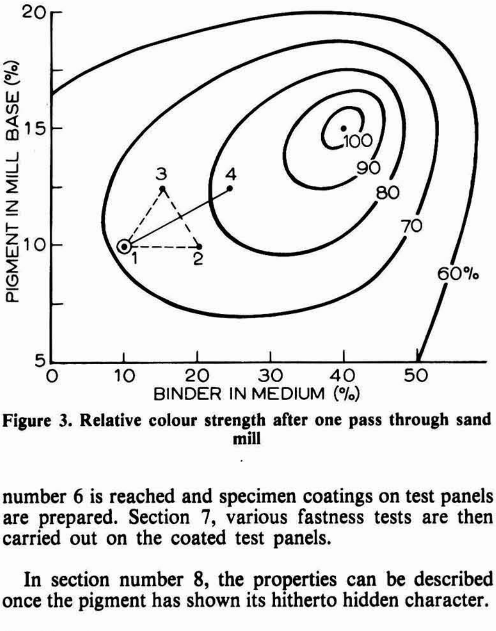


Figure 3. Relative colour strength after one pass through sand mill

number 6 is reached and specimen coatings on test panels are prepared. Section 7, various fastness tests are then carried out on the coated test panels.

In section number 8, the properties can be described once the pigment has shown its hitherto hidden character.

Section 1

There is no universal medium in which all pigments may be dispersed. Thus, the medium in which testing is carried out is more or less laid down by the application for which the paint or ink is intended. The first problem is to find the optimum composition of the mill base. An aid is, of course, the PBS triangle. The aim is not merely to find the area in the triangle in which pigment can be ground, but to find that area in which it can be ground particularly economically and with optimum results in terms of yield.

A series of tests has to be carried out in which the optimum formulation is approached step by step. A recommended strategy is the method of steepest ascent.

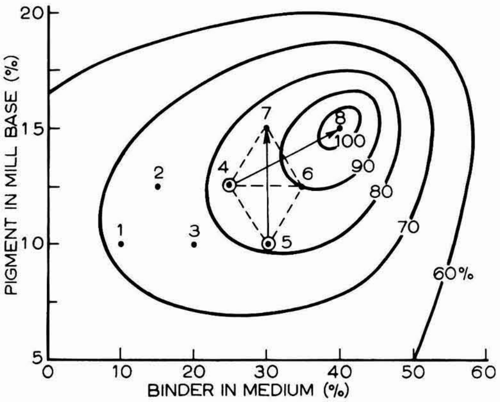


Figure 4. Relative colour strength after one pass through sand mill

An example of the Simplex version of this is briefly described:

The optimum for a new organic pigment is presumed to lie in the area of P = 10 to 20 per cent and B = 20 to 45 per cent, where P = pigment concentration in the mill base and where B = the binder concentration in the medium. The solvent concentration is simply 100 – (B per cent). In this way, for the purposes of discussion in this section, this variable can be eliminated.

If P were varied in increments of 2.5 per cent and B in increments of 5 per cent, 50 mill bases would need to be prepared to cover all possibilities. This would be both tedious and expensive so in the Simplex method three mill bases are prepared from the zone most likely to achieve optimum dispersion.

As can be seen in Figure 3, the best results will be obtained at the point P = 15 per cent and B = 40 per cent. Of the first three tests, No. 1 gave the poorest results. By projecting diametrically opposite No. 1 on the diagram, experiment No. 4 is planned. The results of experiments 2, 3 and 4 are then compared and in this case No. 2 happens to be the worst and, by again seeking the diametrically opposed position, the parameters for the 5th experiment are found.

Thus, after only eight steps in this case, the optimum milling concentration has been determined. Naturally another two or three tests will confirm that the target has been reached. Obviously, other criteria can be adopted for observation other than colour strength after a fixed dispersion time, for instance, throughput.

With the aid of a computer, the Simplex method may be applied to three dimensional problems; in this case the triangle is replaced by a tetrahedron. Again, the next experiment in each case is carried out with the parameters at the point diametrically opposite in space to the corner of the tetrahedron which gave the poorest result.

A statistically designed experiment is recommended if more than one parameter is involved; for instance, if as well as P and B, different solvents or dispersing times have to be optimised. The latter can be critical if it is thought that a long dispersing time might give adverse results.

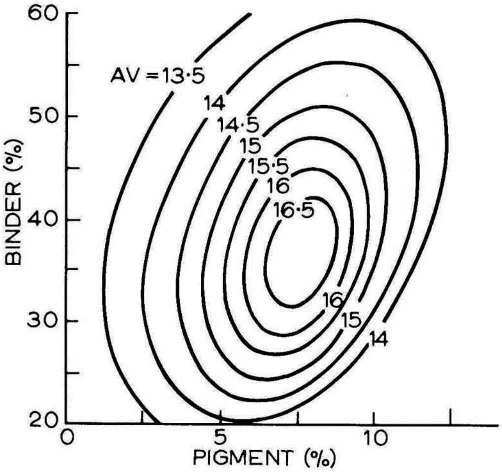


Figure 5. Paliogen Yellow L 1870 dispersed for 30 minutes, colour strength dependence on mill base composition shown

Assuming the dispersing experiment is to be carried out as follows:

P	=	10%	15%	20%
B	=	20%	30%	40%
T (dispersing time)	=	20 mins	50 mins	80 mins

Without the aid of a computer this would require a total of 27 mill bases to be run. The computer, however, is directed to run only 12 experiments with the parameter settings designed to eliminate any false interactions between the parameters, and to ensure that as much information as possible is obtained from the few experiments.

Once all the experiments have been carried out, the results are fed back into the computer, which calculates the coefficient K_i in a model equation e.g.

$$\text{Colour strength} = k_0 + K_1 P t + k_2 B t - k_3 P^2 - k_4 B^2 - k_5 t^2$$

This equation states that good results will be obtained by dispersing at high, but not too high, pigment and binder concentrations and not for excessively long periods of time.

The equation gives a contoured diagram such as for a flavanthrone, with colour strength a function of binder and pigment concentrations (the mill base was dispersed for 30 minutes in a Red Devil). The optimum level for B (binder) is just below 40 per cent and that for P (pigment) about 7.5 per cent. Systematic experiments of this nature are very important; initially, dispersion instability was discovered with the flavanthrone. The results obtained with such sophisticated mill base testing gave an opportunity to stabilise the pigment.

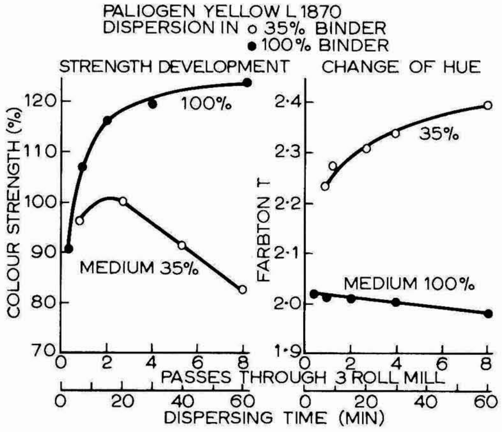


Figure 6.

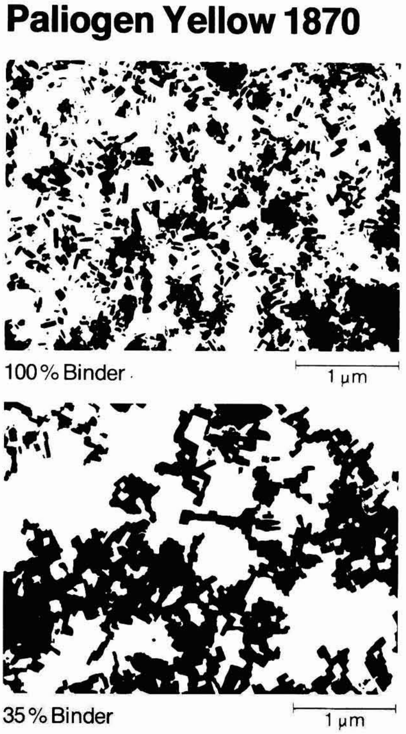


Figure 7.

Figure 6 reveals the dispersion instability that has since been overcome by certain pigment finishing techniques. As can be seen, a great difference in strength occurred when dispersing in a 100 per cent solid media as opposed to the 35 per cent media. Also, strength decreased with time of dispersion in the 35 per cent media and was accompanied by a shift in colour towards red. On the other hand, the 100 per cent medium remained stable or at most became a trace greener.

This phenomenon can be better observed by electron microscopy. Figure 7 shows pigment re-crystallised in parallepipedes which formed super aggregates. Similar

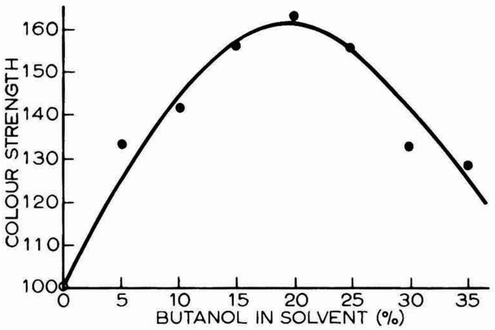


Figure 8. Paliofast Red L 4120 after 30 minutes dispersion in a Red Devil, relative colour strength dependence on butanol content of solvent shown

optimisation experiments have led to other interesting findings, such as that the dispersion of a perylene pigment, whilst not considered a problem, was found to improve when butanol was used in the mill base formulation.

As can be seen in Figure 8, a considerable improvement in the dispersion of perylene pigment can be achieved if butanol is present in small proportions. Obviously this result is not of general validity, but there is probably an optimum xylene/butanol ratio for every pigment, dependent on the properties of the pigment surface.

It is known that phthalocyanine pigments can be dispersed much more readily in xylene than butanol, thus the question arises: are other pigments being dispersed to the optimum degree and with the right solvents?

Section 2

The PC maximum is an important property of a pigment. It is determined by letting down an optimum mill base with resins in stages until a minimum gloss level of 80 per cent is reached.

The PC max is defined as the pigment concentration above which the gloss drops below 80 per cent when viewed at an angle of 20°. At the various stages of letdown, the paint is applied on to a glass sheet and the gloss level measured.

Figure 9 shows a typical plot of gloss against pigment concentration for a typical perylene pigment.

Section 3

The terms transparency and hiding power can be explained as follows:

If a coloured coating is applied to a black and white contrasting substrate, two colour differences can be measured: that due to scattering, Δ ES, and that due to contrast, Δ EC.

Δ ES, the value due to scattering, is the difference between the colour value of the black substrate and that of the coating over the black substrate. It is a measure of the light scattered by the pigment and is one of the components of hiding power.

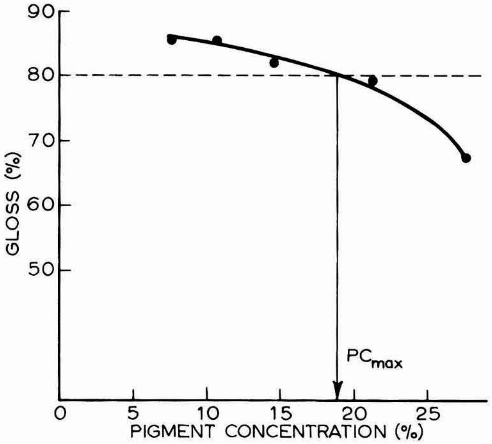


Figure 9. Gloss (20°) of Paliofast Red L 3910 HD, dependence on pigment concentration shown

The relative transparency of a pigment is the colour difference value for a given standard Δ ES (standard) expressed as a percentage of the colour difference value for the specimen Δ ES (specimen). The value for the contrast is the value of the colour difference, Δ EC, between the coating over black and the coating over white; it is a better measure of the hiding power.

Hiding power is nothing more than the reciprocal of the coating thickness for a given contrast value, e.g. ΔE = 1, it can be determined empirically by the method described here or calculated from the spectrum of reflected light. Expressed in terms of the pigment content of the coating, it is referred to as the specific hiding power.

The comparison of hiding power of some typical organic pigments with some colouristically similar inorganic pigments is of importance. The specific hiding power, given in m^2/kg, shows that the light absorption of organic pigments is superior to that of inorganic pigments.

Therefore, if pigments of the same hue are compared, the specific hiding power of the organic pigment is higher. This is largely of value only in the evaluation of the optical performance of a pigment, it is not sufficient to determine whether a certain pigment will allow formulation of a certain paint with a specified obliteration capacity at a given spreading rate. The specific hiding power of an organic pigment is limited in the extent to which it can be used due to the low density and poor finish that would result if the maximum hiding power was attempted.

The realisable hiding power of organic pigments is the value at PC maximum, that is, the hiding power that is achieved when the gloss level of the finish is at 80 per cent. Under these circumstances, the values for inorganic pigments are higher because they have lower specific volumes.

All these remarks have been applied specifically to coatings containing single pigments, but they are of general validity, and also apply to pigment blends e.g. white reductions. These are discussed in sections 4 and 5.

Sections 4 and 5

Here again the aim is simple but the procedure is quite complicated. It is desired to prepare sample panels in standard depths of shade for exposure outdoors and other general testing, the steps are briefly as follows:

Step 1 – the first step is to determine the ratios of coloured pigment to titanium dioxide which achieve the various depths of shade. An amount of optimally dispersed mill base resulting from dispersion tests is taken and mixed with a similarly prepared white paint to achieve an approximate 1/9th standard depth of shade. In step 2 the coating is dried and a reflection curve is plotted. The reduction ratios for 1/1, 1/3, 1/25 and 1/200th standard depths of shade are then calculated with the aid of the FIAF* computer program.

In step 3, the corresponding paints are made up and the pigment concentration is adjusted if the resultant opacity is too high.

Step 4, the paints are sprayed on to Morest charts until the substrate is just obliterated.

In step 5, by a simultaneous application on to a sheet of glass which is weighed before and after coating, the weight of the dry film can be obtained.

The 6th step is to measure the gloss at a 20° angle. If a gloss of 80 per cent is not obtained it will be necessary to return through step 7 to step 3 where, if necessary, new paints will be prepared.

If the gloss is satisfactory step 8 follows, with the measurement of reflection curves to find the depth of shade (B) and the reduction ratio (AV) (Aufhellungsverhaeltnis).

In step 9, the weight of coating necessary to achieve a certain degree of opacity is measured. If it is too low, that is less than 50 g/m^2, the paint is let down with more vehicle than in step 10. If the film weight is too great, higher pigment concentrations are required and it is necessary to descend through step 11 back through to step 3 for new paints to be made up.

In step 12, the depths of shade are re-examined to ensure that they finally lie within the given tolerances, and if not new paints must again be prepared.

In step 13, a point is reached from which specimen coatings can be produced since the reduction ratio in the pigment concentration required to achieve the optimum hiding power at a gloss level of at least 80 per cent is now known. This complicated procedure gives a good idea of what is expected from a paint technologist formulating top coats day in and day out.

The determination of these optimums of gloss and hiding power at standard depths of shade has not always been done in the past in the preparation of exposure work, and this can seriously detract from the value of exposure information since the laboratory work involved is quite substantial. One of the reasons for this technical approach is that the optical performance of pigments, that is their scattering and absorption, is greatly dependent upon the pigment volume concentration. For example, the reducing power of titanium dioxide remains independent of pigment concentration only at pigment volume concentrations (PVCs) of about 8 per cent or less. At higher PVCs, the optical performance progressively decreases as a result of mutual optical hindrance of the particles. In other words, although the reduction ratio of titanium dioxide and a coloured pigment may be retained constant throughout, higher depths of shade will be obtained with higher total pigment concentrations than with lower concentrations.

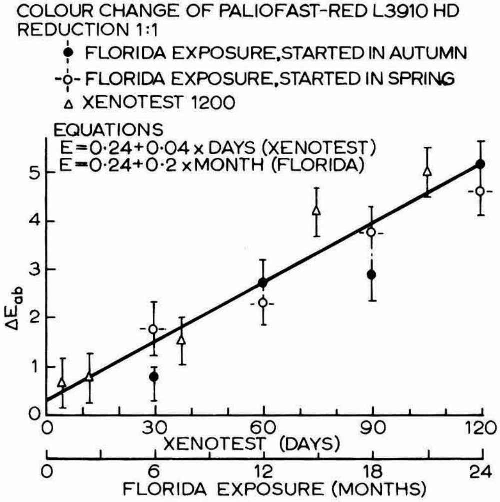


Figure 10.

If the paint is let down, the proportion of light scattered by the titanium dioxide particles increases, with the result that the depth of shade of the coloured blend decreases. Thus, if the PVC is varied for optimising the hiding power, a change in the white to coloured pigment ratio will be required, hence the need for a high degree of precision in this section.

