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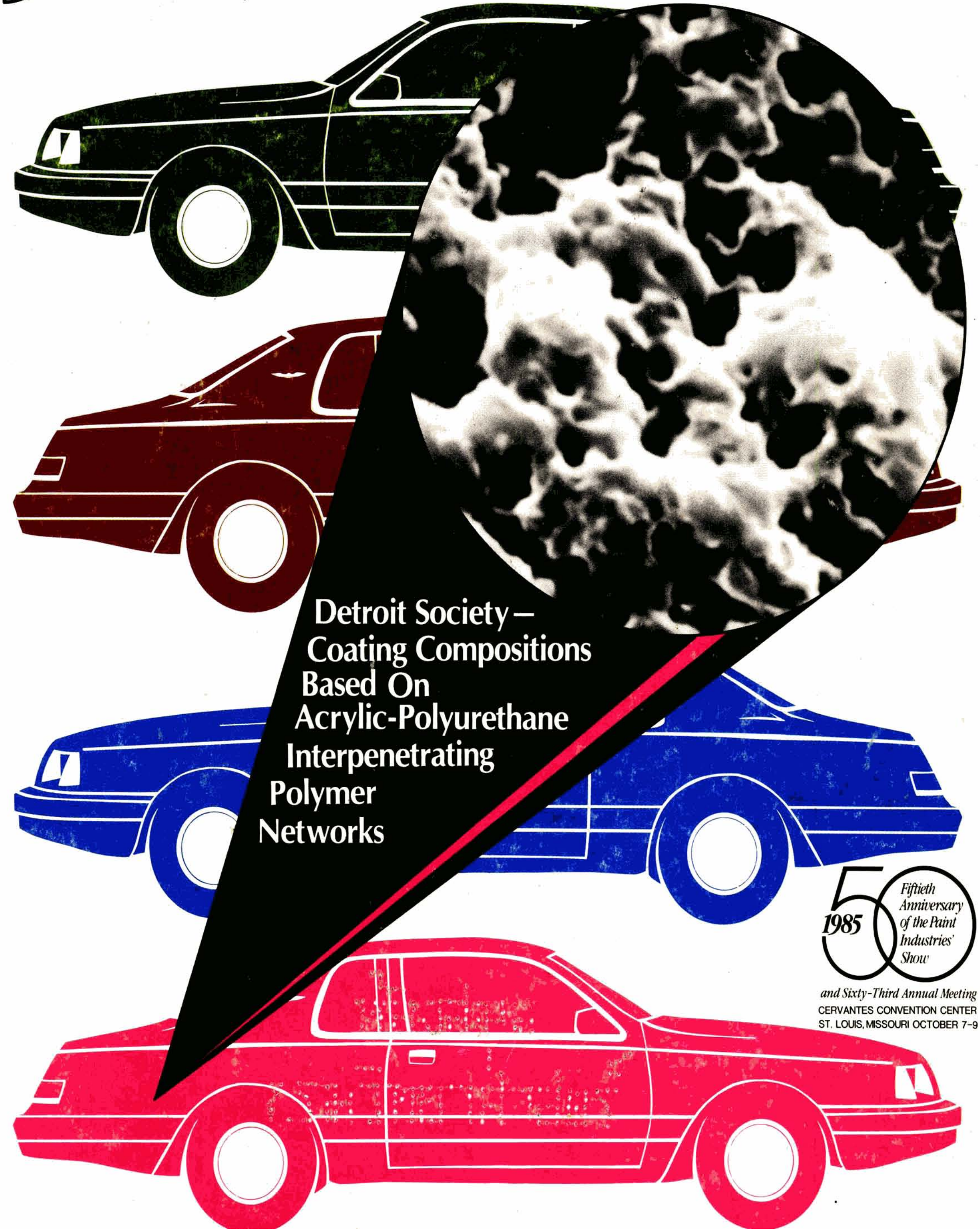
April 1985

jct JOURNAL OF COATINGS TECHNOLOGY



Detroit Society—Coating Compositions Based On Acrylic-Polyurethane Interpenetrating Polymer Networks

1985 50 *Fiftieth Anniversary of the Paint Industries' Show*

and Sixty-Third Annual Meeting
CERVANTES CONVENTION CENTER
ST. LOUIS, MISSOURI OCTOBER 7-9