Section 6

This involves the preparation of test panels applied to bonderised steel which has been properly filled and primed.

Section 7

The 7th stage is to determine the fastness to overspraying after stoving at 120, 150 and 180°C, and to determine the acid and alkali resistances.

At least two panels are normally sent to Florida for exposure, one is returned after one year and the other after two years. It should be mentioned that the exposure site in Townsville, Australia is gaining acceptance and BASF has recently conducted several exposure trials at the Allunga site also in Australia.

Figure 10 shows that 18 months exposure is unsatisfactory since it includes either two winters and one

---

*FIAF is a BASF computer program for colour strength comparisons which works by repetitive assimilations of the depth of shade.

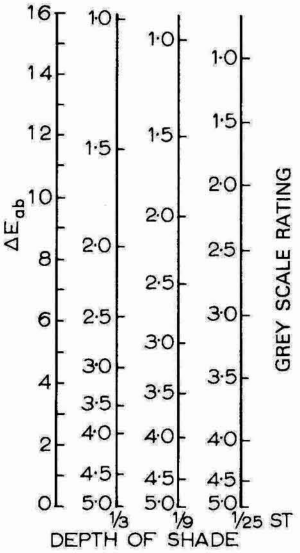


Figure 11. Nomograph for evaluation of grey scale ratings from ΔE values

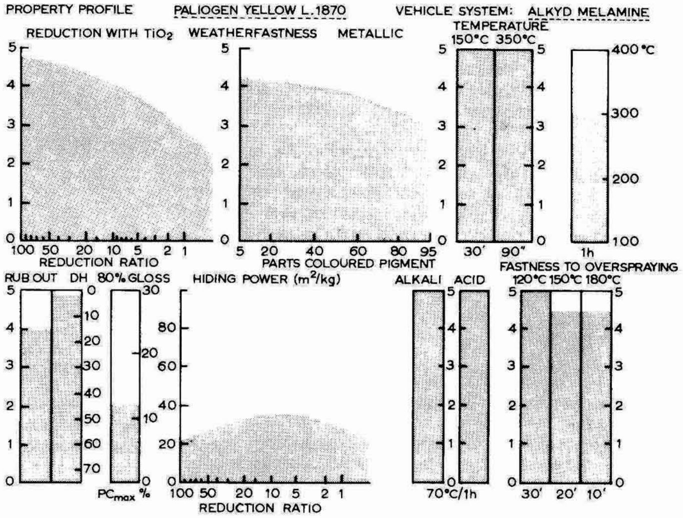


Figure 12.

summer or two summers and one winter. These differences do not exist in a one or two year exposure.

In figure 10, the changes in shade as a function of duration and type of exposure are given for a perylene red. The dark circles represent exposure started in autumn. The light circles represent exposure started in spring. Obviously, the greater change was observed in the panels that were exposed in summer, and it can be seen how they crossed each other during the full 24 months. Also, note the big difference after the 18 month period.

It is also important to know that the relationship between the colour change value Δ E and the energy of radiation is practically linear for pigments that tend to fade. There is also an almost linear relationship between accelerated weathering and Florida exposure within the limits of the data. It would be most desirable if these could be correlated, and intensive experiments are being carried out to determine whether the relationships have any general validity. If successful the advantages are obvious.

Section 8

The 8th and final section concerns the representation of results. It is normal practice to evaluate changes of shade visually on the grey scale for wool. Owing to the subjective nature of this visual assessment, the Δ E values are determined colorimetrically and, with the aid of a nomogram, the ratings are determined.

Allowances are made in the nomogram for the Δ E/grey scale rating relationships for depth of shade, and reproducible results have been obtained with this system.

Another more visual method of expressing results of pigment testing has been developed. It is referred to as a properties profile. It enables the general performance of a pigment to be seen at a glance – Figure 12 shows the profile for a flavanthrone pigment. Beginning from the top left is the weather fastness in terms of the grey scale for both reductions with white and in a mixture with aluminium pigment, the scales along the bottom showing a reduction ratio and the mixing properties respectively.

Next on the right is the heat resistance, showing the satisfactory performance of the pigment at 150°C and 350°C for 30 seconds and 90 seconds respectively, and in the top right it can be seen that the pigment in a suitable vehicle could withstand 350°C for one hour without change.

Ratings for the various acids, alkali and overspray tests are also shown in Figure 12. Of particular interest is the graph in the bottom centre, it displays a relationship between the reduction ratio and the hiding power and, as can be seen, the optimum for this pigment is around 10 parts of titanium dioxide to one of the flavanthrone. At this reduction ratio, the optimum coverage of around 25 m²/kg is attained. To the left it can be seen that the PC max for this pigment is around 12 per cent, but the gloss does not fall below the required 80 per cent.

Next on the left is a scale representing the ease of dispersion. In this case the dispersibility of the product was excellent.

A point is arrived at where the development pigment has been examined and it either has or has not reached expectations. Whatever the case, it is certain that a tremendous amount of work has been done in trying to gauge the potential usage of the newly developed pigment. It is the nature of the surface coatings industry that for each and every application there will be one pigment more suitable than all the rest. It is therefore the technician's aim to seek the perfect pigment even though the perfect pigment may not exist.

[Received 6 October 1981

J. Oil Col. Chem. Assoc., 1982, 65, 223-226

Spectrophotometric studies on hard and soft lac resins

By D. N. Goswami and N. Prasad

Division of Chemistry, Indian Lac Research Institute, Namkum, Ranchi 834010, Bihar, India

Summary

The absorption spectra of hard and soft lac resins have been studied in the complete UV and visible regions. For hard lac, absorption maxima were obtained at 225, 255, 260, around 290 and at 425 nm. For soft lac, absorption maxima were noted at 435, 225 and around 290 nm. The optical density and $E|^{\%}_{\mathrm{cm}}$ values at 425 nm and also the absorbance at 225 nm of a 70:30 (by weight) hard/soft lac mixture were found to be almost identical to those of the parent shellac. It is concluded that the spatial distribution of the different polar groups in the mixture is not identical to that in the parent shellac.

Keywords

Types and classes of coatings and allied products

shellac

Raw materials for coatings

binders (resins, etc.)

lac resin

Processes and methods primarily associated with analysis, measurement or testing

absorption spectroscopy

Des études spéctrophotométriques sur les résines dures ou tendres de gomme-laque

Résumé

On a étudié les spectres d'absorption des résines dures ou tendres de gomme-laque à travers la gamme entière de la lumière et visible et ultra violette. Dans le cas de la gomme-laque dure, on a obtenu les valeurs maximales d'absorption à 225, 255, 260 et à 290 et 425 nm. à peu près. Pour la gomme-laque tendre les valeurs maximales ont ete obtenues à 225, 435 et à 290 nm. à peu près. On a trouvé que la densité optique et les valeurs $E|^{\%}_{\mathrm{cm}}$ ainsi que l'absorbance à 225 nm d'un mélange par poids de 70:30% de dure et de tendre gomme-laque respectivement étaient en grande partie identiques à celles de la gomme-laque mère. On conclut que la répartition spatiale des différents groupements polaires dans le mélange n'est psa identique à celle de la gomme-laque mère.

Spektrophotometrische Versuche mit Hart- und Weichschellackharzen

Zusammenfassung

Die Absorptionsspektra der Hart- und Weichschellackharze wurden über die gesammten Bereiche der sichtbaren und ultravioletten Spektra bestimmt. Im Falle des Hartschellacks wurden Absorptionsmaxima zu 225, 255, 260, und zu etwa 290 und 425 nm. erhalten. Für Weichschellack, wurden Absorptionsmaxima zu 225, 435 und zu etwa 290nm. erhalten. Die optische Dichte und die $E|^{\%}_{\mathrm{cm}}$ Werte zu 425 nm. und sowie die Absorptionsfähigkeit eines 70:30 gewichtsprozentuellen Gemisches von Hart- und Weichschellacken zeigten sich fast identisch zu denen des Stammschellacks. Man schloss dass die räumliche Verteilung verschiedener Polargruppen in dem Gemisch nicht identisch zu derjeniger in dem Stammschellack sei.

Introduction

Refs, 1-3

The absorption spectra of shellac in the complete UV and visible regions have been reported recently[1]. The absorption maxima obtained in the UV and visible regions were interpreted as arising due to the terpenic constituents and erythrolaccin (the colouring materials present) respectively. A relationship between optical density (at 425 nm) and colour index has also been formulated.

It is known that hard lac, soft lac and lac wax are the three major constituents of the natural resin shellac. Hard lac is the main fraction, insoluble in diethyl ether it comprises about 70 per cent (by weight) of shellac. Soft lac, soluble in ether, contributes about 30 per cent (by weight), while lac wax does not comprise more than 5 per cent of the whole lac[2]. Recent studies on the dielectric behaviour of these constituents have revealed that the dielectric relaxation processes of the hard and soft lacs are different[3]. It is of interest, therefore, to investigate the absorption spectra of these constituents for a better understanding of the absorption spectra of shellac. The present paper reports the results of a study on the absorption spectra of hard and soft lacs in ethyl alcohol over the complete UV and visible regions. The absorption spectra of different ratios of mixtures of hard and soft lacs have also been studied to

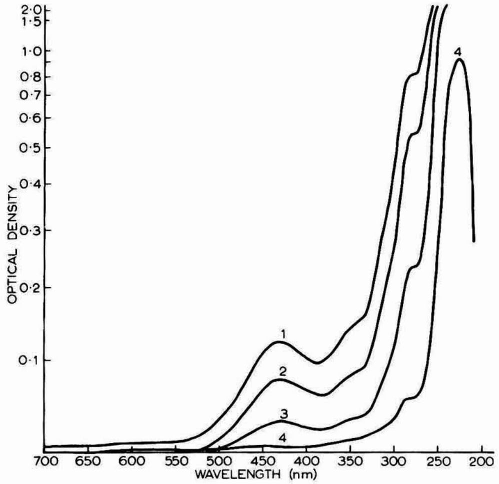


Figure 1. Absorption spectra of soft lac resin, (1) 1.5×10^{-3}, (2) 10^{-3}, (3) 4.5×10^{-4}, (4) 10^{-4} g/ml

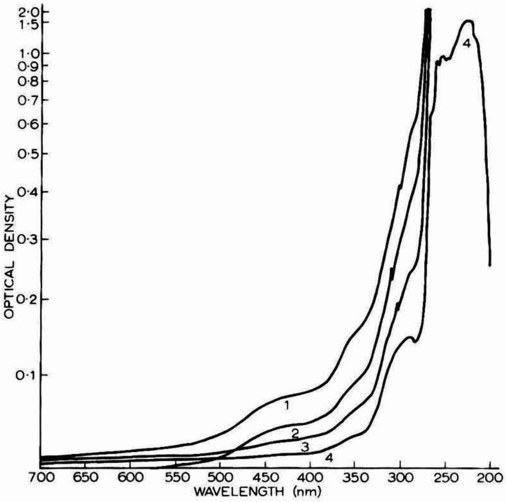


Figure 2. Absorption spectra of hard lac resin, (1) 1.5×10^{-3}, (2) 10^{-3}, (3) 4.5×10^{-4}, (4) 10^{-4} g/ml

confirm whether the spectra of shellac could be represented by a mixture of its constituents.

Materials and methods

Refs, 4, 5

Pure hard and soft lac resins were extracted from a sample of dewaxed shellac following the methods described elsewhere[4,5]. A Beckman DB-GT spectrophotometer together with a Beckman 1005 ten inch recorder were used for obtaining the absorption spectra. The absorption spectra of hard lac, soft lac and mixtures of varying ratios of the two in ethyl alcohol were obtained.

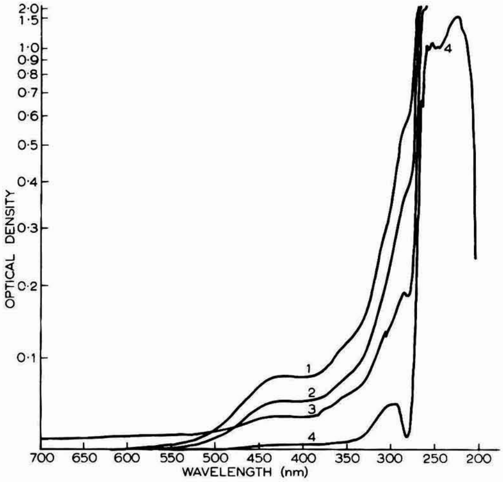


Figure 3. Absorption spectra of the 70:30 mixture of hard and soft lac, (1) 1.5×10^{-3}, (2) 10^{-3}, (3) 4.5×10^{-4}, (4) 10^{-4} g/ml

Results

The absorption spectra of the soft and hard lac fractions of shellac in the complete UV and visible regions at different concentrations are shown in figures 1 and 2 respectively. For soft lac, distinct absorption maxima were obtained at 225 and 435 nm. A small absorption peak and a shoulder were also noticed around 285 and 350 nm respectively. For hard lac (Figure 2), absorption maxima were obtained at 225, 255, 260 and around 290 nm. The absorption maximum at 425 nm was not sharp and two shoulders were noticed at 315 and 215 nm.

The absorption spectra of different ratios of hard and soft lac fractions viz. 70:30, 50:50 and 20:80 are shown in figures 3, 4 and 5 respectively. For the 70:30 hard/soft lac mixture (Figure 3), absorption maxima were obtained at 225, 255, 260, around 290 and at 425 nm. Two shoulders were observed at 350 and 215 nm. For the 50:50 hard/soft lac mixture (Figure 4), absorption maxima were obtained at 227.5, around 290 and at 425 nm. The presence of one shoulder at 350 nm was also noticed. For the 20:80 hard/soft lac mixture (Figure 5), absorption maxima at 227.5, 272.5, around 290 and at 435 nm and one shoulder at 350 nm were recorded.

The absorption at 435 nm by soft lac and by the 20:80 hard/soft lac mixture; and the absorption at 425 nm by hard lac and by the 50:50 and 70:30 hard/soft lac mixtures obey Beer's law for the concentrations studied in the present investigation. The extinction coefficient ($E^{1\%}_{1cm}$) values for all these have been evaluated from a plot of optical density versus concentration at 435 and 425 nm (as described above) and are shown in Table 1. The values of the optical density noted at 425 and 225 nm are shown. The corresponding values obtained for parent shellac have also been included in the same table for comparison. The highest extinction coefficient was obtained for soft lac. It may be seen from the table that the values of optical density at 425 and 225 nm and the extinction coefficient for the 70:30 hard/soft lac mixture are almost identical to those obtained for parent shellac.

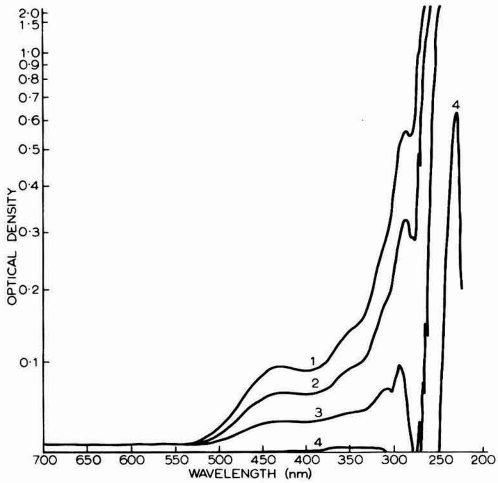


Figure 4. Absorption spectra of the 50:50 mixture of hard and soft lac, (1) 1.5 × 10^{-3}, (2) 10^{-3}, (3) 4.5 × 10^{-4}, (4) 10^{-4} g/ml

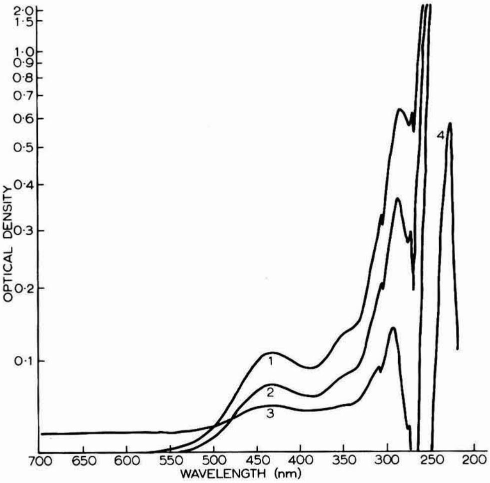


Figure 5. Absorption spectra of the 20:80 mixture of hard and soft lac, (1) 1.5 × 10^{-3}, (2) 10^{-3}, (3) 4.5 × 10^{-4}, (4) 10^{-4} g/ml

Discussion

Refs, 1, 3, 4, 6-8

The study on the absorption spectra of shellac has revealed that the absorption maximum and shoulder obtained at 425 and 350 nm respectively were due to the erythrolaccin. Other absorption maxima obtained in the UV region were due to the terpenic acids present[1]. For hard lac and also for the 50:50 and 70:30 hard/soft lac mixtures, the absorption maxima due to erythrolaccin occurred at 425 nm. For soft lac and in the 20:80 hard/soft lac mixture, however, the peak due to erythrolaccin occurred at 435 nm. This shift in the position may be due to different structural environments in the soft lac. At particular concentrations (e.g. 10^{-3} g/ml), the greater absorbance noted at 425 nm in the case of soft lac compared to that of hard lac indicates the presence of a greater quantity of the lac dye, erythrolaccin, in soft lac than in hard lac. The physical appearance of the two lacs also supports the above observation. Also, for hard lac the absorption peak at 425 nm was not as sharp as that obtained in the case of soft lac.