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VOLUME 57 NUMBER 723

Technical Articles

Open Forum

Federation News

Departments

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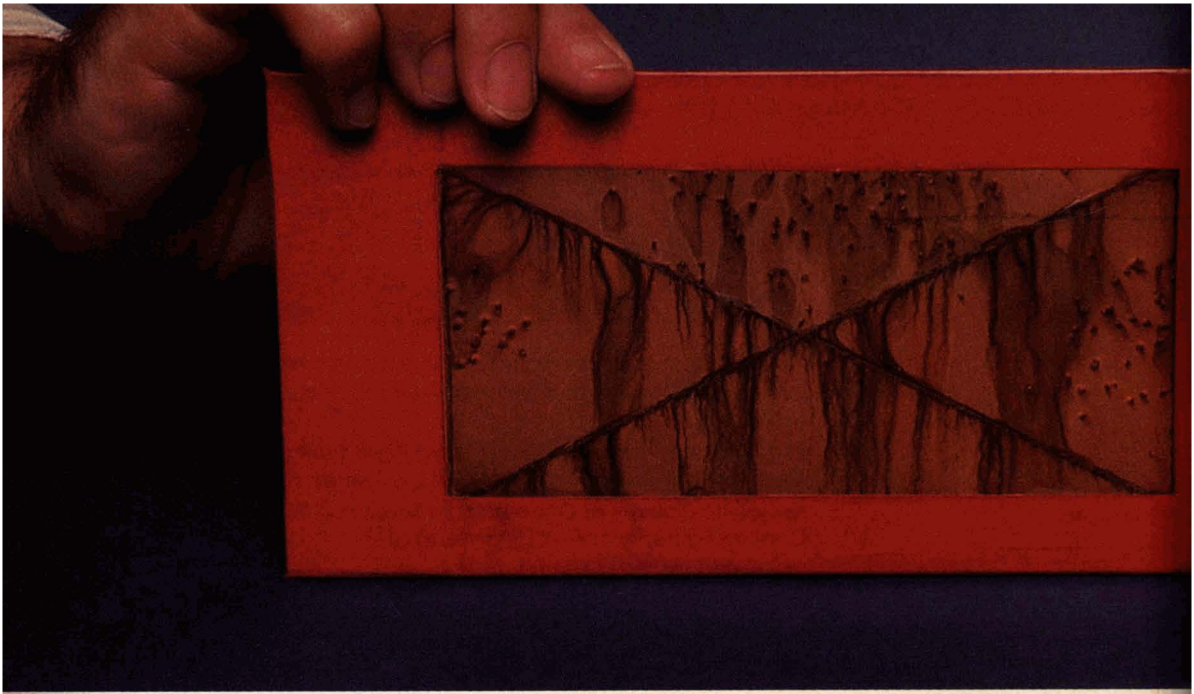
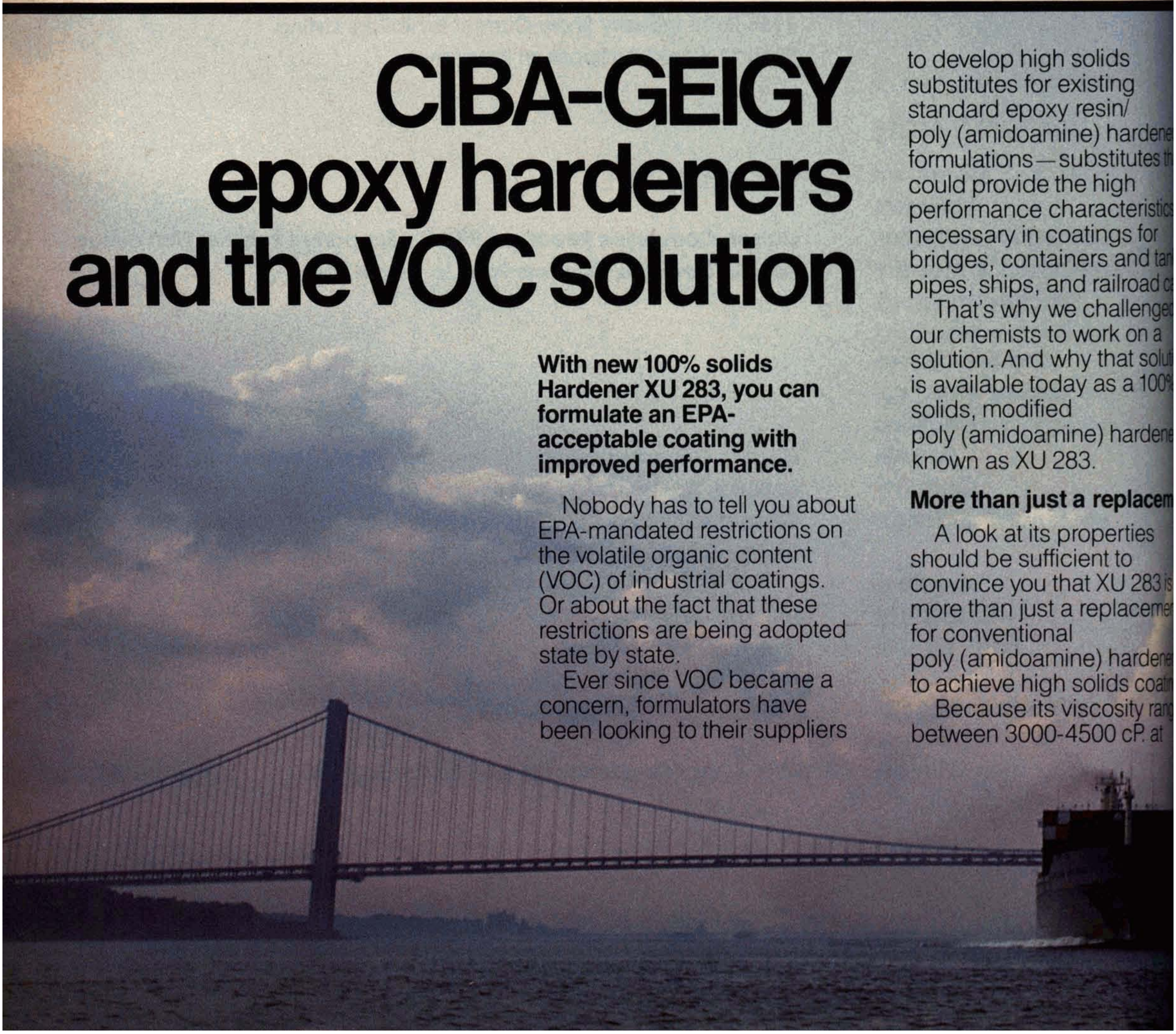




CIBA-GEIGY epoxy hardeners and the VOC solution

With new 100% solids Hardener XU 283, you can formulate an EPA-acceptable coating with improved performance.

Nobody has to tell you about EPA-mandated restrictions on the volatile organic content (VOC) of industrial coatings. Or about the fact that these restrictions are being adopted state by state.

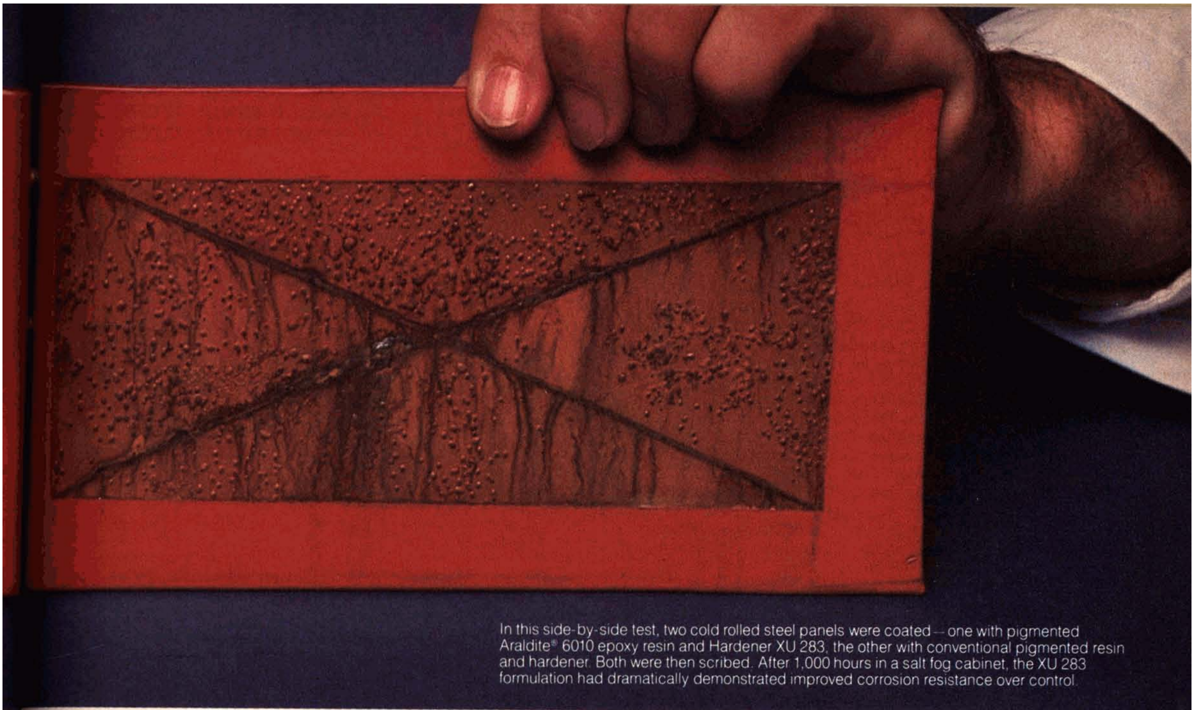
Ever since VOC became a concern, formulators have been looking to their suppliers to develop high solids substitutes for existing standard epoxy resin/ poly (amidoamine) hardene formulations—substitutes th could provide the high performance characteristics necessary in coatings for bridges, containers and tan pipes, ships, and railroad c

That's why we challenged our chemists to work on a solution. And why that solu is available today as a 100% solids, modified poly (amidoamine) hardene known as XU 283.