The absorption spectra of soft lac, of the 50:50 and 20:80 hard/soft lac mixtures in the UV region were found to be similar to those of shellac reported earlier[1]. For hard lac, however, a few additional absorption maxima were obtained. In Table 2, the position of the absorption maxima obtained for different terpenic acids in ethyl alcohol are shown. An inspection of Table 2 and Figure 2 reveals that the peaks obtained at 255 and 260 nm were due to jalaric acid. Since jalaric acid is also present in soft lac, the absence of the above peaks in the spectra of soft lac is difficult to explain. This might be due to the fact that the amount of jalaric acid present in the soft lac (6.5 per cent)[6] is quite low compared to that in the hard lac (27 per cent)[4], and/or that a different structural environment exists in soft lac.

Table 1

Some characteristics of hard and soft lac resins and mixtures of them

Resin	OD at 425 nm conc. 10^{-3} g/ml	$E^{1\%}_{1\,cm}$	OD at 225 nm conc. 10^{-4} g/ml
Hard lac	0.04	0.5	1.7
Soft lac	0.076	0.8	0.91
70:30 hard/soft lac mixture	0.052	0.53	1.6
50:50 hard /soft lac mixture	0.062	0.63	0.62
20:80 hard/soft lac mixture	0.07	0.73	0.581
DL shellac	0.057	0.54	1.65

Table 2

Positions of the absorption maxima of the different constituent terpenic acids of shellac[4]

Name of acid	λ max in nm
Shellolic	229
Epishellolic	228
Jalaric	210, 230 and two small peaks at 260 and 275
Laccijalaric	222
Laksholic	228
Epilaksholic	225
Unidentified new acid	210, 225 and 274

A study of the UV absorption spectra of polyvinyl alcohol derived from polyvinyl acetate polymerised in the presence of acetaldehyde has revealed that absorbance at 225 and 275 nm increases with an increase in the aldehyde content of the vinyl acetate monomer[7]. The absorption peaks obtained at 225 and around 275 nm in the present study may be interpreted as arising from the aldehyde groups present in the terpenic acids.

The close similarity of the values of optical density at 425 and 225 nm, and in the $E_{1cm}^{1\%}$ values of the 70:30 hard/soft lac mixture with those values of the parent shellac (Table 1) supports the proposition that shellac is a mixture of 70:30 parts of hard and soft lacs. The study on the dielectric behaviour of shellac and mixtures of hard/soft lacs has also led to the same conclusion. The dielectric parameters obtained for the 70:30 hard/soft lac mixture were found to be almost identical to those of the parent shellac[3]. The spectrum in the UV region for the 70:30 hard/soft lac mixture, however, was found not to be identical to that of shellac[1]. This suggests that the spatial distribution of the different polar groups in the mixture is not identical to that in the parent shellac. Bhattacharya[8] suggested that the constituent lacs exist in the parent lac in the form of a solid solution or even joined by some kind of weak chemical bonding, and that once the original arrangement is disturbed it cannot be restored.

Acknowledgement

The authors are indebted to Dr T. P. S. Teotia, director of the Indian Lac Research Institute, Mr S. Kumar, head, Division of Chemistry, and Dr B. B. Khanna also of the Institute for their kind interest in the work. The authors are also thankful to Mr Deodhari Singh, Mr T. K. Saha and Mrs P. Devi for technical assistance.

[Received 10 August 1981

References

1. Goswami, D. N., Prasad, N. and Das, R. N., *JOCCA*, 1981, **64**, 20.
2. Bhattacharya, G. N., *Indian J. Phys.*, 1944, **18**, 97.
3. Goswami, D. N., *J. Appl. Polym. Sci.*, 1979, **24**, 1977.
4. Prasad, N., PhD Thesis, Ranchi University, India, 1979.
5. Khurana, R. G., Singh, A. N., Upadhye, A. B., Mhaskar, V. V. and Sukh, Dev, *Tetrahedron*, 1970, **26**, 4167.
6. Chatterjea, J. N., Sengupta, S. C., Mishra, G. S. and Agarwal, S. C., *Indian J. Chem.*, 1976, **14B**, 719.
7. Yamaguchi, T., as cited in 'Encycl. Polymer Sci & Tech.', Vol. 14, *Interscience*, 1971, pp. 177-8.
8. Bhattacharya, R., as cited in 'Chemistry of Lac', Bose, P. K., Sankaranarayanan, Y. and Sengupta, S. C., *Indian Lac Research Institute*, Ranchi, India, 1963, ch. iv, p 61.

J. Oil Col. Chem. Assoc., 1982, 65, 227-238

Aqueous gloss enamels*

By A. Mercurio, K. Kronberger and J. Friel

Rohm and Haas Company, Research Laboratories, Spring House, Pennsylvania 19477 USA

Summary

A new approach to quick-drying high gloss enamels involves the use of high molecular weight aqueous latex polymers in which the desired enamel hardness and resistance properties are achieved through a high polymer glass transition temperature. Coalescence of such hard latex polymers under ambient drying conditions requires judicious selection of coalescing solvents, for which a useful model is proposed. Another key for good brushing application is use of a new class of aqueous thickeners which provide more alkyd-like flow/build rheology than conventional cellulosic thickeners. The resulting coatings represent faster-drying, non-yellowing and low air polluting replacements for solvent-based oxidising alkyd enamels and which, moreover, possess all of the convenience and safety features of an aqueous system.

Keywords

Types and classes of coatings and allied products

- enamel
- gloss finish
- water base paint

Raw materials for coatings

binders (resins, etc.)

- emulsion resin

surface active and rheological agents

- thickener

Processes and methods primarily associated with

drying or curing of coatings

- coalescence
- drying

Properties, characteristics and conditions primarily associated with

materials in general

- glass transition point

bulk coatings and allied products

- water dispersible

dried or cured films

- adhesion
- film defect
- performance

Miscellaneous terms

- formulation

Peintures-émulsions brillantes

Résumé

Une nouvelle tentative à la mise au point des peintures-émulsions de haut brillant comprenant l'emploi des latices aqueuses de polymères de poids moléculaire élevé où les caractéristiques exigées de dureté et de résistance du feuil sont assurées par la température de transition vitreuse élevée du polymère. On propose un modèle èfficace pour faciliter la sélection prudente des solvants qui provoquent la coalescence de telles latices dures de polymères sous les conditions ambiantes de séchage. Une autre possibilité pour assurer de bonnes caractéristiques de brossabilité, c'est l'emploi d'un nouveau type d'èpaissisant aqueux qui met en èvidence des caractèristiques rhéoloigiques plus semblables à celles des résines alkydes que celles des épaissisants cellulosiques usuels. En tant que remplacements des peintures alkydes solubles en solvants organiques et séchant par oxydation, les peintures décrites dans cet article sont résistantes au jaunissement, sèchent plus rapidement, dégagent moins d'agents polluers à l'atmosphère. En même temps elles possèdent tous les avantages et les caractéristiques de sécurité des systèmes aqueux.

Glanzdispersionsfarben

Zusammenfassung

Ein neuer Versuch zur Formulierung der Hochglanzlacke befasst sich mit der Anwendung der wässrigen Latex von Hochmolekulärgewichtpolymeren, wobei die erwünschten Härte- und Beständigkeitseigenschaften durch die hohe Glasübergangstemperatur der Polymeren erreicht werden. Um die Koaleszenz des solchen Hartpolymerlatex, bei den Umgebungstrocknungsbedingungen zu erzielen, wird ein nützliches Vorbild vorgeschlagen, auf dass man die notwendige und sorgfältige Wahl der zweckmässigen Lösungsmittel erreichen kann. Eine andere Möglichkeit die gute Streichbarkeit zu begünstigen, befindet sich in der Anwendung einer neuen Klasse wässriger Verdickungsmittel, die rheologische Eigens-

---

*Presented by A. Mercurio at the OCCA meeting in Rotorua, New Zealand 2 August 1980

chaften ä'hnlicher als der der Alkydharze wie der der konventionellen Celluloseverdickungsmittel zeigen. Die folgenden Beschichtungen darstellen schnellertrocknende, vergilbungsbeständige, niedrigumweltfeindliche Ersätze für auf organische Lösungsmittel gegründete lufttrocknende Alkydharzlacke, und die ausserdem alle Vorteile und Sicherheitseigenschaften eines wässrigen Systems besitzen.

Table 1
Attributes of acrylic latex coatings

1. Good film properties

   Good early water-resistance, water white colour, colour retention, heat/oxidation/weathering stability, adhesion

2. Convenience factors

   Easy application, low-odour, fast-dry, water clean-up, good can stability

3. Safety and health elements

   Non-flammability, low toxicity, low air pollution

4. Economics

   Cost competitive with alkyds now, with continuously increasing solvent costs favouring latex

1. Introduction

Because of the many benefits of water-based paints, there has been widespread effort to extend water-borne technology to the high gloss trade sales enamel field now dominated by solvent-based alkyds. Prominent technical approaches have included improved latex vehicles, alkali-soluble modifiers for latex vehicles and water-reducible alkyds, none of which up to now have been fully satisfactory.

The acrylic latex systems offer a particularly attractive complement of attributes, as enumerated in Table 1. These include good film properties, convenience of use, relative freedom from safety and health hazards and increasingly favourable economics relative to solvent-based alkyds. But up to now, these vehicles have been most suitable for flat, sheen and semi-gloss paints rather than high gloss enamels. There are several reasons for this; but key deficiencies have been sufficient vehicle hardness to accommodate minimally pigmented full gloss formulations and an enhanced degree of film perfection desired in a brush or roller applied paint with a high level of reflectance to the viewer.

This paper will review means of achieving these goals by combining new hard latex vehicle with a recently introduced alkali-soluble latex thickener which provides alkyd-like paint rheology.

2. Design of hard latex polymer

Ref. 1

(A) Glass transition temperature

Solvent alkyds, of course, achieve their good hardness and resistance properties by becoming crosslinked via air oxidation of their drying oil fatty acid components. This approach can be considered with a latex vehicle, but it will require synthetic breakthroughs and, in any event, will introduce the unwanted alkyd problems of can stability, slow-cure, yellowing and outdoor degradation into an acrylic latex system. An alternative approach is the use of inherently hard latex vehicles with higher glass transition temperatures than at present.

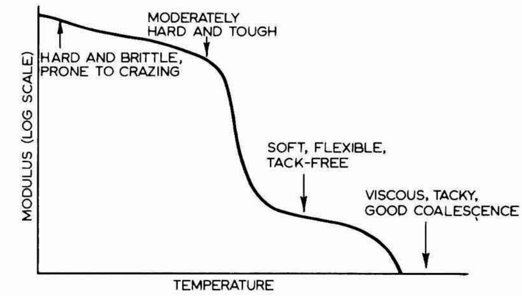


Figure 1. Typical modulus/temperature curve for thermoplastics

Figure 1 is a generalised modulus/temperature curve for a high molecular weight thermoplastic acrylic latex vehicle. By copolymerising monomers which form "hard" or "soft" polymers, the curve can be shifted to the left or right so as to offer polymers which at room temperature are hard and brittle, moderately hard and tough, soft and flexible or very soft and tacky. Up to now, thermoplastic latex polymers for air dry coating applications have been of the soft/flexible variety, with glass transition temperatures of perhaps 5 to 20°C. These have been selected for the very good reason that latex particle coalescence and good film formation upon water evaporation, whether assisted by coalescing solvents or not, will proceed with a high degree of assurance when the glass transition temperature is below room temperature. On the other hand, the hard/tough coatings we desire require polymers with glass transition temperatures above room temperature; their use will run the risk of improper coalescence, even in the presence of coalescing solvents, as described later.

Table 2

Approximate glass transition temperature (T_g) and hardness of commercial thermoplastic coating systems

Application	Binder	Approx. system T_g (°C)	Knoop indenter hardness number (KHN)
Automotive topcoats	Plasticised PMMA	55	10
Furniture finishes	Plasticised nitrocellulose		9
Metal finishes	Plasticised solution vinyl	45	7
Clear wood finishes	Shellac	45	5
Factory-applied cement/ asbestos coatings	Hard acrylic latex	65	12

Table 3

Experimental emulsion E-1630 typical physical properties

Appearance	White milky liquid
Solids content	45% by weight
Supplied pH	8
Viscosity at 25°C	500 cps
Weight per US gallon	8.64 lbs
Bulking value of dry solids (gal/lb)	0.110
Approximate polymer T_g	50°C

(B) Dry and wet adhesion

Unlike industrial coatings outlets for hard thermoplastics, where the surface of the substrate to be coated is well defined and controlled, the trade sales enamel markets must contend with a variety of poorly defined surfaces. Prominent among these are new wood, old paint, porous wall board, cementitious surfaces and bare metal substrates. While proper priming before applying a gloss topcoat is essential and likely on substrates such as porous board and bare metal, a successful gloss enamel will be applied directly and must adhere to surfaces such as old alkyd repaint or new wood. This kind of adhesion capability, both under dry and wet conditions, has been designed into many of the softer acrylic latex vehicles, but it is needed even more with hard latex polymers that experience higher peeling/cracking stresses at the coating/surface interface.

Figure 2 demonstrates that good wet adhesion to an old alkyd surface can be achieved with a properly designed hard acrylic latex vehicle, but the rule for latex vehicles borrowed from other fields is poor wet adhesion, as evidenced by the coating on the left hand side of the same alkyd surface. Both coatings were allowed to dry for one or two weeks before they were spotted with water for half an hour and subjected to cutting action with a set of spaced razor blades and then a scotch tape pull.

(C) Characteristics of specially designed hard latex

The physical properties of a new hard latex polymer with good adhesion characteristics, designated Experimental Emulsion E-1630, are listed in Table 3. The vehicle has a white milky appearance, 45 per cent solids content by weight and low viscosity, much like the familiar softer latex vehicles. However, as supplied, this latex is not a film former, and it must be formulated with specially selected solvents or coalescing agents, as described in the next section.

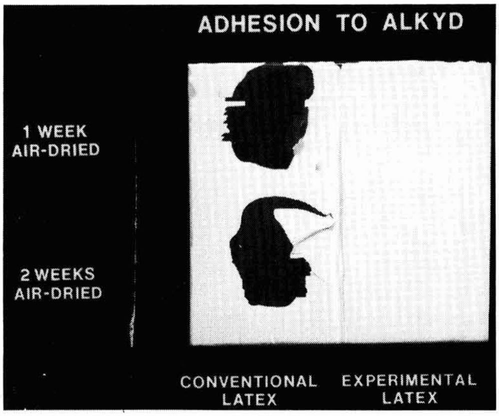


Figure 2.

3. Coalescing solvents for hard latex

Refs, 4-6

(A) Model for coalescents

Hard latex polymers can be, and routinely are, coalesced into good continuous films by use of solvents that temporarily plasticise the hard compositions and then leave by diffusion and evaporation after they have done their job. These solvents are called coalescents, for short. The thermodynamic compatibility of a solvent with the polymer and its inherent plasticising ability are, of course, the first factors to be considered in assessing coalescent effectiveness. But equally important is the mode of distribution of the solvent between the aqueous continuous phase and the dispersed latex phase. A number of workers have carefully measured such distribution coefficients for a variety of solvents and latex compositions[22]. Hoy[2] of

Table 4
Classification of co-solvents by distribution pattern

Area of solvent concentration	A	AB	ABC	C
Action of solvent	diluent	active coalescent	coupling solvent	wet-edge agent
General type	–aromatics –aliphatics	–esters –ketones	–ether-alcohols	–glycols –hydrophilic ether-alcohols
Advantages	–some coalescence	–best coalescence	–good coalescence –open time	–open time –freeze/thaw stability
Deficiencies	–inefficient –difficult to incorporate –tendency to lower gloss	–low open time –early blocking	–stability –can be wicked away –variable flow/ levelling –early blocking and tack –early water resistance	–non-coalescence –can be wicked away –early water resistance –early recoatability

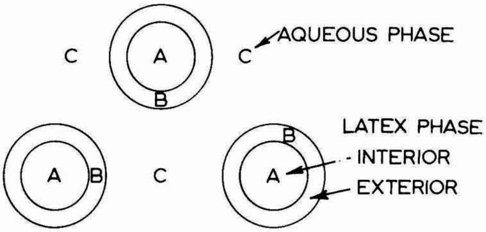


Figure 3. Model for coalescent distribution in latex

Union Carbide has shown quantitative correlation between amount of coalescent distributed in the polymer and subsequent efficiency in aiding film coalescence. Consequently, redistribution of coalescent into the polymer phase from the aqueous phase does not occur to a sufficient degree as water evaporation apparently is not fast enough to aid coalescence during the critical film forming stage.