More than just a replacem

A look at its properties should be sufficient to convince you that XU 283 is more than just a replaceme for conventional poly (amidoamine) hardene to achieve high solids coati

Because its viscosity rang between 3000-4500 cP. at



In this side-by-side test, two cold rolled steel panels were coated—one with pigmented Araldite® 6010 epoxy resin and Hardener XU 283, the other with conventional pigmented resin and hardener. Both were then scribed. After 1,000 hours in a salt fog cabinet, the XU 283 formulation had dramatically demonstrated improved corrosion resistance over control.

25°C, XU 283 requires gnificantly less solvent than tandard poly (amidoamine) ardeners to reach a workable stage. Because XU 283 is ompatible with solvent-free quid epoxy resins such as our raldite® 6010 epoxy resin, no duction period is necessary and, consequently, coatings can be applied immediately after mixing. In addition, XU 283 also resists blushing and exudation.

But what about end results? In the final analysis, how does XU 283 compare to onventional products? The fact is, crosslinked with Araldite 6010 resin, XU 283 actually improves the performance and application of coatings formulations. Compared to formulations using unacceptably high solvent levels, XU 283 produces harder film; exhibits better chemical (see chart), corrosion and abrasion resistance; and can provide excellent mechanical properties, especially adhesion. Add all this to the high solids content of XU 283 and its elimination of sweat-in time, and you can understand why initial response from formulators has been overwhelmingly enthusiastic.

Chemical resistance

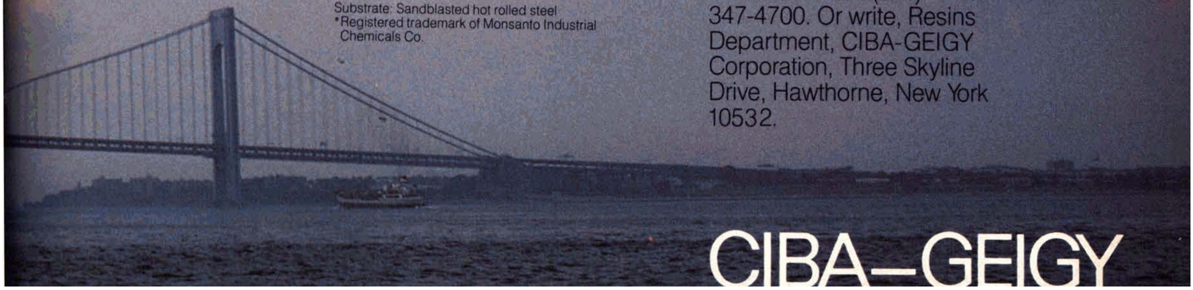
Immersion tests
12-16 mils; Cured 7-10 days @ R.T.

Formulation	1	2
	Parts by weight	
Araldite® 6010	100	—
XU 283	70	—
"1" Type Epoxy Resin	—	100
Conventional Polyamide	—	54
	Weeks to failure (1 Year Test)	
Tap water	>52	8
Deionized water	>52	8
Sea water	>52	>52
50% NaOH	>52	>52
10% NH_4OH	>52	1
10% H_2SO_4	>52	2
10% HCL	>52	1
Unleaded gas	>52	>52
Diesel fuel	>52	>52
Skydrol® 500B*	>52	8
Heptane	>52	>52
Xylene	>52	16
MEK	12	1
Isopropanol	>52	26
50% Ethanol/H_2O	>52	8

Note >52=Unaffected after 1 year.
Substrate: Sandblasted hot rolled steel
*Registered trademark of Monsanto Industrial Chemicals Co.

Technology and service.

Facing up to EPA-mandated restrictions on VOC is an important challenge to the coatings industry. But it's a challenge we're prepared to help you resolve. Call the CIBA-GEIGY Resins Department—your source for high technology specialty resins and hardeners, and responsive service—at (800) 431-1900. Ask for Rob Crespi. In New York call (914) 347-4700. Or write, Resins Department, CIBA-GEIGY Corporation, Three Skyline Drive, Hawthorne, New York 10532.



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1315 Walnut St., Phila., PA 19107

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Comment

Play Ball!!!

The long winter is finally over and we have reached that time of the year when life seeks again to reestablish itself in the order of things. Among the many signs of spring in the northern hemisphere, from Osaka, Japan to Boston, Massachusetts, is the annual "Rite of Spring"—baseball, turning the fans' minds from remembrances of snow and ice towards the possibility of a championship season.

In the coatings industry, spring is also the season of meetings. Seminars, symposia, conferences, and conventions, like the robin appear early. The Federation is especially pleased to sponsor several gatherings worthy of your consideration. These are (in order of appearance) the Symposium on Color and Appearance Instrumentation (SCAI), being held this month in Pittsburgh; the Seminar on Recent Developments in Home Painting, May 14–15 in Baltimore; and the 62nd Annual Meeting and 50th Anniversary Paint Industries' Show, October 7–9 in St. Louis.

We are looking forward to a successful SCAI and we hope you have made your reservations early since attendance is limited. This month's issue of the JCT features program and registration information for the Recent Developments in Home Painting Seminar (see page 14). Again, registration is limited, so late-comers may be disappointed. Finally, the May JCT will feature registration and housing information for the 1985 Annual Meeting. This will be one of the biggest events ever held by the Federation (a record number of exhibitors have already signed up for the Paint Show), and the Program Committee, chaired by Joe Vasta of DuPont, is preparing an agenda of presentations to match.

We invite you to join the Federation team and be part of the best triple play combination of the year.

Robert F. Ziegler

Robert F. Ziegler,

Editor

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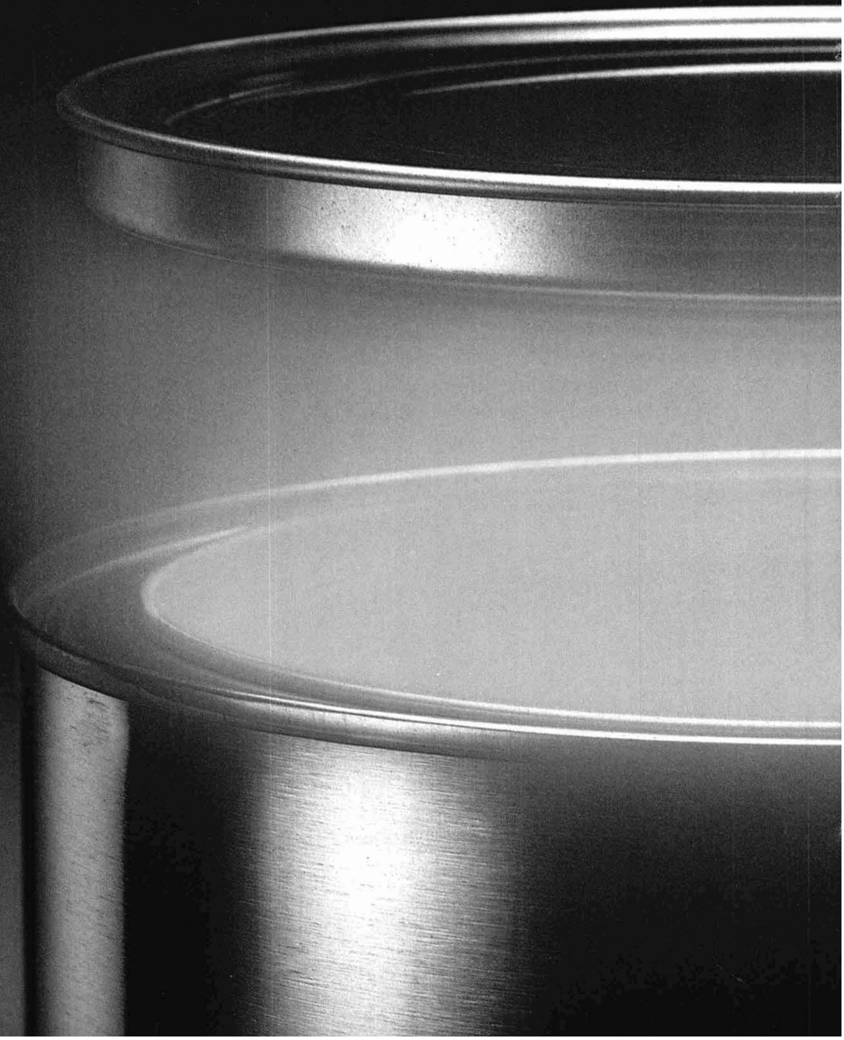
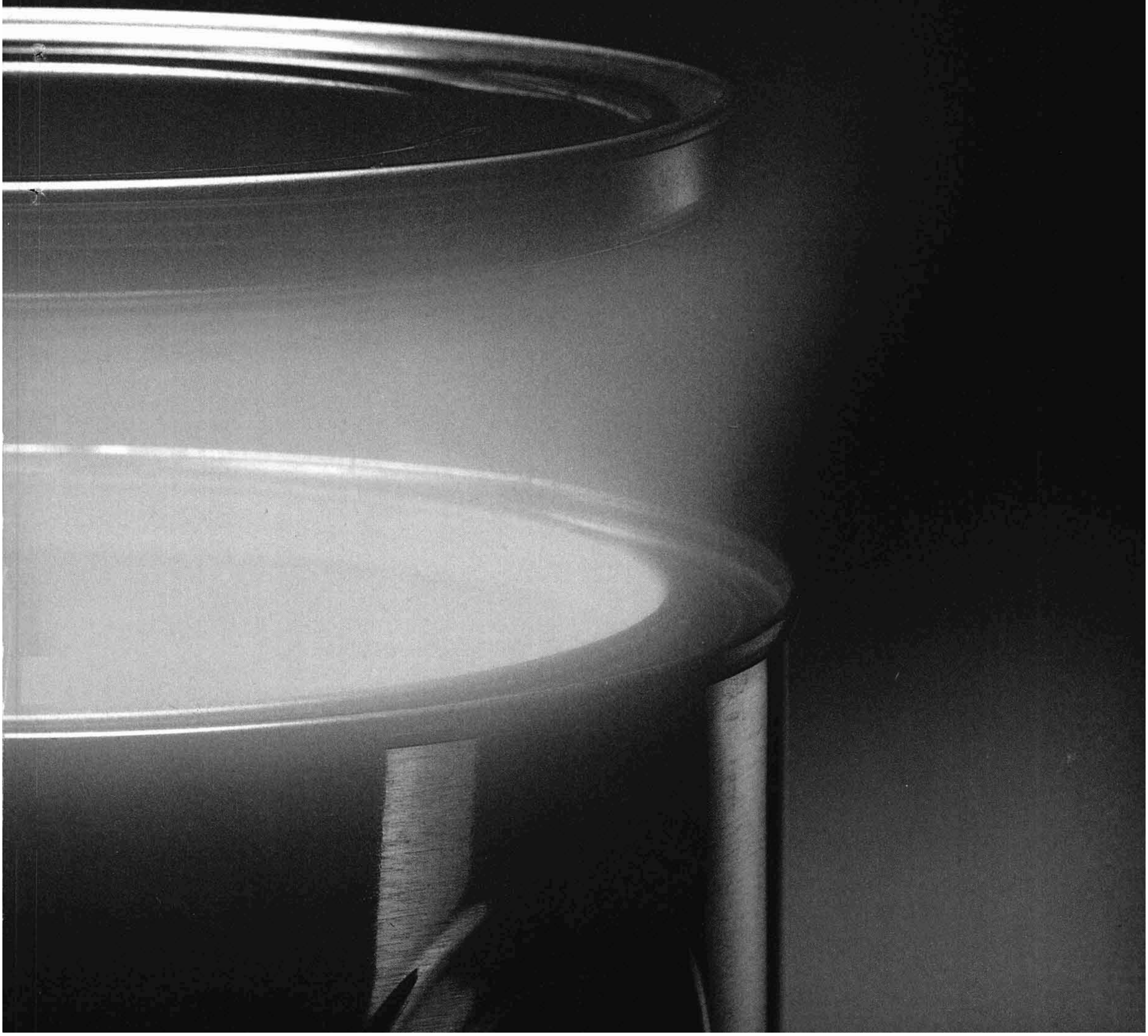




Abstracts of Papers in This Issue

COATING COMPOSITIONS BASED ON ACRYLIC-POLYURETHANE INTERPENETRATING POLYMER NETWORKS—Detroit Society for Coatings Technology

Journal of Coatings Technology, 57, No. 723, 23 (Apr. 1985)

Interpenetrating polymer networks were synthesized from a two-component polyurethane and an unsaturated urethane modified acrylic copolymer. The two-component polyurethane was prepared from hydroxyethylacrylate butyl methacrylate copolymer and cured with an aliphatic polyisocyanate. The unsaturated acrylic copolymer was made from the same hydroxy-functional acrylic copolymer modified with isocyanatoethyl methacrylate (IEM).

Simultaneous IPN's were prepared from the above two polymer systems at various ratios. The IPN systems were characterized by means of mechanical properties and morphology.

REACTIONS OF N-BUTANOL WITH AN ALKYD RESIN DURING HEAT-AGING: AN ANALYTICAL STUDY—J.J. Engel and T.J. Byerley

Journal of Coatings Technology, 57, No. 723, 29 (Apr. 1985)

Water-soluble and high-solids alkyds and polyesters are commonly sold in strong, oxygenated solvents. Primary alcohols have been used, but films with slow dry and poor water resistance resulted from unwanted reaction between the alcohol and the resin.

The purpose of this work was to identify the products formed by the reaction of n-butanol with an alkyd resin during heat-aging. An alkyd resin was dissolved in n-butanol and stored at 185°F for two weeks. The volatiles were removed by vacuum distillation and analyzed by gas chromatography. The resin was fractionated by gel permeation chromatography and analyzed by mass spectrometry.