From experience, it is helpful to use a model for coalescent distribution that has one greater level of complexity, namely, the ability of the coalescent to plasticise/solubilise the surface region of the latex particle as well as the particle interior, as shown in Figure 3. Typically, the surface region of a latex particle contains surfactant, persulfate ion polymer end groups, carboxylate anions and other more hydrophilic components than the major fraction of the polymer which resides in the latex centre. The surface region has a composition similar to a water-reducible/water-solubilisable polymer.

A first approximation of just how hard a latex enamel should be can be gleaned from examination of the characteristics of already optimised commercial thermoplastic coatings used in other outlets[1]. The information on commercial coatings presented suggests that a glass transition temperature in the range of 45-65°C and with Knoop Indenter Hardness Numbers of 5 to 12 is a good target area.

This model helps to discriminate between two kinds of solvents both of which distribute almost completely into the polymer phase, but with the more hydrophilic submembers of the group giving better effectiveness and better gloss in pigmented coatings. Conversely, it explains why mineral thinners and high boiling aromatics which may be introduced inadvertently into a latex coating via a defoamer or oil modifier, etc., can enhance the action of the main coalescent even while adversely affecting gloss and while being ineffective coalescents when used alone.

(B) Selection of specific types

Table 4 presents a general classification of solvent categories by their dominant distribution patterns, and characterises the advantages and deficiencies of each type. The classification is an oversimplification that will be subject to many exceptions, but it is a zero order approximation that is easy to deal with and remember. Generally speaking, the glycols are non-coalescents which stay in the aqueous phase where they impart wet-edge and freeze-thaw properties. The ether-alcohols are good coalescents that also serve as their own wet-edge agents due to approximately equal distribution between polymer and aqueous phases. But, for good coalescence, they must be used at about twice the level of the very efficient esters and ketones, which distribute almost completely into the polymer. Also, the ether-alcohols as well as the glycols can be wicked away with the water phase when latex systems are applied to porous substrates. High levels of any good coalescing solvents can cause localised kickout, heat and freeze-thaw stability problems, but a double level of ether-alcohols can be particularly troublesome. A superior balance of performance is usually obtained by blending the efficient ester coalescents with glycol wet-edge agents rather than using ether-alcohols for both functions. Aromatic and aliphatic solvents are generally inefficient coalescents and may reduce gloss. Furthermore, both aromatic and ketonic solvents are usually disliked because of their strong and distinctive odour.

As a final word on generalities, it should be noted that

> "Hoy[2] of Union Carbide has shown quantitative correlation between amount of coalescent distributed in the polymer and subsequent efficiency in lowering the minimum film formation temperature. This suggests that re-distribution of coalescent into the polymer phase from the aqueous phase during drying has not occurred to a sufficient degree to aid coalescence during the critical film forming stage when latex particles first touch and begin to sinter. Upon further water evaporation, coalescing solvent must perforce re-distribute into the polymer phase and this will help longer film coalescence."

Table 5
Specific examples of solvents by distribution pattern
(in approximate order of decreasing volatility)

	Area of solvent concentration			
	A	AB	ABC	C
Fast	xylene	ethoxy ethylacetate	propoxy propanol	ethoxy ethanol
	butyl benzene	isophorone	butoxy ethanol	
	dodecane	butyl benzoate		methoxy ethoxy ethanol
		diisopropyl adipate	hexoxy ethanol	propylene glycol
↓		Texanol®	butoxy ethoxy ethanol	ethylene glycol
Slow	ink oil	dibutyl phthalate	hexoxy ethoxy ethanol	diethylene glycol

Texanol® is 3-hydroxy–2,2,4–trimethyl pentyl isobutyrate from Eastman Chemical Co.

Table 6
Film formation/properties from co-solvents (20% on latex solids)

Solvent	Type	Film form. at 23°C, 50% RH	Thickening	Heat stability at 60°C, 10 days	Freeze-thaw stability, cycles
Ethylene glycol	C	no film	no	pass	5
Propylene glycol	C	no film	no	pass	5
Methoxy ethoxy ethanol	C	mft cracks	no	pass	5
Butoxy ethoxy ethanol	ABC	good film	moderate	pass	1
Butoxy ethanol	ABC	good film	moderate+	pass	0
Texanol	AB	good film	high	pass	0
Solvesso 150	A	good film	high	pass	0

the hydrogen bonding parameters of the solvents are of key importance in determining their distribution patterns. Thus, while hydrogen bonding generally increases in going from aliphatics to esters to ether-alcohols to glycols, it obviously is possible to alter the overall hydrophobic/hydrophilic balance in any given family and cause a crossover in distribution pattern.

Table 5 lists a number of specific solvents by the over-simplified dominant distribution pattern. Moreover, it covers solvents with a range of volatility in order of decreasing volatility[4]. Based on the generalities made earlier, coupled with a major concern that coalescents which evaporate too fast may result in occasional catastrophic cracking failures, the authors' *a priori* recommendation is to use relatively slow evaporating ester coalescents in combination with glycols of moderate evaporation rate, namely, the framed areas in Table 5.

When a variety of solvents are added to the hard acrylic latex, E-1630, at the 20 per cent on latex solids level, the expected trends are observed. Note in Table 6 that ethylene and propylene glycols as well as methoxy ethoxy ethanol are poor coalescents, cause little or no latex thickening and have good freeze-thaw stability; methoxy ethoxy ethanol does display a low level of coalescing ability, however. The other solvents all produce visually good films at room temperature, poor freeze-thaw stability, and, with Texanol and high boiling aromatic Solvesso 150, show greater latex thickening than with the butyl ether alcohols. However, good film formation under adverse drying conditions needs to be demonstrated in order to compete with existing systems.

Based on earlier discussions, it can be surmised that adverse drying conditions include low ambient temperatures and application to porous substrates that can wick away coalescents. But there is one other critical factor, namely, relative humidity[5]. Figure 4 shows the drying characteristics of an E-1630 precursor under three relative humidities[6]. While the film at each relative humidity becomes tack-free at about the same point of 80 per cent water evaporation, the time required for the water to evaporate becomes increasingly longer at higher relative humidities. A serious consequence affecting film quality can occur when the rate of water evaporation relative to coalescent evaporation becomes slow enough to cause noticeable depletion of coalescent before and during the critical film forming stages.

Recognising these problems, a search was made for a set of conditions that were not so severe as to guarantee

Table 7
Film formation/hardness/stability with favoured coalescents
(all formulations also contain 40 lbs/100 gallons of methoxy ethoxy ethanol)

Coalescent (% on latex solids)	Film cracking at 10°C/75% RH/16 hours		Indenter hardness (Knoop number)			Freeze-thaw stability cycles
	on black vinyl	on wood	2 wk	4 wk	8 wk	
Texanol (15%)	none	none	1.0	1.1	1.3	5
Texanol (10%)	none	moderate	1.0	1.3	2.0	
Texanol (7½%)	moderate	heavy	1.9	1.7	2.9	
Diisopropyl adipate (15%)	none	none	1.2	1.8	4.0	5
Butyl benzoate (15%)	none	none	2.0	4.3	6.7	2
Butyl benzoate (10%)	slight	heavy	2.8	4.4	6.3	

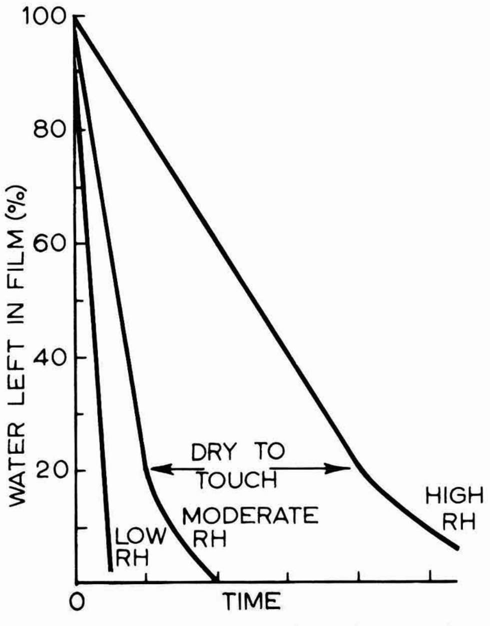


Figure 4. The effect of relative humidity on the evaporation of water from a latex paint film

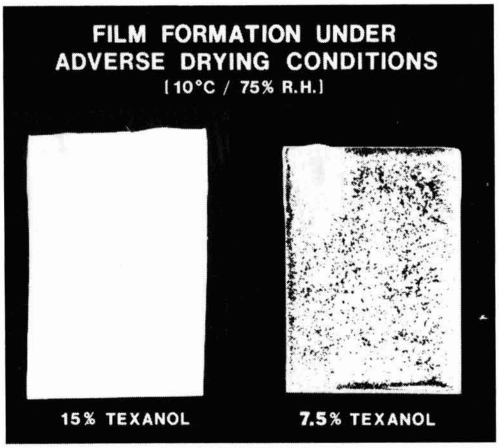


Figure 5.

failure of a hard latex system, but rather that represented a realistic level. Paint drying conditions of 10°C and 75 per cent relative humidity were selected for both a sealed substrate (black vinyl chart) and a realistic porous substrate (bare white pine). Moreover, a decision to proceed only with interior enamel applications until tests outdoors warranted broader recommendations was made. Figure 5 shows the results of such testing on black vinyl sheets for E-1630 white formulations containing 7.5 and 15 per cent Texanol on polymer solids in addition to 40 lbs/100 gallons of methoxy ethoxy ethanol wet-edge agent. Note the cracking with the 7.5 per cent level of Texanol, which did not take place at the 15 per cent level. The results of additional tests of this sort with faster evaporating coalescents are presented in Table 7. While 15 per cent Texanol offers good coalescence, full hardness development will take a long time with this material at room temperature. The 15 per cent levels of diisopropyl adipate and butyl benzoate coalescents also pass the film formation tests and with better hardness development characteristics. But butyl benzoate is of questionable value because of its distinct odour and marginal freeze-thaw stability.

4. Formulation of coatings

Refs, 7,8

(A) Thickener selection

Until fairly recently, latex paints have not had optimum rheology for applying uniform thick coatings by brush, roller or other means. Brush applied paints in particular have suffered from poor build and poor levelling, evidenced by the weak hiding in the valleys of brush marks. Figure 6 is a schematic representation of the apparent viscosity versus shear rate of a conventionally thickened latex paint versus a Newtonian fluid and versus a paint with "ideal" rheology. Note that the Newtonian fluid is not an ideal paint because its viscosity at low shear rate is too low, causing pigment settling in the paint and sagging on vertical surfaces. Furthermore, the relatively

Table 8

Thickening of E-1630 white paints by Primal RM-4 VS. Primal RM-5 (all paints adjusted to 95 Krebs Units)

	RM-4			RM-5		
Level (solids on latex solids)	2%	4%	8%	1%	2%	4%
Volume solids	37%	35%	32%	37%	35%	32%
ICI viscosity (poise)	1.0	1.4	1.7	1.0	1.4	1.8
Qualitative ratings (10 = perfect)						
Flow/levelling	5½	7	9	6	7½	9
Sag resistance	9	8	7	9	8	7
Alkali resistance	9	7	6	9	9	7
					optimum	

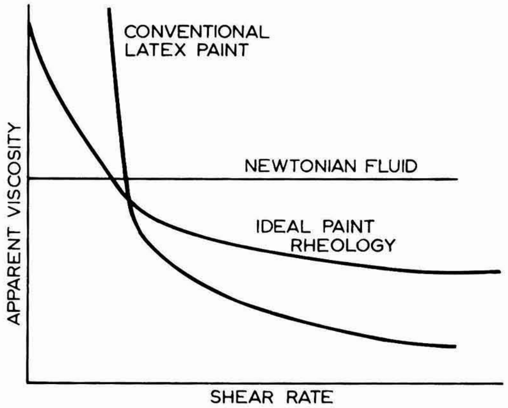


Figure 6. Rheological characteristics of paint systems

Table 9

Primal RM-5, Typical physical properties

Description	Anionic alkali soluble acrylic emulsion
Appearance	White milky liquid
Solids content	30% by weight
Supplied pH	2.7
Viscosity at 25°C	<100 cps
Weight per US gallon	8.8 lbs
pH Range of efficient thickening	8 and above
Preferred pH range	8 to 8.5
Neutralisers	NH_3, DMAE (with E-1630, offers better gloss/ sag resistance)

high viscosity at high shear rate makes it difficult to apply by brush or roller. At the other extreme is the conventional latex paint which is easy to apply, yet the very low high shear viscosity causes problems of overspreading and the very high viscosity at low shear rate prevents the levelling of brush marks or roller ribs. The "ideal" rheology optimising these opposing effects is shown by the third curve. Over the years, the designers of alkyd resins and paints have achieved this "ideal" rheology and hence the frequent reference to alkyd-like rheology.

Within the last few years, anionic alkali-soluble thickeners which impart this rheology have been introduced. These thickeners have been variously referred to as associative thickeners, rheology modifiers or polymeric surfactants. Table 8 presents results of studies on E-1630 paints thickened with Primal®* RM-4 and RM-5, which offer the aforementioned rheology. The experiment was designed to enable variations to be made in the level of added thickener while keeping paint viscosity constant at a value of 95 Krebs Units by allowing paint volume solids to vary. Paint build and flow/levelling improved with increasing levels of either thickener, but so did sagging tendency. Futhermore, the Primal RM-5 proved to be about twice as efficient as Primal RM-4 in this system, thereby achieving the desired thickening with less detraction from alkali resistance caused by high levels of alkali-soluble thickener. The 2 to 4 per cent level of RM-5 solids on latex solids appears to be optimum.

The typical physical properties[7] of this new thickener are presented in Table 9. Primal RM-5 is supplied as a white milky liquid at 30 per cent solids (by weight) on the acid side. Efficient stable thickening is achieved by "neutralising" the thickener to pH 8 or above with base and water prior to its addition to a paint. In the specific context of E-1630-based paints, dimethyl amino ethanol is preferred as the neutralising base for RM-5 rather than normally recommended ammonia because it provides enamels with better gloss, sag resistance and stability without serious detraction from resistance properties that might show up in softer, less resistant vehicles. In clear E-1630 formulations, where pigmented gloss is not an issue, the standard ammonia neutralisation recommendation is used.

There was a striking difference in the appearance of brush applied conventional acrylic latex paint vehicles thickened with hydroxyethyl cellulose versus the E-1630 vehicle thickened with Primal RM-5. Using the same E-

---

*The trade name Acrysol® is used in North America

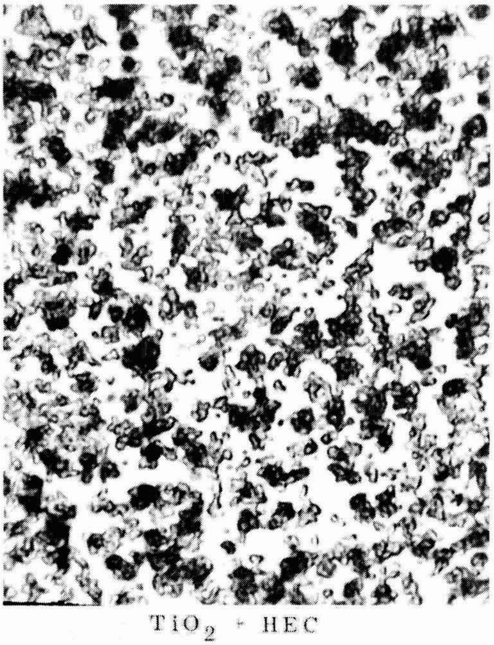


Figure 7.