HYDROLYTIC STABILITY OF OLIGOESTERS IN SIMULATED WATER-REDUCIBLE COATING FORMULATIONS—K.L. Payne, F.N. Jones, and L.W. Brandenburger

Journal of Coatings Technology, 57, No. 723, 35 (Apr. 1985)

Twelve oligoesters were prepared by partial esterification of trimellitic anhydride (TMA) and of "dimer acid" with 1,6-hexanediol, neopentyl glycol, 1,4-cyclohexanedimethanol, and 1,4-cyclohexanediol. Solutions of these oligomers in water/n-propoxy isopropanol were adjusted to pH 8.2 to 8.8 with N,N-dimethylethanolamine and were aged at room temperature, at 120°F (49°C) and at 135°F (57°C). Hydrolytic stability was estimated by weekly titration of alkaline oligomer solutions to observe change in acid number. Key results were:

(1) Partial ester of TMA suffered substantial hydrolysis within a few weeks at 120°C and 135°F. At room temperature they were fairly stable, exhibiting increases in acid number of 1% to 19% in 10 weeks, and of 5% to 23% in 30 weeks.

(2) Partial esters of "dimer acid" hydrolyzed much more slowly than partial esters of TMA. Acid numbers increased only 6% to 12% after 8 to 10 weeks at 135°F.

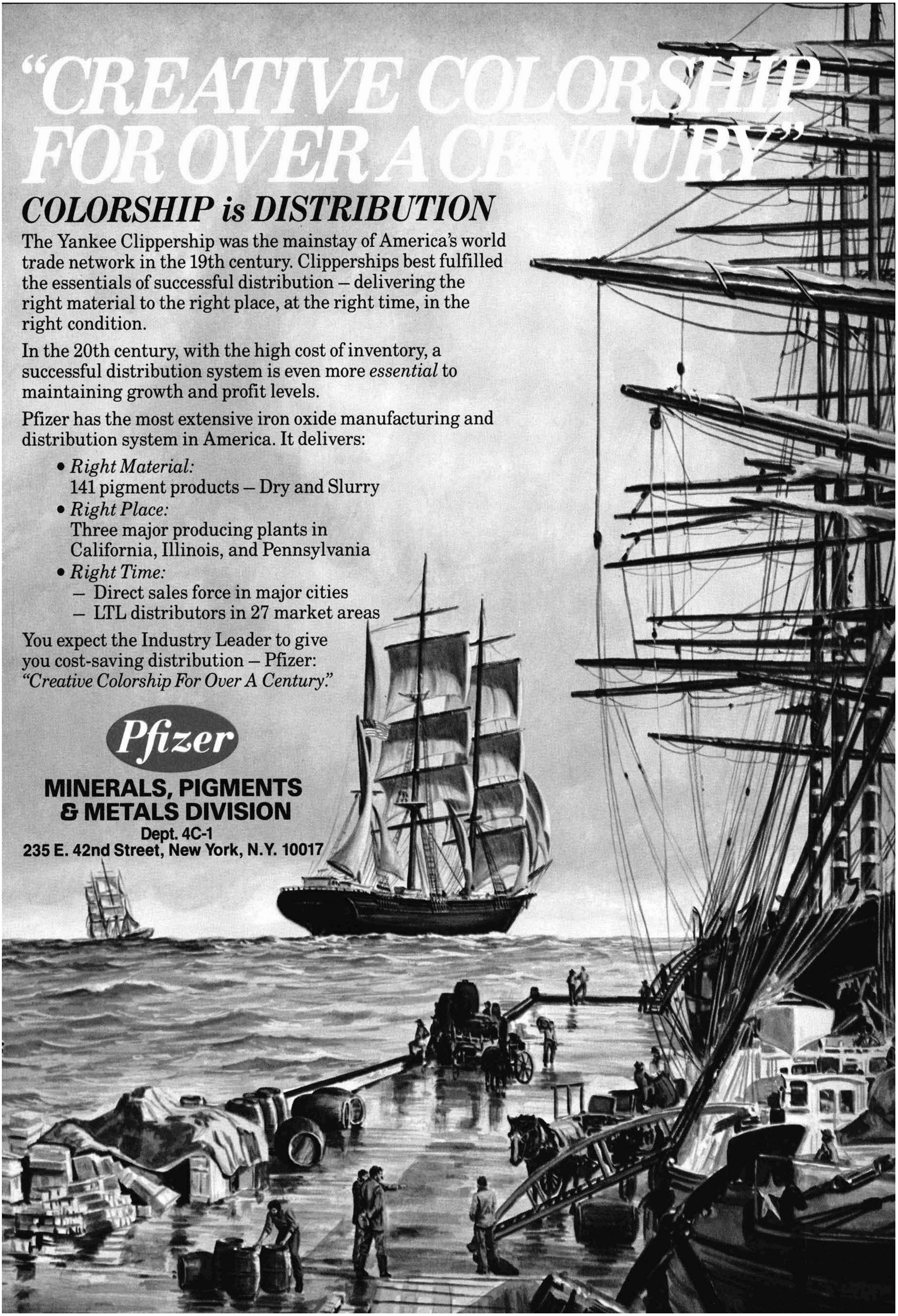
(3) The data did not yield clear-cut correlations of alcohol structure with stability.

Model compounds were studied to gain insight into the effect of alcohol structure on hydrolytic stability. Monoesters were prepared by partial esterification of phthalic anhydride with four monoalcohols. Solutions of these monoesters in water/n-propoxy isopropanol were adjusted to pH 8.1 to 8.3 with N,N-dimethylethanolamine and aged at 135°F. Hydrolytic stability was strongly influenced by alcohol structure in the following order: cyclohexanol (forms the most stable phthalate monoester); neopentyl alcohol; cyclohexyl methanol; and n-hexanol (forms the least stable phthalate monoester).

CHROMATOGRAPHIC CHARACTERIZATION OF EPOXY RESINS—D.R. Scheuing

Journal of Coatings Technology, 57, No. 723, 47 (Apr. 1985)

High performance size exclusion and reverse phase liquid chromatography can be applied to the characterization of epoxy resins of a wide molecular weight range. Oligomer separations differentiate "advancement" epoxy resins, formed by reaction of bisphenol A with a liquid epoxy resin, from "taffy" types manufactured by direct addition of epichlorohydrin to bisphenol A. Reactions of various molecular weight oligomers can be monitored by reverse phase LC. Several types of reverse phase packings are compared in terms of analysis time and selectivity.



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Federation News

Program Announced for FSCT Seminar, May 14-15 On Recent Home Painting Developments

Focusing on the theme, "Recent Developments in Home Painting," a seminar will be featured as the opening segment of the Federation's second annual Spring Week. To be held at the Omni International Hotel, Baltimore, MD, on May 14-15, the seminar will cover all areas of original and maintenance painting of the home. In addition, a cooperative "dialogue" will be provided for those involved in the manufacture, sale, specification, and application of paints and coatings.

The program will offer presentations by knowledgeable speakers representing various industry groups, who will offer up-to-date information on all aspects of home painting. Several open-forum periods will be featured, at which registrants will have an opportunity to question speakers on specific problem areas.

Program

The program is scheduled to include the following:

Tuesday, May 14

Welcome—Joseph A. Bauer, President, Federation of Societies for Coatings Technology

Opening Remarks—Royal A. Brown (Seminar Moderator), Technical Advisor, Federation of Societies for Coatings Technology

The Future of the Home Market to the Year 2000—Edward Bourguignon, Marketing Research Associate, Glidden Coatings and Resins Div., SCM Corp., Cleveland, OH

How will projected demographics change the home market? Will different types of dwellings be needed? A market research scientist reports on his company's studies and offers some predictions on these and other aspects of the homes of tomorrow.