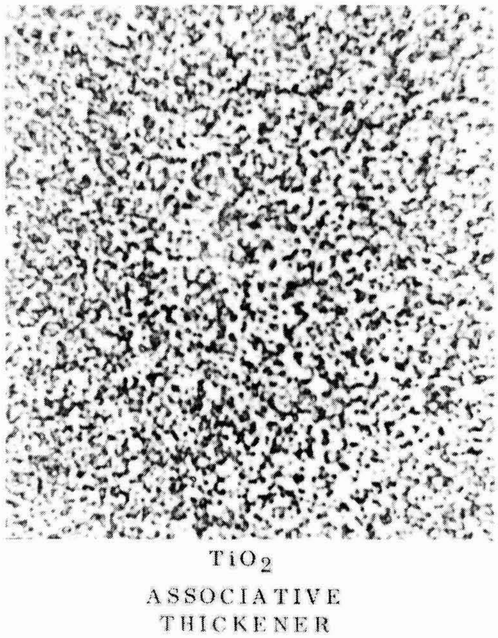


Figure 8.

1630 throughout, there was a major improvement in appearance of brush applied paints when Primal RM-5 thickener was compared to HEC or conventional polyacrylate alkali-soluble thickeners.

In these experiments, not only is the flow and levelling noticeably improved, so is the absolute level of gloss and gloss clarity. The smoothness of the film and the high gloss potential of E-1630 are two of the reasons for this. But a third contributing factor is the tendency of hydroxyethyl cellulose thickener to cause microscopic flocculation in paints to which it is added as opposed to Primal RM-5 with which no flocculation takes place. This flocculation effect is vividly demonstrated in Figure 7 which is a 2,300 × magnification of a TiO_2 dispersion to which HEC thickener has been added. In contrast is the behaviour shown in Figure 8 taken at the same 2,300 × magnification with the same TiO_2 dispersion to which Primal RM-5 has been added. Additional experiments have indicated that the absence of flocculation is a general characteristic of properly designed associative thickeners/rheology modifiers.

(B) Additives to minimise surface defects

The design of a successful aqueous gloss enamel not only means the achievement of excellent flowing/non-sagging paint, it also requires the avoidance of unsightly pinholes, craters and crawling patterns during application. Table 10 lists several classes of additives that can be used for this purpose.

An absolutely essential exercise that must be conducted with aqueous enamels is the identification of suitable defoamers. Fortunately, an increasing variety of defoamers for latex paints have become available, and the suppliers are quite helpful in offering advice and service to solve specific problems.

One must pay attention both to initial defoamer effectiveness and to its persistance through paint processing and storage. Since defoamer effectiveness is affected by practically every ingredient in a latex paint, it is important to leave the optimisation of defoamer type and level to the last step in formulation development. Although there is trial and error experimentation involved, a general approach is to use the potent silicone defoamers in pigment grinds, where they will be well incorporated and less prone to cause cratering. Where this is not possible, a more easily dispersed non-silicone defoamer can be

Table 10
Necessary/helpful additives to reduce paint defects

A. Defoamers – necessary (usually 1 to 5 lbs/100 gallons)
  - Add potent silicone types in pigment grind, e.g. Foamaster DS
  - add easily dispersed non-silicone types in letdown, e.g. Foamaster AP

B. Anticrater agents – helpful (≈1 to 3 lbs/100 gallons)
  - Add anticrater agent to compensate for poor or excessive defoamer incorporation, e.g. Troykyd Anticrater

C. Wetting agents – helpful (≈1 to 2 lbs/100 gallons)
  - Add wetting agent for wetting of low surface energy/oily surfaces from which latex paint may crawl, e.g. Triton GR-7M

Table 11
Clear formulation based on emulsion E-1630

Materials		Pounds	US gallons
Water		133.9	16.13
Latex vehicle (exptl. E-1630 – 45%)		583.8	67.48
Wet-edge agent (methoxy ethoxy ethanol)		52.6	6.19
Coalescent* (Texanol®)		26.3	3.31
Non-silicone defoamer (Foamaster® AP)		5.0	0.65
Thickener (Primal® RM-5 – 30%)	premix	17.5	2.01
28% ammonia		33.5	4.03
Water		1.5	0.20
		854.6	100.0

Weight solids	31%
Volume solids	29%
Viscosity (krebs)	78
pH	7.8
Coalescent (on latex solids)	10%
RM-5 (solids on latex solids)	2%

*Diisopropyl adipate can be substituted at 15% level

introduced in the paint let-down steps. Sometimes it is beneficial to add both defoamer types in the same paint[8]. Also, the mode of paint application will have considerable bearing on foam generation with brush application being better than roller and with short-nap rollers being a must.

Poorly incorporated defoamers or other low surface energy occlusions can result in cratering defects on a paint surface. Special proprietary anticratering agents, such as Troykyd® Anticrater, are available to ameliorate these problems when they occur. A related kind of defect is crawling caused by non-wetting of oily spots or low surface energy areas on the substrate surface. A hydrophobic surfactant, Triton® GR-7M, has been employed successfully in overcoming occasional problems in this area.

(C) Starting point formulations

The individual paint formulation trends detailed in earlier sections are combined to produce starting point formulations. Table 11 is a clear 100 gallon formulation for experimental emulsion E-1630 which uses 52 lbs of methoxy ethoxy ethanol as a wet-edge agent and 10 per cent Texanol, or alternatively 15 per cent diisopropyl adipate, as coalescent. The primal RM-5 latex thickener is used with ammonia neutralisation at the 2 per cent solids on latex solids level.

Table 12 lists a tintable white formulation for E-1630 which contains about 200 lbs per 100 gallons of TiO_2 that has been Cowles ground in the normal fashion. Methoxy ethoxy ethanol is again used as wet-edge agent at the 45 lb level and Texanol is the coalescent at the 15 per cent level on polymer solids. Primal RM-5 is neutralised with DMAE and added at the 3 per cent solids on polymer solids level for enhanced gloss, flow and sag resistance. For light tint colours, up to six ounces of a glycol/surfactant predispersed universal colourant, such as Tenneco's Colortrend® 8800 series, can be added without serious compromise of film properties.

A deep-tone black formulation for E-1630 is presented in Table 13, and it serves as a model for other deep-tone colours. Here, predispersed colours in polymer/water medium, such as Harshaw's Aurasperse® line, are preferred because they detract less from film properties versus glycol/surfactant dispersed universal colourants. Acceptable deep-tint formulations are also achieved simply by mixing some of the tintable white with the deep-tone colours.

The ability to achieve good results over a range of colours was demonstrated by smooth glossy palettes of coatings that were achieved when brush applied to Morest charts.

5. Performance of latex gloss enamels versus control paints

This concluding section emphasises the very good balance of properties achieved with gloss enamels based on hard E-1630 latex in combination with Primal RM-5 rheology modifier and specially selected coalescing solvents by comparing their performance with those of commercial gloss or semi-gloss paints based on conventional latex vehicles, a water-reducible alkyd and solvent-based alkyds. Table 14 lists the application and drying properties accumulated in this comparison. Note that at similar Krebs Unit viscosities for all paints, the E-1630 enamel achieves flow and film build that is in the category of the alkyds and better than with conventional latex systems even though its brush drag (indicated by ICI viscosity) is intermediate to the two other classes of paint and definitely less draggy than with solvent alkyds. The early dry speed of the E-1630 paint is better than with all other systems, as indicated by excellent recoatability results after 3 hours and 24 hours drying. Finally, roller spatter and sagging tendencies of the paint are also quite good, with the low spatter characteristics being better than with conventionally thickened latex systems and the slight sagging tendency being much preferred over the noticeable sagging of alkyds. Figure 9 shows the actual sagging results observed when paints were brush applied to Morest charts and allowed to dry in a vertical position.

Table 12

Tintable white formulation based on emulsion E-1630
(tintable with up to 6 oz./gallon of colourant such as Colortrend•8800 Series)

		Pounds	US gallons
Cowles grind			
Wet-edge agent (methoxy ethoxy ethanol)		45.5	5.35
Pigment dispersant (Tamol• 731 – 25%)		7.9	0.86
Pigment wetting agent (Triton• CF-10)		1.8	0.20
Silicone defoamer (Foamaster• DS)		2.5	0.33
TiO_2 (TI-Pure• R-900)		196.5	5.66
Letdown			
Water		90.7	10.89
Latex vehicle (exptl. E-1630 – 45%)		525.0	60.90
Coalescent (Texanol•)		35.4	4.47
Preservative (Dowicil• 75)	premix	1.3	0.15
Water		23.0	2.76
Thickener (Primal• RM-5)	premix	23.6	2.69
Dimethyl amino ethanol		4.3	0.58
Water		43.0	5.16
		1000.5	100.00

PVC	18%
Weight solids	44%
Volume solids	33%
Viscosity (Krebs)	95
pH	8.5
Coalescent (on latex solids)	15%
RM-5 (solids on latex solids)	3%

Table 13

Deep-tone (black) formulation based on emulsion E-1630

Materials		Pounds	US gallons
Water		74.1	8.88
Latex vehicle (exptl. E-1630 – 45%)		561.1	65.10
Wet-edge agent (methoxy ethoxy ethanol)		43.2	5.07
Coalescent (Texanol•)	premix	29.1	3.68
Non-silicone defoamer (Foamaster• AP)		4.0	0.52
Preservative (Dowicil• 75)	premix	1.3	0.15
Water		23.1	2.77
Predispersed colour (Aurasperse• W-7012 black – 42%)		20.1	2.03
Thickener (Primal• RM-5 – 30%)	premix	32.4	3.69
DMAE		6.0	0.81
Water		58.8	7.06
Surface wetting agent (Triton• GR-7M)		2.0	0.24
		855.2	100.00

PVC	1.3%
Weight solids	31%
Volume solids	29%
Viscosity (Krebs)	90
pH	8.6
Coalescent (on latex solids)	12%
RM-5 (solids on latex solids)	4%

Table 14
Application/drying of E-1630 enamel versus controls

	E-1630	Commercial semi-gloss and gloss white paints				
	Gloss latex white	Semi-gloss latex – B	Semi-gloss latex – C	Water-reducible alkyd	Solvent alkyd – A	Solvent alkyd – B
Krebs Units	94	86	83	92*	103	86
ICI (poise)	1.5	0.9	1.4	0.9*	2.5	3.7
Natural spread (g/ft^2)	10.6	8.8	9.5	13.5*	9.2	11.2
Flow/levelling (10 = perfect)	9	4	7	9½	9	9½
Sagging	slight	none	none	heavy	slight	severe
Roller spatter	trace	moderate	moderate	v. slight		v. slight
3 h recoat	excellent	fair-poor	poor	fair	fair	fair
24 h recoat	excellent	fair	fair	good	v. good	v. good

*enamel is not stable

Table 15
Performance properties of E-1630 enamel versus controls

	E-1630	Commercial semi-gloss and gloss white paints				
	Gloss latex white	Semi-gloss latex– B	Semi-gloss latex – C	Water-reducible alkyd	Solvent alkyd – A	Solvent alkyd – B
20° gloss	66	21	33	50	66	25
60° gloss	86	69	72	81	85	76
RT blocking (2 psi/16 h) (10 = perfect)						
Face to face (1 day)	6	4	4	6	4	0
Face to back (1 day)	8	6	5	0	6	2
Face to face (1 week)	7	8	7	9	6	8
Face to back (1 week)	10	9	9	4	8	8
Removal of stains (avg. of 8)	85%	84%	68%	71%	83%	88%
60° gloss retained after alkali scrub	96%	46%	75%	59%	96%	98%
Long term yellowing	none	none	none	bad	definite	definite

The performance property comparisons for dried paints are presented in Table 15. Note that the gloss of the E-1630 enamel is equal to that of the alkyd paints and superior to that of conventional latex systems.

Certainly, alkyd paints have the potential for higher gloss than paints of E-1630, as may be needed in automotive refinish or similar uses, but E-1630 enamels seem more than adequate for the trade sales enamel gloss range. The room temperature blocking resistance results with coated Morest charts placed coated face to coated face and coated face to uncoated back show E-1630 enamels to develop blocking resistance much faster than alkyds or conventional latex paints. However, once the alkyd surface becomes fully oxidised and crosslinked, then its blocking resistance will be as good as E-1630 paints at

Figure 9.

room temperature or slightly elevated temperatures and superior at temperatures of 50°C and higher; but again, E-1630 enamels seem more than adequate for trade sales enamel conditions. The ease of stain removal and the alkali scrub resistance characteristics of E-1630 enamels are definitely in the solvent alkyd category and better than conventional latex polymer paints or especially water-reducible alkyd paints. Also, the E-1630 enamels have the non-yellowing traits of the acrylic latex coatings as opposed to the definite yellowing in alkyds.

A final word should be mentioned about the expected exterior weathering characteristics of E-1630 paints. Gloss, colour and mechanical property retention should be good and typical of moderately hard acrylic copolymers. Cold-cycle crack resistance should be good on stable substrates such as primed metal, hard woods and other well primed surfaces. There is definite potential for cracking on soft woods, although it is conceivable that special primers that cushion the cold-cycle stresses may overcome this. But the concern which requires more attention and field testing is the possibility that long spells of cold damp weather after painting may cause premature cracking due to incomplete film coalescence.

6. Acknowledgement

The latex gloss enamel developments at Rohm and Haas Co. have involved many co-workers who are too numerous to mention. The authors must, however, acknowledge the major contributions of Ms C. Lane and Messrs A. Brown, P. Finegan, M. Freedman, R. E. Harren, B. B. Kine, W. Kooch, A. Kowalski, E. Leasure, D. Schall and J. D. Scott.

[Received 6 October 1981

7. References

1. Zimmt, W. S., *Ind. Eng. Prod. Res. Dev.*, 1979, **18**(2), 97.
2. Hoy, K. L., *J. Paint Technology*, 1973, **45**(**579**), 51.
3. Sullivan, D. A., *J. Paint Technology*, 1975, 47(610), 60.
4. Hoy, K. L., *J. Paint Technology*, 1970, **42**(541), 76.
5. Dillon, P. W., *J. Coatings Technology*, 1977, 49(634), 38.
6. Calder, V. L., Proceed. 4th Water-Borne Coat. Symp., New Orleans, LA, 1977, 2, 27.
7. Rohm and Haas Co., Bulletin 81A33, April, 1980.
8. Maher, R. P. and Antonucci, E. M., *Mod. Paint & Ctgs.*, 1979, 43.

Next month's issue

The Honorary Editor has accepted the following papers for publication. They are expected to appear in the July issue:

A new development in water-based can coatings* *by C. G. Demmer and N. S. Moss*

Lower temperature curing blocked isocyanate for use in powder coatings† *by P. MacBride*

Factors affecting metal marking of organic coatings *by L. A. Simpson*

Film characteristics of linseed epoxy esters prepared from novolac based polyepoxide resins *by A. K. Vasishtha and D. Agrawal*

**This paper formed the basis of lectures delivered at the OCCA-34 Exhibition, 27-29 April 1982.*
†This paper was awarded the Roon Award.

reviews

Further information on any of the publications reviewed may be obtained by circling the appropriate *Reader Enquiry Service* number on the form at the back of the *Journal*. Enquiries will be forwarded to the publisher.

Dispersion of Powders in Liquids, with special reference to pigments

Edited by G. D. Parfitt
Third edition
Applied Science Publishers, London and New Jersey, 1981
pp. xv + 518

The reviewer of the third edition of a work such as this has the advantage of being able to compare what was said of the previous editions. The first was reviewed by J. A. L. Hawkey (*JOCCA*, 1970, **53**, 299) and the second by R. J. Cole (*JOCCA*, 1973, **56**, 320) and as the aims and general make-up of the work remain the same, nothing further need be added here. However, several modifications have been made to the content and the contributors. The book now runs to ten chapters, as a result of the addition of an entirely new chapter by Lowrison entitled "Fundamentals of the Breakdown of Solids". As would be expected, the treatment in this chapter is highly theoretical and assumes a reasonable knowledge of calculus on the part of the reader. Though not strictly new, chapter 5 "Principles of Precipitation of Fine Particles" has been substantially rewritten by Füredi-Milhofer and Walton, the latter being the author of the chapter in the previous edition. The present reviewer finds himself in some sympathy with Mr Cole, who questioned the wisdom of including this topic in a volume dealing with dispersion. Whilst he would not wish to see the complete exclusion of the subject, he does wonder whether the amount of space devoted to it is justified.

The authors of chapters 1-3 are, as in the second edition, as follows: "The Fundamental Aspects of Dispersion" by Parfitt; "Properties of the Solid-Liquid Interface of Relevance to Dispersion" by Jaycock; "Electrical Phenomena Associated with the Solid-Liquid Interface" by Smith. These, together with chapters 5 and 6, constitute a comprehensive theoretical treatment of the subject of dispersion. The remaining chapters go some way to relating the theory to the everyday business of dispersion. In chapter 4 Black considers "Surface Active Compounds and their Role in Pigment Dispersions", whilst in chapter 7 Wheeler deals with the "Technical Aspects of Dispersion". The subject of the "Assessment of the State of Dispersion", originally contributed by Bell and Crowl, has been completely rewritten for this edition by Lawrence. Jefferies has revised his original chapter on the dispersion of inorganic pigments to take into account newer developments in this field. Finally the "Dispersion of Organic Pigments" has been treated by McKay and Smith.