What's New in Home Building?—Hugh Winstead, Director of Engineering Design, The Ryland Group, Inc., Columbia, MD

A builder discusses new developments and practices in home building, including changes in materials and methods. He explains his view on the painting operation, and what he expects from the original paint job.

Painting Metal and Masonry Surfaces—Robert L. Collins, Technical Manager, National Trade Sales Products, The O'Brien Corp., South Bend, IN

The selection of paint products for coating concrete, cement block, stucco, and brick is critical to good performance. The coating of metal surfaces, such as aluminum, steel, and zinc must be done correctly to achieve durability. Proper surface preparation and coating selection are discussed by an experienced paint scientist.

Painting Wood and Wood Composition Substrates—Roger E. Haines, Technical Director, Farmland Industries, Inc., Kansas City, MO

Wood, plywood, hardboard and other wood composition materials used in modern home construction, must all be properly painted for attractive appearance as well as protection. A technical expert discusses coating selection, proper coating techniques, along with potential problems and their solutions.

Architectural Wood Stains—William S. Hahn, Technical Director, Olympic Stain, Seattle, WA

Many home owners prefer the natural look of wood and choose to stain wood surfaces rather than paint them. Some textured wood lends itself better to staining than painting. The types of stains available and their recommended uses are discussed by the technical director of a major manufacturer.

The Exterior Latex or Oil Paint Dilemma—James Edwards, Technical Director, Consumer Products Div., Valspar Corp., Minneapolis, MI

Both latex and oil-type exterior paints offer good protection and service life. An expert paint chemist discusses the merits of each, along with recommendations for where and when each should be used, whether one can be applied over the other, and the degree of performance that may be expected. Geographical location can be an important factor.

Open Forum

Speakers will assemble for a discussion period.

Wednesday, May 15

How the Architect Views Paint and Painting—Stuart Liss, Stuart Liss & Associates, Architects, Arlington, VA

An architect discusses his role in specifying the paint materials for the houses he designs. Included are choosing the paint, developing and monitoring adherence to specifications, and service life expectations from the original paint job, including his expectations in terms of service life, decoration and protection.

Preparing and Painting Drywall Surfaces—A. Clarke Boyce, Technical Service Manager, Resins Div., Nacan Products Ltd., Toronto, Ontario, Canada

Drywall construction is used extensively in the interior of most new homes. An expert discusses the correct materials and methods to be used in sealing, taping joints, and painting drywall surfaces.

Selecting and Using Caulks and Sealants—Dr. Robert M. Evans, Center for Adhesives, Sealants and Coatings, Case-Western Reserve University, Cleveland, OH

Many new sealants are available for use by both the professional painter and the home owner. The merits of each are described, along with recommendations for their use in caulking and sealing foundations, door frames, windows, skylights and other areas of the home where such protection is needed.

Common Paint Problems and How to Treat Them—Ron Raley, Product Manager-Architectural Coatings, Devoe and Raynolds Co., Louisville, KY

What causes paint to sometimes crack, blister, peel, and chalk? An experienced paint chemist explains how the proper cleaning and surface preparation, plus the selection of the correct coating, can help avoid these problems. A variety of common paint failures and suggested solutions are discussed.

Open Forum

The Importance of Paint Quality—Thomas Hill, Laboratory Manager, Pratt & Lambert, Inc., Buffalo, NY

Considering the high cost of labor to apply paint, it is economically sound to use high-quality paint products. Included in this discussion are the superior performance of high-quality paints, suggestions for the procurement of good quality products, and an explanation of what constitutes quality.

The Role of the Painting Contractor—Wayne Jolly, President, W. W. Jolly Painting Contractors, Randallstown, MD

A painting contractor discusses his relationship with the builder, paint dealer, and the home owner. Discussed are his methods of paint application and the desirable properties of the paint from his point of view.

How the Paint Dealer Can Help—Woody Cornetta, Owner, Elmont Paint and Wallpaper, Inc., Elmont, NY

The independent paint dealer can be of tremendous help in selecting the "right paint for the job." An experienced dealer explains his role and provides some observations on paint and paint selection.

Registration

To register, fill out accompanying form and return with payment to Federation of Societies for Coatings Technology, 1315 Walnut St., Suite 832, Philadelphia, PA 19107. Registration fee is $125 for FSCT members, $155 for non-members; after May 1, registration fee is $165 for everyone. (Payment must be in U.S. funds, payable in U.S. banks.)

Included in the registration fee is continental breakfast, luncheon, coffee breaks, and copies of talks, as well as bus transportation to airport at completion of seminar. *Please note reference to bus on registration form, and check appropriate box.*

Note: No refund for cancellations received after May 9.

Housing

Headquarters hotel is the Omni International, located at Charles Center in downtown Baltimore.

RECENT DEVELOPMENTS IN HOME PAINTING SEMINAR

REGISTRATION FORM

Registration fees: $125 (FSCT members); $155 (non-members)
$165 for everyone after May 1

Check must accompany registration form. Make checks payable to Federation of Societies for Coatings Technology. (Payment must be made in U.S. funds payable in U.S. banks.)

NICKNAME (For Registration Badge) ____________________

FULL NAME ____________________

JOB TITLE ____________________

COMPANY ____________________ PHONE ____________________

ADDRESS ____________________

CITY ____________________ STATE ____________ ZIP __________

Name of Federation Society
of Which You Are A Member ____________________

I will ☐ will not ☐ use the Federation bus to Baltimore International airport at conclusion of seminar.

No refund for cancelled registrations received after May 9.

Return form and check to: Federation of Societies for Coatings Technology
1315 Walnut Street, Suite 832,
Philadelphia, PA 19107

IF YOU REQUIRE HOUSING AT THE OMNI HOTEL

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Please reserve: Single ____________ Room rate is $79 single and $96 double occupancy (per night)

Double/Twin __________

*Reservations will be held until 6:00 pm. If arrival is after 6:00 pm, hotel requires deposit for one night's lodging to guarantee availability. Make check payable to "Omni International Hotel."

Note: Reservations must be made no later than April 30 to guarantee availability and rates.

Return this form to: Federation of Societies for Coatings Technology
1315 Walnut Street, Suite 832, Philadelphia, PA 19107

Committee Activities

LIAISON

Report on IUPAC's Supported Polymer Films Group

The International Union of Pure and Applied Chemistry (IUPAC) is the only international organization that is involved with *all* branches of chemistry. Its purpose is the advancement of chemistry and the worldwide dissemination of such knowledge. Created after World War I, the IUPAC, in the United States, is an arm of the National Academy of Sciences—National Research Council. A total of about fifty national organizations are represented in IUPAC.

We in the organic coatings industry are fortunate to be represented by a Supported Polymer Films Group (SPF) which is a part of the large Macromolecular Division of IUPAC.

The following is a progress report of the Group by its Secretary, Dr. J. Sickfeld, of Germany. Included are the minutes of the Group's meeting in Lugano, Switzerland, September 26, 1984. It is felt that the information of these activities will be of interest to all JCT readers.