Although the number of post-1970 references at the end of most chapters remains rather small, almost all chapters show evidence of careful revision or re-drafting.

In his introduction to this edition, Professor Parfitt expresses the view that in the years since 1969, the date of the first edition, the gap between theory and practice in this field has narrowed *significantly*, but this has not been borne out in the experience of this reviewer, and chapters 1-3, 5 and 6 may still seem rather heavy going to some of those dealing with the practicalities of paint making. Nevertheless, Professor Parfitt is to be congratulated yet again on a good job well done. The work is extremely well printed and this reviewer could find but one omission and one misprint both of which are so trivial that it would be churlish to draw attention to them specifically.

Reader Enquiry Service No. 21

S. A. Ray

---

occa meetings

For information on membership of OCCA, enquiries should be sent to the Association's offices, see front cover for address.

London

Optimised TGIC levels in polyester powder coatings

Mr D. Lawlor of Grilon (UK) Ltd presented his lecture to a small audience at the Great Eastern Hotel, Liverpool St on Thursday 14 January 1982. Section Chairman, Mr B. Gilliam, noted in his welcome that the combined effects of the rail strike and the extreme weather conditions had made travel in London very difficult.

Mr Lawlor, who is a London Section Committee member, detailed the use of triglycidyl isocyanurate (TGIC) in polyester powder coatings.

The use of polyester/TGIC powders has increased steadily in recent years. Originally the polyester used had acid values in the 45-55 range requiring a polyester/TGIC ratio of 90:10. The current practice is to use a polyester with an acid value of 30 in a 93:7 ratio. The effect of varying the ratio between 95:5 and 92:8 was examined.

Three commercially available polyesters were used for tests; two with conventional 20 minutes at 165°C cure and one with a low cure of 10 minutes at 160°C. Powders

occa meetings

were prepared in the standard manner and pigmented with titanium dioxide and coated onto steel panels for mechanical tests; the panels were "trike" degreased and for chemical tests had been phosphated with Bondar 130.

Gelation times did not vary greatly with TGIC ratio for any given temperature. The two standard cure resins gave similar gel times. An indication that gel time need not be an accurate guide to reactivity was shown by the differing rates of cure as measured by Erichsen indentation.

Generally speaking the normal mechanical and optical tests carried out on the coatings did not reveal great differences apart from the fact that the very low TGIC level coating gave noticeably lower values in most tests. Longer cure times in all but the 95:5 panels gave equivalent test results for all TGIC levels.

Most polyester powders are used outside and Mr Lawlor described the effects of accelerated weathering on the mechanical and optical properties of the coatings. Gloss drops slowly after, up to 2,000 hours in an Atlas Weatherometer, then somewhat faster. This trend is largely independent of TGIC. Looking at the effect of Atlas weathering on mechanical properties, a rapid fall off at both high and low TGIC levels in both Erichsen indentation and reverse impact strength was seen.

It is worth noting that pendulum and cross cut values did not show the same trends, suggesting that weathering at all but optimum 93:7 to 92:8 TGIC levels dramatically affects flexibility and adhesion related properties.

Concluding, Mr Lawlor stated that since flexibility and adhesion are so vital to the life of a coating, as opposed to its appearance, the optimum level of 92.5:7.5 polyester/TGIC, using an acid value 30 polyester, thus justifies current practice.

A vote of thanks was given by Dr T. Banfield and acknowledged in the usual way.

B. A. Canterford

Scottish

Haloflex polymers for water-based paints

A meeting of the Scottish Section was held in the Albany Hotel, Glasgow on Thursday 11 February 1982, when Dr A. Burgess of ICI, Mond Division, spoke on "Haloflex polymers for water-based paints".

Dr Burgess described initially the reasons behind the move in the early 1970s to develop water-based protective coatings. The increasing costs of solvent, which accounted for the largest volume cost in the production of oil-based paints, together with restricted availability of solvents and the ecological acceptability of water-based compositions prompted a search for systems which would have the properties of a good solvent-based system such as chlorinated rubber. The need for a polymer with good freeze/thaw stability, low permeability to water and oxygen, good resistance to atmosphere, particularly in coastal conditions, and capable of forming tough, flexible films with good adhesion to steel and concrete led researchers to consider high chlorine content latex polymers.

A number of polymers using vinyl chloride as the hard monomer and vinylidene chloride as the soft monomer were prepared by copolymerisation using a small amount of acrylic monomer as plasticiser. This early work culminated in the development of Haloflex 202. This polymer was a good film former with good chemical resistance and had a lower water vapour permeability than chlorinated rubber. The film gave good results on salt spray testing and Florida exposure. Initially, however, great difficulty was experienced in formulating a suitable primer incorporating the polymer.

Normally, latex paints were formulated at alkaline pH in the range 7-9. However, formulation of a high chlorine content latex into paint at this pH resulted in a pH drift and dehydrochlorination. Attempts were made to buffer the paint but were not successful. Finally it was decided to formulate the paints at an acid pH and eventually a storage stable paint was developed. Dr Burgess gave examples of the formulation and emphasised the need for care in such formulations.

The resulting primer formulation gave good adhesion on steel and good intercoat adhesion with most proprietary top coats. In addition, the primer yielded good results on salt spray testing and compared favourably with solvent alkyds in the American Salt Spray test. These results were illustrated with an excellent collection of slides. One encouraging consequence of the acidic formulation was a much reduced tendency to give flash rusting.

Dr Burgess went on to describe the development of top coats using new latices with a reduced level of vinylidene chloride. Top coats have been difficult to stabilise with Haloflex 202 and a new polymer Haloflex 208 with slightly lower permeability had been developed to overcome this problem. This product was still under test, but results to date were extremely promising.

The new paints could be applied by brush, air spray or airless spray providing the drying temperature was not less than 10°C and was at least 3°C above the dew point. It was also not desirable to apply the paint when the relative humidity was greater than 90 per cent. At 20°C the paint was touch dry in ten minutes, through dry in 30 minutes and would not flash rust.

Dr Burgess concluded by listing application areas for the new products which included factory applied steel primers, container interiors, ship interiors, concrete, cement, asbestos, wood (primer), adhesives, printing inks, paper coatings, and possible outlets in the textile and gypsum industries.

It was particularly gratifying that a large attendance was present to hear such a well delivered and interesting

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 53rd 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)
P.O. Box 560876, 8290 S.W. 120th Street, Miami, Florida, U.S.A. 33156

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

Reader Enquiry Service No. 117

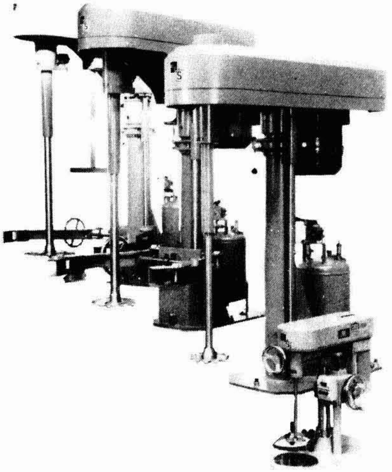
Mix with members of the SUSSMEYER/ OLIVER + BATLLE family.

The comprehensive Sussmeyer/Oliver + Batlle Mixer/Disperser range offers you everything you're ever likely to need, from small laboratory Units of 1.4hp to giant production models of 95hp, including paste mixers having batch capacities of up to 2 tonne.

These superbly engineered machines have been developed to perfection during years of experience in World markets, and are in wide use throughout the paint, ink, ceramic, adhesives, pharmaceutical, plastics, cosmetics and chemical industries.

Features include variable operating speed ranges, full control instrumentation and special vessel location devices which keep abreast of Health and Safety regulations.

For full information on this exciting equipment range, contact OBS Machines Limited, sole sales and service concessionaires for the U.K. and Eire.



OBS MACHINES LIMITED

Plant & Equipment for the Paint,
Ink and allied Chemical Industries —
Sales — Service — Spares

OBS

Unit 34 Heathfield, Stacey Bushes,
Milton Keynes, MK12 6HR
Tel. Milton Keynes (0908) 313186 or 313373
Telex: 825663 (OBSUK G)

Reader Enquiry Service No. 195

occa meetings

lecture, a fact attested to by the generous applause following the vote of thanks.

J. H. Coy

Eastern

Paint viscosity/temperature phenomena

A meeting of the Eastern Branch was held at the Murrayfield Hotel, Edinburgh on Wednesday 17 February 1982 when the speaker was Dr N. Reeves of NL Chemicals, Livingston who lectured on "Alternative means of controlling paint viscosity/temperature phenomena".

The rheology of coatings in everyday work was a topic often not clearly understood and at present was determined on an empirical basis. The effect of temperature variations on such a property served to further complicate the issue. However, a start had to be made in trying to explain the effect, and the speaker illustrated work that had been done in this area with four quite different rheological additives. These were, an amine modified bentonite clay, modified castor oil, modified PE wax and a modified castor oil complex. The rheology of coatings containing these additives was studied using viscosity profiles determined by the method of Ehrlich, Patton and Franco. The data obtained were analysed in terms of several well established mathematical viscosity/temperature relationships, such as the Andrade equation and the Lewis and Squires relationship, using both pigmented (white) and unpigmented long oil alkyd systems. No conformation to mathematical relationships was obtained. Consequently, the prediction of viscosity from measurements at one or more temperatures was unreliable. The choice of additive had a significant effect on the viscosity/temperature response of a coating system. The mode of action of the amine modified bentonite was relatively insensitive to temperature variation, it resulted in a marked increase in viscosity of the system and maintained the viscosity with increasing temperature. The modified castor oil additive similarly increased the viscosity of both pigmented and clear systems, but this effect decreased with increasing temperature. The remaining two additives showed marked viscosity increases when pigment was included in the system while also exhibiting decreases in viscosity with increasing temperatures. Consequently, temperature required strict control during paint testing. The different effects of the inorganic additive, bentonite, and the organic additives, particularly with regard to their interaction with pigments, should be borne in mind when formulating coatings.

A period of discussion followed a very interesting lecture. The vote of thanks was given by Mr T. L. Humphrey, Chairman, Scottish Section, and heartily endorsed by all present.

J. H. Coy

West Riding

Curing behaviour of stoving paints

A lecture meeting of the West Riding Section was held at the Mansion Hotel, Leeds on 2 February 1982.

Mr Prosser of the Paint Research Association (PRA) kindly stepped in at the last minute to present a paper on the above subject. After Mr Prosser had given a brief review of the activities of his interest group within the PRA, he went on to introduce the three discussion topics. These were: how to establish the degree of cure, the rheology of powder coatings and infrared curing of stoving systems.

The state of cure was said to be determined by means of the thermal analysis technique, which was discussed in detail.

It was explained that when stoving systems are heated up, there is seen to be a pause in the rate of increase in the rise in temperature. The temperature rise is measued by means of a differential thermal analyser (DTA).

Mr Prosser explained that the DTA may be used to follow the heat changes within a paint system during cure, and how the resultant thermograms could pinpoint endothermic and exothermic reactions. Thermograms were shown illustrating the cure cycles of an epoxy powder coating, clear polyurethane and clear polyester coatings.

The use of DTA in measuring glass transition temperatures (T_g) of paint systems was described. In crosslinking reactions where water is produced as a by-product, it was said that T_g measurements could be used to follow the reaction changes.

In introducing the second part of his lecture, Mr Prosser described a new viscometer developed by the PRA. This viscometer was said to be designed to measure rheology changes within a curing powder coating. It was demonstrated that this new instrument could be used to measure the effect of changes in cure temperature and pigment type on the viscosity of a powder coating and, therefore, their influences on the surface appearance of the final paint film.

The final topic was on the infrared curing of stoving systems. Mr Prosser gave a quantitative review of the mechanism of IR curing. He went on to explain how the energy output of different types of IR lamps varied.

In addition, the influence of film thickness, colour, distance of lamp from paint surface on rate of heating was discussed.

Mr Prosser concluded his lecture by demonstrating how IR curing could be used to reduce stoving schedules, and how these compared with conventional convection oven heating.

After a lengthy and enthusiastic question time, a vote of thanks was given by Mr Hugh Young.

D. V. Maltman

occa meetings

Midlands

Water-dispersible epoxy resins for surface coatings

The final technical meeting of the Midlands Section was held on 18 February 1982 at the County Cricket Ground, Edgbaston, Birmingham, where members and guests heard Mr A. Schnelle of Ems-Chemie AG, Switzerland deliver a paper entitled "Water-dispersible epoxy resins for surface coatings". Because Mr Schnelle's English was not very good, Mr David Lawler read the paper while Mr Schnelle described the slides.

The speaker said that these dispersion systems can be used on both mineral and metal substrates as sealers, primers or topcoats. At present, the hardeners used in these systems are limited in their resistance to chemicals and so do not obtain quite such good results as the organic solvent-based equivalents.

The speaker went on to describe, with the aid of slides, the preparation of these water-based systems and emphasised the care that needs to be taken in the mixing of the two components.

A lively question time followed the talk and the meeting closed with a vote of thanks proposed by Mr G. Gough and endorsed by the audience in the usual manner.

B. E. Myatt

Manchester

Polymers without carbon

Held on 12 February 1982 at the All Saints Lecture Theatre, Manchester Polytechnic. Thirty-five members and guests were present to hear Dr B. R. Currell of the Thames Polytechnic, Woolwich, deliver his lecture.

Dr Currell defined his lecture title as "Materials having an inorganic macromolecular backbone". They could contain carbon but not as a carbon backbone and the actual backbone itself should contain covalent bonding. Examples of materials to be discussed were:

> Antimony oxide, boron nitride, chrysotile asbestos, plastic sulfur, polydimethylsiloxane, molybdenum disulphide, silica (glass materials).

The lecturer then proceeded to elaborate on the above both by classification and example; the information can be summarised thus:

Classification of polymers

Homopolymer – plastic sulfur
Heteropolymer – antimony oxide, sulfur trioxide, polydimethylsiloxane etc

Chain structures

Polydimethylsilane
Polydimethylsiloxane
Polydichlorophosphazine
Sodium polyphosphate – carrol salt

Silicates

Vast majority are polymers (macromolecular)
Calcium silicate
Mica/talc/kaolin
Linear silicates
Trimethylsilination

Silicones

Oils/grease/rubber/resin

Glass

High tensile strength/thermal stability

Phosphate

Glasses
Polyphosphonates

Sulfur

Orthorhombic/monoclinic/polymeric
Concretes – increased tensile strength and chemical resistance compared to OPC based concretes

The lecture was concluded by a discussion period which included the following subjects: phosphate glasses, polyphosphite structures, sulfur concretes, silicon coatings by vapour deposition, polymeric sulfur and vulcanisation and finally, hybrid materials.

The vote of thanks proposed by Frank Courtman was unanimously acknowledged.

Carbon black for paint and printing ink systems

The final student lecture in the 1981/82 programme was held at the Manchester Polytechnic, All Saints Lecture Theatre on Wednesday, 17 February 1982. It was presented by Mr P. Gallagher, field technical service manager, European Special Blacks, Cabot Carbon Ltd, who is a Section member and is based at Stanlow, Cheshire, the site of the UK Manufacturing Plant.

Twenty-five members and guests heard Peter Gallagher commence his lecture by describing the history of carbon black. He related its original use in 1880 for paints, printing inks and lacquers and stated that it was no longer "cheap and nasty" but that black was beautiful and subject to special marketing in relation to motor cars etc. It was interesting to note that Cabot have 12 manufacturing plants in the USA and, in addition to carbon black, manufacture fumed silicon dioxide and energy (sound) absorbing resins (EAR).

A film entitled "Carbon black – king of the pigments" was then shown and was followed by a slide presentation during which Peter illustrated the following points:

1. Full description of the manufacture of furnace blacks, using air/natural gas at 2,000°F with a crude oil feedstock and subsequent water quenching at 400°F
2. Production performance as related to residence time/oil jet angles
3. Gas consumption at Stanlow equal to a town the size of Chester
4. Filtration/pelletisation/drying of carbon black
5. Grades – 90 per cent pelletised for Banbury mixers/rubber industry
   – 100 per cent fluffy for paint and printing ink industries
6. Structure – important to note existence as primary aggregate only and not as individual particles
7. Conductivity (electrical) – properties
8. Surface area – relationship with jetness/tint strength/cost per unit weight
9. Surface area – measurement methods
   nitrogen adsorption most accurate, manufacturing grade variation between 200 m^2/g to a remarkable 1,000 m^2/g
10. Internal surface area – measurement of porosity effect
11. External surface area – iodine or cetyl trimethyl ammonium bromide used due to their size relative to that of the rubber molecule
12. Structure factors and general properties
    – gloss and oil absorption, higher structures → easier dispersion viscosity, loading capacity, oil absorption method using dibutyl phthalate and a Z blade to give a stiff mass end-point.
    Volatile content at 960°F, density
13. Relative costs – £400/tonne for rubber grades, £8000/ tonne for new grades of SA 1,050 m^2/g

A discussion period followed the lecture and the following subjects were brought up:

1. pH values of carbon black usually between 7-8 but acid treatment reduced pH level to 2.5
2. Particle size relative to furnace factors such as velocity, residence time, angle, distance etc
3. Electrical conductivity

The vote of thanks for this interesting lecture was proposed by Ray Stott and supported in the usual way.

F. B. Windsor

Trent Valley

UV and electron beam methods of curing

The fourth technical meeting of the Trent Valley Branch entitled "UV and electron beams: energy efficient methods of curing" was presented by Mr K. O'Hara of Cray Valley Products Ltd on 11 March 1982.

The purpose of the paper was to discuss the advantages to be gained in energy and cost through the use of radiation curable finishes, and also to review the types of polyacrylate monomers available with which to formulate such coatings. Formulation of finishes from these raw materials although similar in many ways to the conventional solvent-based product does require additional factors to be taken into account.

It has been shown that radiation (electron beam (EB) or UV) curing processes offer the coatings user considerable savings in energy consumption when compared with other surface coatings and are the most efficient of all the industrial paint processes studied. Additional advantages of radiation curing are improved product quality and increased product throughout.

Examples of case studies of radiation curing were examined in some depth and included: paper varnishing (book jackets, record sleeves and printed card), printed circuit boards and flush door manufacturing. The resin types of the groups of polyester resins, acrylic prepolymers and monomers or polyacrylate diluents used in the processes were also discussed together with their toxicology and safety.

It was not suggested that either UV or EB curing processes offer a panacea for the coatings user confronted with increasing costs or possible pollution problems. Because of the directional nature of both UV light and high energy electrons both processes are more suited to line fed flat stock rather than complicated shapes. The extremely short cure cycle, lack of heat and other factors make formulation of finishes with good adhesion to bare metal surfaces more of a problem than with solvent-based thermosetting paints. Most of the present applications rely on coating methods such as curtain coating or some form of roller coating. However, the advent of robot spraying equipment might well change this.

Current European usage of resins and polyacrylates for radiation curing is estimated to be about 2,000 tonnes p.a. and it is anticipated that this will grow significantly over the next decade as a wider adoption of existing applications takes place and as more end-users are found for these processes.

Radiation curing does not compete with other recent developments in the coatings industry such as high solids, powder coatings, water-based etc., rather it compliments them. Each does, and will have, its own part to play in future coatings technologies.

After a very lively discussion the vote of thanks was proposed by Mr Stan Watson.

J. C. Ellis

news

Further information on any items mentioned below may be obtained by circling the appropriate *Reader Enquiry Service* number on the form at the back of the *Journal*. Enquiries will be forwarded to the organisation concerned.

Taiwanese paintshop order

Haden Drysys, the paint finishing plant company of Haden Ltd, has received an order worth £0.75m for the supply of a complete paintshop to Hua Tung, Taiwan's first heavy commercial vehicle manufacturer.

The order covers equipment for pre-treatment, priming and topcoat spraying of commercial vehicle cabs, together with all associated air replacement plant and paint shop services.

Hua Tung is owned jointly by General Motors and local Taiwan interests.
Reader Enquiry Service No. 31

Paint additives from Troy

The extensive range of paint additives manufactured by the Troy Chemical Corporation of the USA is now available in the UK from Laporte Industries Ltd.
Reader Enquiry Service No. 32

New test facility opens

DSET Laboratories, Inc. has announced the opening of a new materials weathering laboratory complex in southern Florida. The new facility is known as the Everglades Testing Laboratory, Florida Division of DSET Laboratories, Inc. and is located approximately 10 miles south-west of Homestead, Florida.
Reader Enquiry Service No. 33

new products

New pigments

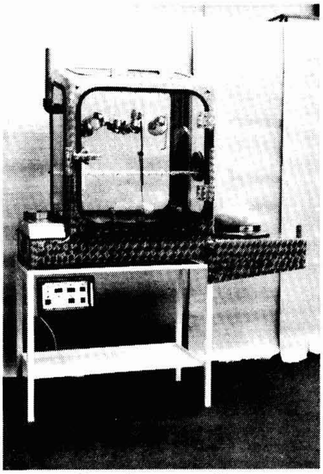
Hoechst has enlarged its Colanyl pigment range with a number of new products.

The first two products belong to the Hansa (R) Yellow series, a class of pigments discovered by Hoechst at the turn of the century, but claimed to be still the most important organic pigments for decorative paints.

Colanyl Yellow 12G is a very strong, bright greenish shade yellow with good light fastness. Although based on Pigment Yellow 3, it is said to be up to 50 per cent stronger than conventional preparations, making it ideal for paints where economy is essential.

Colanyl Brilliant Yellow 5GX is much redder in shade but is also very strong. It is based on Pigment Yellow 74, which, Hoechst says, gives it twice the strength of conventional Hansa Yellow G preparations, with improved light fastness. It is therefore ideal for applications where economies have to be made without sacrificing properties.

Hoechst says the main characteristics of these new Colanyl preparations are their excellent compatibility with a wide variety of emulsion systems, thus avoiding the phenomenon of colour development during application, and a pourable consistency which facilitates easy handling.
Reader Enquiry Service No. 34



Corrosion test cabinet

Corrosion test cabinet

The Kesternich Corrosion Test Cabinet manufactured by H. Kohler KG is now available in the UK through Christison (Scientific Equipment) Ltd.

The cabinet is designed to simulate nine different corrosion test conditions which are covered by or included in 15 different national and international specifications.

The distributor says the stainless steel 300 litre cabinet has been designed to meet any test specification and features automatic ventilation for each test cycle, automatic control, simplified loading, and test solutions need only be added once per day. The cabinet provides both salt spray and sulfur dioxide conditions.
Reader Enquiry Service No. 35

Corrosion inspection and control

Sheen Instruments Ltd is now in the corrosion inspection and control business. The first four instruments in a new range of equipment for measuring anticorrosion coating quality, corrosion rates and general anticorrosion protection were recently unveiled.

Alan Routs, managing director of Sheen Instruments Group, stated:

"As the long established UK market leader in the design and manufacture of testing instruments for the surface coatings and printing inks industries, this is a logical step for us. For some time we have had a growing number of requests from customers for corrosion control and inspection instruments. Our recent acquisitions of Voss Instruments and Startronic Ltd in particular added considerably to our R and D facilities and has enabled us to speed up the development of our new corrosion control range."

Of the new instruments being marketed so far, considerable interest has been aroused by the Positest "banana" gauge for quick, non-destructive measurement of coatings on a magnetic base.

Interest is also being shown in the dc Holiday Detectors, which test for defects in the dielectric type of anticorrosion coatings. This has a comprehensive range of electrodes and other accessories enabling it to be used in virtually any location.

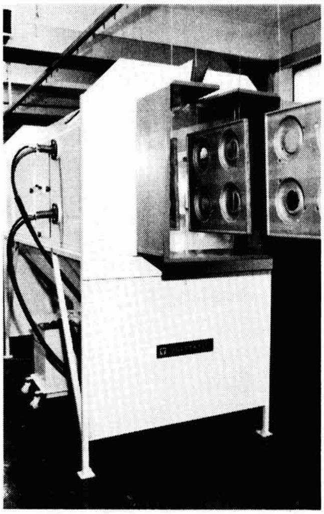
There is also a pin-hole detector for wet testing of coatings which uses low voltage, thus minimising the likelihood of rupturing the film.

Also available is a new digital coating thickness gauge, the SS 1200, which can measure the dry film thickness of paints, plastics, glass and other non-magnetic coatings including zinc on iron and steel.
Reader Enquiry Service No. 36

Chalking tester

Based on the analytical principle of nephelometry and the adhesive tape method, the Helmen Chalkingtester H 100 has been developed by Mr T. Helman in co-operation with G. Zehnter Electronics. It is an optical precision instrument which works independently from the mains. The instrument indicates directly percentage relative chalking values.

Among the many advantages claimed for the Chalkingtester H 100 are that it is much more sensitive to the important onset of chalking, that it is more sensitive with coloured chalking paints than other systems, and that it gives remarkably consistent results.
Reader Enquiry Service No. 37



Volstatic's speedcoater Powder Coating System – a two 4-gun system with integral fluid bed powder feed

New powder coating system

Volstatic Coatings Limited has launched a new fully automatic self-contained electrostatic powder coating system. Called speedcoater, the new system is designed for high speed coating.

Speedcoater can coat any individual component or combination of components that will pass through an opening 750 mm high by 600 mm wide.

The powder is supplied from a fluid bed feed unit that is integral to the system and is applied by four individually charged sprayguns.

The speedcoater will coat up to 10,000 components an hour, depending upon the track speed, the size of the components and the method of jigging.

First time deposition of powder is said to be of a very high order and the bulk of any overspray falls directly back into the fluid bed so that reclaim volumes are considerably reduced and expensive recycling systems are not necessary.
Reader Enquiry Service No. 38

600°C baths and portable balances

Baird & Tatlock has introduced fluidised baths that can be used in the same manner as oil or water baths and that can operate up to 600°C. Under stabilised conditions they are reportedly able to achieve a temperature stability of ±1°C at 550°C.

Also new from the company are the Ohaus Port-O-Gram portable balances. Weighing 1.24 g, the balances are said to be compact enough to fit into a coat pocket. There are two models available; they have capacities of 199.9 g and 1,999 g with combined tare and weighing capacities of 249.9 g and 2,499 g respectively.
Reader Enquiry Service No. 39

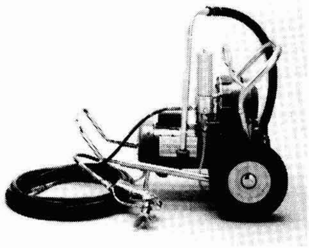
New box oven

Mindon Engineering Ltd has just introduced a new type of box oven for laboratory and process evaluation purposes.

The oven, which has a 27 cubic ft capacity, has been designed to provide accurate temperature control and even heat distribution up to a maximum temperature of 250°C.

Electronic temperature control is fitted as standard with the option of an automatic process timer.

If required the ovens can be supplied with thermocouple pockets in the side walls to enable cables and thermocouples to be placed in the oven whilst maintaining contact with an external source. An insulated glass window can be fitted in the door of the oven if required.
Reader Enquiry Service No. 40



The EM-480 from Graco

New airless sprayer

The EM-480 is a new airless sprayer introduced by Graco which is said to be capable of producing up to 190 bars of pressure for the extra power needed to spray heavy coatings; it delivers up to 1.7 litres/minute.

The unit comes ready to spray. It includes a fluid supply hose with inlet screen, a pressure controller, an on/off switch, an outlet manifold with 60 mesh filter, 15 metres of high pressure fluid hose, an extra flexible whip-end for easy gun movement, a Graco airless spray gun and a Reverse-A-Quick self cleaning spray nozzle with a tip choice from 0.011 to 0.021 inch. The unit is powered by a powerful electric motor (500W/0.75 HP) which is totally enclosed and fan cooled with thermal overload protection and automatic reset switch. The motor drives a paint pump which Graco says can handle all types of paints.
Reader Enquiry Service No. 41

news

Paint additives

Aynchem Limited is now producing a range of three new paint additives to be marketed under the trade name Aynaid. They are formulated to improve such characteristics as slip, scratch and abrasion resistance, reflectance and smoothness, reduction of orange peeling and cratering, and receptivity to re-coating.
Reader Enquiry Service No. 42

literature

Silicas and silicates

An illustrated booklet just issued by the Applied Silicas Division of Joseph Crosfield & Sons Limited gives comprehensive details of the company's range of precipitated silicas and silicates.
Reader Enquiry Service No. 43

Boric acid in dye manufacture

Borax has issued a booklet on "The use of boric acid in the manufacture of dyestuffs" by Dr A. J. H. Mercer. The 26 page publication details numerous mechanisms and synthetic routes showing the type of reactions and some of the dyes involved when boric acid is used in dye manufacture. The booklet has a list of references and is free upon request or by appropriately filling in the Reader Enquiry Service form at the back of the *Journal.*
Reader Enquiry Service No. 44

Products directory

The revised and improved second edition of the Paint RA's "Paint and Pretreatment Products Directory" (2nd edition) brings readers up to date with new products and the book as a whole profits from the experience of users of the first edition.

Considerable effort has been made to list the products of UK paint companies and overseas paint manufacturers trading in the UK.

For convenient reference, over 3,000 trade names have been grouped alpha-

news

betically in 12 main sections, each covering a product area (e.g. building surfaces, corrosion prevention, metal finishing).

The directory caters, through quick reference sub-sections, for those wishing to trace a given trade name (and its type) and for those seeking a list of all UK products suitable for a particular end-use.

Although not a textbook of paint technology, prefaces to each main section outline the types of products available and give guidance as to their use. Names, addresses and telephone numbers of the manufacturers together with useful contacts are given, cost £20 (£15 to Paint RA members).
Reader Enquiry Service No. 45

people


R. Chester

Mr Rex Chester, chairman of Donald Macpherson Group plc, has been elected the new President of the Paintmakers Association of Great Britain.

Mr Chester has been closely involved with the Paintmakers Association as a Council member since its inception, then as inaugural Chairman of the Statistical Committee and now as Chairman of the Large Manufacturers Group. He has also represented the Association on the Council of the Paint Research Association.

Mr Chester has been chairman of Donald Macpherson Group since 1968, and has spent all his business life within the Group. They are one of the leading suppliers of industrial coatings to manufacturing industry, suppliers of fixtures and fittings to the furniture industry and DIY trade and, of course, are well known for their Cover Plus range of paints sold exclusively through Woolworth.

Three new directors have been appointed to the board of International Paint-Industrial Coatings: **Mr R. N. Doidge-Harrison** is director responsible for Packaging and Coil Coatings, **Mr C. D. Melia** is director responsible for Industrial Paints, and **Mr D. Shaw** is appointed manufacturing director.

occa news

For information on membership of OCCA, enquiries should be sent to the Association's offices, see front cover for address.

News of members

Mr K. Stoodley


K. Stoodley

R. J. Hamer Ltd has announced that Mr Keith Stoodley, a member of OCCA's Professional Grade, has been appointed to the post of technical director.

After initial experience with Berger Paints, Mr Stoodley spent many years in a technical capacity with the Mcpherson Group, ending as technical director of Porter Paints Ltd. He joined R. J. Hamer Ltd late in 1981 and brings valuable extra experience that will help the company's planned expansion.

Mr Sidney Wyatt continues in his existing post of works director.

A. McLean

Mr A. McLean (President 1977-79) took up an appointment from 1 April 1982 as technical director of Cruachan Paint Co. Ltd, Canal Street, Kirkintilloch, Glasgow.

Jordan Award

The Jordan Award Committee now invites applications for the seventh award of £100. The rules of the Award are:

1. The Award will be made for the best contribution to the science or technology of surface coatings by a member of the Association of any nationality working in either the academic or industrial field who is under the age of 35 at the date of application.

2. The final date for submission of applications will on this occasion be 31 December 1982 and it is hoped to present the award at the Association's Conference in the following year.

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

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

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

occa news

Norman Cochrane (left) receiving the Association's Commendation Award for services to the Association from the President (right) (Mr D. J. Morris) at the West Riding Section's 23rd annual dinner dance held on 27 November 1981.

Midlands Section

A super gourmet evening

The annual Newton Friend meeting of the Midlands Section was held this year at the Halesowen College of Further Education, Whittingham Road, Halesowen.

Members, guests and their ladies were treated to a "Super Gourmet Evening" prepared and served by the catering students at the college.

The evening commenced with a brief description given by one of the students of the preparation and content of each dish. The audiece was then left to enjoy each course as it was served and it was unanimously agreed that it was an excellent meal. The evening closed with a vote of thanks to the students and staff of the college for the high standard of preparation and service that had been achieved. This was warmly endorsed by the audience in the usual way.

B.E.M.

Thames Valley Section

Annual dinner dance – 12 February 1982

The Thames Valley Section held its annual dinner dance at the traditional venue of Great Fosters, Egham. This year, as in previous years, the Great Hall provided its magnificent setting for a delightful evening. The number of people attending was such that the hall was comfortable without being overcrowded. We would take this opportunity of thanking all the people who supported the Thames Valley Section at its economically priced evening, and thank them for making the evening so successful.