MILTON A. GLASER,
Federation Liaison Committee
Federation Delegate to IUPAC

A moment of silence was observed in memory of Mr. Gunnar Christensen, a distinguished colleague and a member of the Working Party for many years.

Report of the Chairman

Chairman Dulog reported on the meeting of Commission 4 of IUPAC-MMD which took place in Paris at the same time as the recent FATIPEC Congress, and on the last meeting of IUPAC at Lyngby, Denmark. He also reported that the Macrosymposia in Prague in 1986 will be concerned with polymeric membranes and crosslinked polyepoxides. Individual membership in IUPAC-organizations was approved by most participating organizations.

Concerning publication policy, there was a strong statement from the publications officer in Oxford to publish only in *Pure and Applied Chemistry*. The attitude of the SPF-group towards this is well known and is unchanged.

As the Chairman was unable to participate in the meeting of IUPAC, he will send a report detailing the activities of the group since the last meeting in Dubendorf, April 28–29, 1983.

The Chairman also discussed the difficulties in finishing certain projects and in getting other projects started with respect to the economic situation and the need of industry to cooperate in procuring the needed testing materials and in taking part in certain laboratory work.

Current Works

Dr. Hvilsted has prepared the final draft of his report on "Analysis of Emulsion Paints," which will be published in *Progress in Organic Coatings*.

New Projects

Following a meeting on the proposed project, "Characterization of Cure of Coatings in Relation to Performance Properties," Dr. Kooistra proposed, that following four years of discussion and a disappointing lack of interest, he relinquish the leadership of the project. In subsequent discussions no specific resolutions were forthcoming nor was the project dropped.

The Working Party formally adopted a proposal by Chairman Dulog concerning "Corrosion Protection by Organic Coatings," with respect to combined efforts by various laboratories and different methods.

Dr. Gerster reported on his results concerning the possibilities of measuring the dielectric constant on the surface of thin films to characterize the degradation as a consequence of weathering. Although the technical problems were solved with the cooperation of Centre Electronique Horloger, there is still the continuing problem of the manufacture of the measuring device. Subsequently, Dr. Oesterle pointed out that it would be desirable to measure the dielectric constant of the whole film because there are a number of possibilities for the characterization of the outmost surface.

Dr. Oesterle proposed the study of the formation of free volume and the influence of free volume as a fundamental quality of the behavior of paint film, e.g., the interaction between water and the polymeric binder which determines the properties and the performance of the film. As possibilities, Dr. Hansen mentioned thermal analysis, measurement of T_g, and variation of expansion coefficient. It was resolved to keep the proposal in mind for future discussion and all members were requested to be aware of the literature of approaches and developments concerned with the problem of free volume and to also contact other experts working in this field.

Attendance

The following attended the meeting: L. Dulog, Chairman; J. Sickfeld, Secretary; P. Askestad; U. Biethan; W. Funke; A.C. Gerster; C.M. Hansen; S. Hvilsted; M.G. Kooistra; K.M. Oesterle; E.V. Schmid; A. Toussaint; and U. Zorll.

J. SICKFELD, *Secretary*


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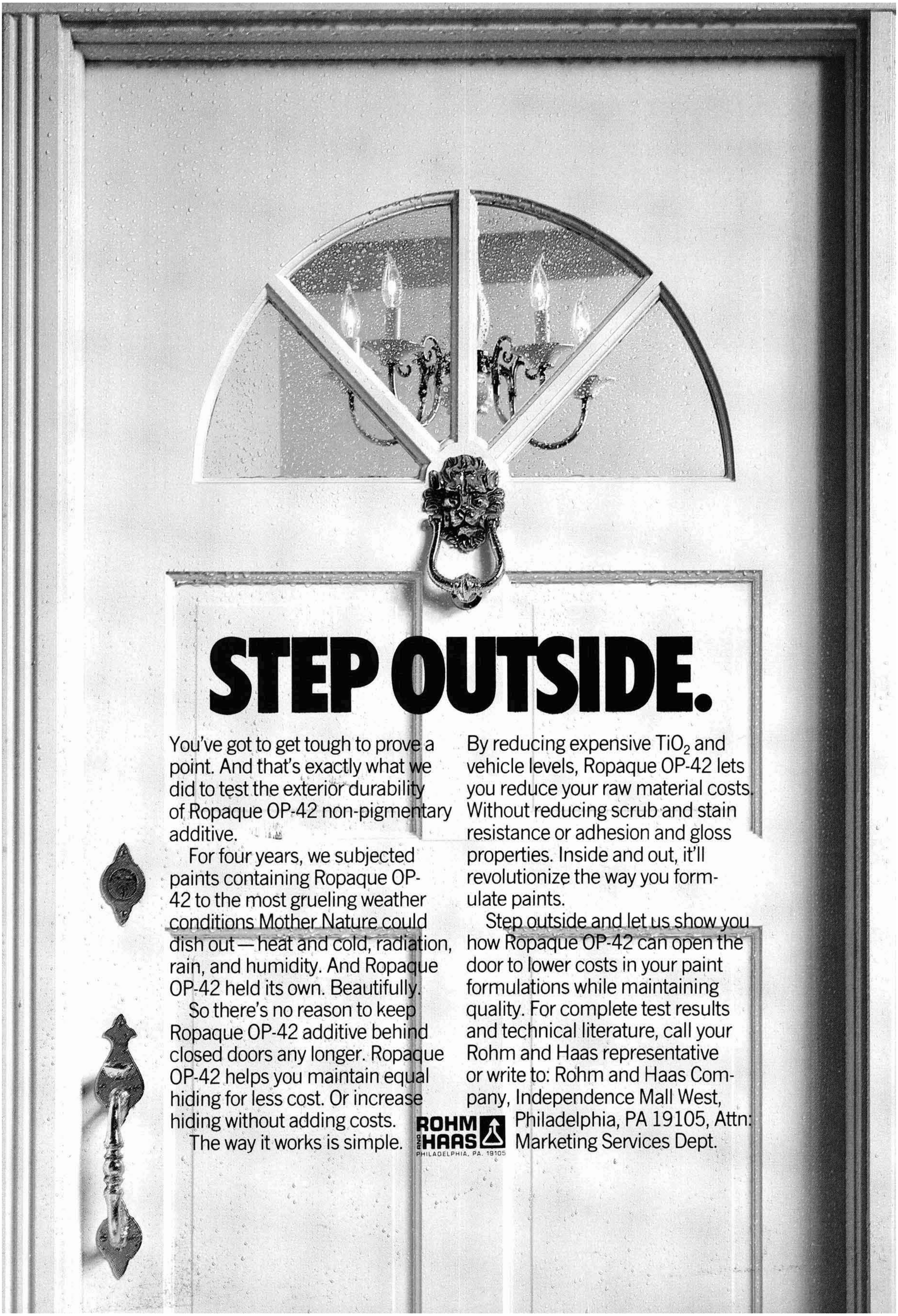

Odorless Paint Developed by Battelle Institute to Retard Fire

A water-based paint, developed by Battelle Institute's Columbus Laboratories in conjunction with the David Taylor Naval Ship R&D Center, is designed to be fire-retardant and odorless. In addition, the paint can be safely applied to ship bulkheads while the craft is docked or at sea. Its non-emission composition prevents it from emitting potentially volatile and toxic organic components or solvents either during or immediately after its application.