The Section Chairman welcomed, as usual, over 100 guests, and was particularly pleased to welcome Mr Hamblin and chairmen from other sections.

Great Fosters again provided an excellent dinner which was followed by a cabaret from Partners in Time, barber shop singers. Dancing was to the Keynotes band who provided a range of music to suit all tastes.

The evening drew to a pleasant close at 1.00 a.m.

A.W.F.

Manchester Section

Informal buffet dance

The Royal Air Force Association Club, Sale was the venue on Friday 12 March for the 1982 informal buffet dance. A grand total of 173 Section members and their guests commenced arriving at 8.00 p.m. by transport which included three coaches having commenced their journeys in foreign parts such as Bury, Bolton and Blackburn.

This year's dance was performed to the music generated by the Timmy Gee Disco, and though a "one man band" his enthusiasm and sense of humour ensured maximum occupancy on the dance floor coupled with the reward of spot prizes.

After an interval occasioned by the serving of an excellent buffet, the dancing recommenced only to be interrupted by the now annual fund raising event for the Wings Appeal achieved by target practice on a bottle of the hard stuff with 10p coinage; approximately £40 was raised and then dancing again until 12.30 a.m. and the journey home.

This dance, generally agreed to be the best to date, was organised by the plus two partnership of Arthur Thornhill and David Clayton to whom the crowning glory is awarded.

F.B.W.

Shown at the Thames Valley dinner dance are (from left to right) Mr F. B. Redman (Chairman, Manchester Section), Mrs Redman, Mr G. Fowkes (Chairman, Bristol Section), Mrs Fowkes, Mr B. F. Gilliam (Chairman, London Section), Mrs Gilliam, Mr V. Moore (Chairman, Thames Valley Section), Mrs Moore, Mr R. A. C. Chappell (Chairman, West Riding Section), Mrs Chappell, Mrs Hyde, Mr N. Hyde (Hon. Social Secretary, Thames Valley Section), Mr R. H. Hamblin (Director & Secretary)

occa news

Eastern Branch

Annual Burns Supper

The annual Burns Supper organised by the Eastern Branch was held in the Commodore Hotel, Edinburgh, on Friday 12 February 1982. The increasing popularity of this event resulted in an attendance of some 68 members and guests. The traditional six course meal was preceded by Selkirk Grace given by Mr A. McKendrick. The Address to the Haggis was rendered by Mr A. Hamilton who must rank as one of the best exponents of this work in Scotland. Following the meal the toast to the Queen was proposed by the Chairman, Mr T. McMahon, and the entertainment commenced.

Mr P. Whitelaw proposed the Immortal Memory and greatly honoured the Eastern Branch by selecting this occasion as his last public appearance in this role after many years associated with Burns. A selection of Burns' songs was given by Mr A. Hamilton accompanied on piano by Mr M. Temple. Mr H. Ward gave a beautiful rendering of Holy Willie's Prayer and later in the evening Tam O'Shanter. Mr J. Toovey proposed the toast to the lassies. Pipers Mr G. Langlands and Mr C. McPherson ably entertained the party. Thanks to all the guests and artists were proposed by Mr G. H. Hutchinson, and a short congratulatory speech was given by Scottish Chairman Mr Tom Humphrey. The formal part of the evening concluded with the singing of Auld Lang Syne.

The party continued to the small hours of the morning. To those who have not yet attended such a function, it is suggested that they take a chance to sample traditional Scottish song and poetry together with marvellous hospitality in attending next year's supper. Thanks are due to the Eastern Branch Committee who work so hard to make this event the success it is. It is perhaps appropriate to conclude in the following manner.

"This nicht wi' Burns will mak' us fitter, to bear our load and wiser, kindlier, maybe better we'll tak' the road."

J.H.C.

Shown at the Newcastle Section's dinner dance held on 16 February 1982 from left to right are: Mr R. H. Hamblin (Director & Secretary), Mr C. N. Finlay (Vice President), Mr H. Fuller (Hon. Social Secretary, Newcastle Section), Mr D. J. Morris (President), Mr E. A. Watson (Chairman, Newcastle Section), Mr I. B. Bolam (Vice Chairman, Newcastle Section), Mr R. A. C. Chappell (Chairman, West Riding Section), Mr V. A. Moore (Chairman, Thames Valley Section), Mr F. Hellens (past Chairman, Newcastle Section), Mr J. Fowles-Smith (Chairman, Trent Valley Branch), Mrs Finlay, Mrs Fuller, Mrs Morris, Mrs Watson, Mrs Bolam, Mrs Chappell, Mrs Moore, Mrs Hellens, Mrs Fowles-Smith.

new members

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

Ordinary Members

BLUE, ALISTAIR, BSc, MIMF, Fry's Metals (Graphics) Ltd, Avis Way, Newhaven, East Sussex *(London)*

BRIGGS, JOHN, 6 Drumonyne Close, East Herrington, Sunderland, Tyne & Wear *(Newcastle)*

CAMPBELL, GRAEME GEORGE, BSc, Shell Chemicals NZ Ltd, PO Box 2091, Wellington, New Zealand *(Auckland)*

DENT, GRAHAM JOHN, LRSC, DMS, 56 Harwood Lane, Harwood, Bolton BL2 3QX *(Manchester)*

FINDLAY, ANDREW CHARLES, 32/34 Elliot Street, Glasgow G3 8EA *(Scottish)*

FURTADO, STEPHEN EDGAR JOSEPH, BSc, PhD, MIB, 22 Highworth Drive, Springwell Village, Gateshead, Tyne & Wear *(Newcastle)*

JOAV, ALBERTO ROBBIN, Box 65, Strand, Republic of South Africa *(Cape)*

LIBICEK, BOLESLAV, c/o Mrs M. E. Barker, Huletts Aluminium Ltd, PO Box 74, Pietermaritzburg 3200, Republic of South Africa *(Natal)*

MAN, TAK-MING, Institute of Textiles & Clothing, Hong Kong Polytechnic, Kowloon, Hong Kong *(General Overseas)*

PYE, ROBERT, 5 Athol Park, Sunderland, Tyne & Wear *(Newcastle)*

SHARMA, KIRAN BALA, 39 Chaucer Avenue, Cranford, Hounslow, Middlesex TW4 6NA *(London)*

Associate Members

AFZAL, MIAN KAMRAN, Evershine Paints (Eastern) Ltd, D/24 South Avenue, SITE, Karachi, Pakistan *(General Overseas)*

COHEN, RACHELLE ESTHER, 407 Port Sdy, Beach Road, Mouille Point, 8001, Republic of South Africa *(Cape)*

Registered Students

CHAN, MAN SHUN, 55 Ash Grove, Bradford, West Yorkshire BD7 *(West Riding)*

DRAKE, GARRY, W W Hill Son & Wallace, Elton Street, Cambridge Industrial Estate, Salford, Lancashire *(Manchester)*

Dynamit Nobel
CHEMICALS

DYNAPOL® L
and lacquers stay in brilliant shape

DYNAPOL® L

A range of high molecular, linear, saturated polyesters, based on terephthalic acid. DYNAPOL L lacquer films have extremely high adhesive strength; they are highly elastic, scratch-resistant and impact-resistant, and they do not yellow with age. Can be stamped and deep-drawn, even under extreme loads. Also tasteless and completely safe in terms of foodstuffs legislation (BGA and FDA). Suitable for use as external and internal lacquer on packagings, and for sterilizable applications.

DYNAPOL L is available with different elasticity and viscosity properties as DYNAPOL L 205, L 206, L 411 and L 850. Main applications: roll-coating on sheet and strip, decorative lacquers for packagings, internal protective lacquers for packagings, coilcoating primers and top-coats, foil lacquers.

DYNAPOL® LH

A range of linear, saturated polyester resins containing hydroxyl groups. For use in combination with cross-linking resins to obtain top-quality, full-bodied industrial stoving finishes. DYNAPOL LH lacquer coatings are flexible, have good deformation properties and excellent adhesion to a metal substrate, such as aluminium, steel or galvanized steel. Good surface hardness and resistance to weathering and chemicals.

Especially suitable for outdoor use particularly in the building industry.

Also suitable for use on packaging materials.

DYNAPOL LH 812 and LH 818 are fully compatible with the high molecular polyesters of the DYNAPOL L range. Their properties may be modified and rendered suitable for specific applications by combined use of DYNAPOL L/LH grades. Main applications: coilcoating lacquers, decorative lacquers for packagings; also suitable for UV printing methods.

DYNAPOL polyesters at a glance:

DYNAPOL	L 205	L 206	L 411	L 850	LH 818	LH 812
Form supplied	Granules	Granules	Granules	Granules	Solution	Solution
Hydroxyl No.	10	5–10	5–10	5–10	15	35
Molecular weight	15000	18000	16000	14000	7000	3000
Glass temperature	70 °C	70 °C	50 °C	40 °C	40 °C	30 °C

Further products in development.

DYNAPOL® L
DYNAPOL® LH

Our range of products also includes:

DYNAPOL® H
Branched polyester containing hydroxyl groups

DYFLOR® 2000
Polyvinylidene fluoride for EPS and WS coatings

DYNASIL®
Silicic acid ester

DYNATRI® LQ
Specially stabilized trichloroethylene

RHENOFLEX®
Chlorinated polyvinylchloride

ICDAL® TE/TI
Electrical insulating lacquer resins

WITAMOL®
Plasticizers, phthalic acid ester

WITACLOR®
Plasticizers, chloroparaffin

® = registered trade mark

Name: ______________________

Company: ______________________

Address: ______________________

____

Tel.: ______________________

Please send me special, detailed information on basic and ancillary materials for surface protection. I am particularly interested in:

____

____

____

E A 1 2 3 4 5 6 7 8
B 1 2 3 4 5 6 7 8

Dynamit Nobel
Aktiengesellschaft
Sparte Chemikalien/M5

D-5210 Troisdorf

Federal Republic
of Germany

At your disposal in Great Britain:
Dynamit Nobel (U.K.) Ltd.
302/308 High Street
GB-Slough (Berks.) SL 1 1 HF
Tel.: 0753/71851
Tx.: 848 243

Dynamit Nobel Aktiengesellschaft
GB Kunststoff-Rohstoffe/Fette-LK
D-5210 Troisdorf
Federal Republic of Germany

Dynamit Nobel
CHEMICALS

Printed on MEDIAPRINT 170 g/qm –
a product of Feldmühle

LK 118/5. 81/60.000

CLASSIFIED ADVERTISEMENTS

Classified Advertisements are charged at the rate of £5.00 per single column cm. Advertisements for Appointments Wanted are charged at £1.50 per line. A box number is charged at £1.00. They should be sent to D. N. Buddles, Assistant Editor, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF. JOCCA is published EVERY month and Classified Advertisements can be accepted up to at least the 12th, and in exceptional circumstances the 20th of the month preceding publication. Advertisers who wish to arrange for an extension of the copy deadline should contact the Assistant Editor, D. N. Buddles at the address given above (telephone 01-908 1086, telex 922670 OCCA G).

APPOINTMENTS

SITA

Mander Kidd (S.A.) Pty Ltd. Cape Town

Paste Inks Product Manager

This is an excellent appointment for a technologist with a flair for management and customer liaison.

The person to be appointed will have had at least 10 years' relevant ink experience.

An excellent salary commensurate with age, experience and qualifications will be negotiated. Fringe benefits include use of company car, life assurance, pension scheme and medical aid. Generous relocation expenses.

Candidates should write or 'phone in absolute confidence to:

G. P. Birtles, SITA Ltd.,
13th Floor, Crown House, Morden, Surrey.
Tel: 01-542 4213 or 01-543 2378.

BACK ISSUES ON OFFER

Journal Back-issues on Offer

A complete set of *JOCCA* dating from 1965, a number of *Science News* issued by *Penguine Book Co*, New Scientists, and a "Lecture Series" issued by the *Royal Institute of Chemistry* are offered to anyone who may be interested. *Please contact:*

Mrs Y. Wiseman
61 Casimir Road
London E5 9NU

SITUATIONS WANTED

Permanent or Temporary Employment sought

Due to early retirement, member with 27 years experience of pigment evaluation is available for temporary or permanent employment.

Replies in confidence to Box No. 509

SITUATIONS WANTED

Member with wide experience seeks challenging position

Member of OCCA (in the Professional Grade) with extensive experience in the surface coatings industry: R & D, technical service, market R & D, marketing and sales, advertising and PR. Also, experienced technical writer, general lecturer, book contributor and author of many papers. Seeks an executive sales/marketing or technical appointment; positions in general management, admin., PR, teaching or scientific journalism would be eagerly considered.

Replies in strictest confidence to Box No. 511.

Part-time Consultancy

Recently retired CBE, PhD with wide experience in the UK and abroad in company management and selling seeks part-time consultancy.

Replies in confidence to Box No. 510

JOCCA

READER ENQUIRY SERVICE – JUNE 1982

For further information circle items listed below:

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40
41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80
81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100
101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120
121	122	123	124	125	126	127	128	129	130	131	132	133	134	135	136	137	138	139	140
141	142	143	144	145	146	147	148	149	150	151	152	153	154	155	156	157	158	159	160
161	162	163	164	165	166	167	168	169	170	171	172	173	174	175	176	177	178	179	180
181	182	183	184	185	186	187	188	189	190	191	192	193	194	195	196	197	198	199	200

Please also send information on the following items relating to the above:

. . . .

Name . . . .

Job Title . . . .

Company . . . .

Address . . . .

. . . .

. . . .

Country . . . . Telephone . . . .

Photocopy or cut out this form and send to:

Journal of the Oil and Colour Chemists' Association

Priory House, 967 Harrow Road, Wembley, Middx., HA0 2SF, England
Telephone: 01-908 1086 Telex: 922670 (OCCA G)

AT ANY TIME DAY OR NIGHT
TELEX YOUR CLASSIFIED FOR JOCCA
Telex 922670 (OCCA G)

INDEX TO ADVERTISERS

Further information on any of the products advertised in this *Journal* may be obtained by circling the appropriate number on the *Reader Enquiry Service (RES)* form. The numbers are shown in brackets below. Enquiries will be forwarded to the organisations concerned.

A

Atlas Electric Devices Co. . . . . (192) ii

C

Crown Decorative Products Ltd v

D

Dynamit Nobel AG . . . . (193) Insert

H

Hardman, E., Son & Co. Ltd . . . . (172) i

M

Marine Finishes vi
Manville . . . . (194) viii
MSE Scientific Instruments Ltd . . . . . (167) Cover

O

OBS Machines Ltd . . . . (195) vii
OCCA Australia . . . . (129) vi
OCCA Conference . . . . (199) Cover

P

Paint Research Association . . . . (110) ii

Q

Q-Panel Co. . . . . (169) i

S

Sachtleben Chemie GmbH . . . . (116) Cover
Sanyo-Kokusaku Pulp Co. Ltd . . . . . . (112) ii
Sub-Tropical Testing Service Inc. . . . (117) vii

Reader Enquiry Service No. 116

VISCOSITY
Tough questions. Simple answers.

Is there a cost effective, easy to operate viscometer capable of producing high quality results in all fields of product development and quality control work?

Is there a high performance viscometer that will give results over a very wide shear rate range i.e. 50,000:1?

Is there a viscometer that can replace most common instrument types - cone and plate, concentric cylinder, immersion - simply by changing sensors?

Is there a viscometer that can be extended from direct operator control, to external programming, with automatic data analysis and recording?

THE ANSWER

HAAKE ROTOVISCO RV12 VISCOMETER.

Which viscometer control unit is so advanced that it incorporates control electronics, shear rate programmer and x-y-recorder for recording flow curves automatically?

Which viscometer is specifically designe for ease of operation - with features like an automatically calibrated x-y recorder, self testing functions and protection against overload or operator error?

Which viscometer performs routine measurements at the touch of a button?

THE ANSWER

HAAKE ROTOVISCO RV100 VISCOMETER.

How can you find out more about Haake viscometers and regional seminars on viscosity measurement techniques?

FOR ALL THE ANSWERS

Contact MSE today. Please send me further details of Haake viscometers ☐
Please send me information about seminars ☐
Name__________
Company__________
Address__________

MSE Making Science More Effective
MSE Scientific Instruments,
Manor Royal, Crawley, West Sussex, England. Telephone: Crawley (0293) 31100. Telex: 87119.
A member of FISONS Scientific Equipment Division

Reader Enquiry Service No. 167

Printed by Campbell Graphics Ltd, Newcastle upon Tyne NE6 1AS and Suite 108, Temple Chambers, Temple Avenue, London EC4. Tel. 01-583 3190

-7 กย