Conventional paints used by the Navy can be applied only when the craft is docked, and at least five days before going to sea so the paint can dry and the fumes dissipate. By being able to use the paint in non-ventilated areas at sea as well as on shore, the Navy can save money merely by eliminating ship downtime. One shipyard estimated that the new paint reduced the cost of painting an aircraft carrier by $400,000.

The new paint also saves space on board since no storage of solvents is required as with oil-based paints. This saves about four pounds per gallon in storage, mainly because of the reduced stock of solvents.

Although its primary application is for ship bulkheads, particularly in poorly ventilated areas, the paint also is used as a flame-retardant and to cover and protect any corrosion-prone surface. According to researchers, the paint surface is durable, washable, recoatable, and has a long shelf-life. It can also be used in a wide range of temperatures and it spreads evenly with the use of conventional equipment.

NPCA Sells Verlan Ltd. Insurance Co.

The National Paint and Coatings Association has announced the sale of its captive insurance company, Verlan Limited, along with its two wholly-owned subsidiaries, Verlan Limited—Vermont and Coatings Industry Services, to a group of some 50 policyholders. The sale includes the entire stock, assets, and liabilities of the three companies.

Verlan President, John Montgomery, stated, "The sale of Verlan to its policyholders not only assures continuity of operations, but by converting from an association captive to an industry captive company, we can now make specialized professional engineering and loss control services available to related industries, such as printing inks, adhesives and sealants, etc."

The companies will continue to operate out of their present offices in Hamilton, Bermuda; Barre, VT; and Washington, D.C.

Bruning Paint Acquires Kyanize Paint, Inc.

A corporation formed by Bruning Paint Co., Baltimore, MD, has announced the acquisition of Kyanize Paints, Inc. The transaction is expected to be completed in early 1985. Kyanize Paints, founded in 1899, is primarily a trade sales paint manufacturer with plants in Everett, MA and Springfield, IL.

Japco Announces Expansion Plan

Jamestown Paint & Varnish Co. (Japco), Jamestown, PA, has begun an expansion project which will include additional office, laboratory, and plant facilities.

Designed to increase plant output by 50% and provide more comprehensive research and development services to customers, the expansion involves the renovation of 2600 sq ft of the facility and a 1200 sq ft addition.

Improvements will include a warehouse for additional raw material storage, a warehouse for finished product storage, an upgrading of the entire production facility to improve productivity and material flow, and doubling of laboratory space and addition of personnel. New laboratory equipment includes a Weather-Ometer, QUV environmental testing equipment and salt test spray equipment.

Standard Container Changes Name

Standard Container Co., the plastic and metal container manufacturer headquartered in Fairfield, NJ, has changed its name to Brockway Standard, Inc.

Founded more than a century ago, Standard Container Co. became a part of Brockway, Inc. through an acquisition in 1979. Headquartered in Brockway, PA, Brockway, Inc. produces plastic, glass, and metal containers.

In addition to its corporate headquarters in Fairfield, Brockway Standard operates manufacturing centers in Picayune, MS; Homerville and Morrow, GA; Dallas, TX; and Birmingham, AL.

Study Seeks to Improve Auto Paint Adherence

Researchers at Battelle-Geneva are conducting a study to improve the adherence of automotive paints and adhesives on hot-dipped zinc-coated sheets. Expected to benefit from this multiclient study are paint producers, phosphate bath producing companies, automobile manufacturers, and galvanized and electrocoated steel manufacturers.

In carrying out their study, researchers will prepare samples in the laboratory with various zinc deposits. After the samples have been sorted, degreased, and phosphated according to different conditions, researchers will apply cataphoretic and powder epoxy paints as well as adhesives.

Adhesion tests will be performed according to automotive standards to obtain correlations between adhesion and process parameters. Parameters to be considered include steel quality, zinc-layer composition, post dipping treatments, storage conditions, degreasing, and the influence of phosphating treatments.

The study is still open for membership.

Announcing faster delivery and service for Du Pont Elvacite® acrylic resins

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Du Pont now has national distribution for ELVACITE resins. A coast-to-coast chain of distributors, experienced in specialty chemicals, can bring you faster delivery of ELVACITE resins. Quick deliveries are only a phone call away. Take advantage of combining orders for ELVACITE with other materials from your local distributor.

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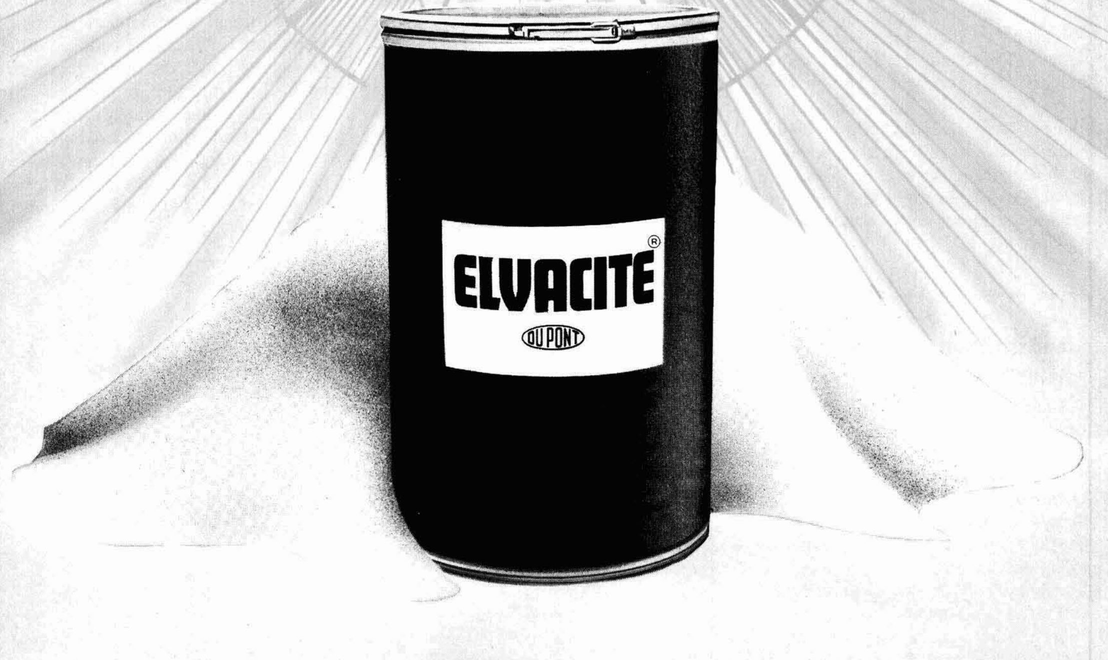

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NL Abandons Pigment Acquisition

NL Industries, Inc., New York, NY, has announced that it would not pursue its acquisition of American Cyanamid's titanium dioxide pigment business because the Federal Trade Commission voted to file suit challenging the transaction.

When the proposed acquisition was announced, NL indicated it would consider expanding the American Cyanamid TiO_2 plant located in Savannah, GA. Now NL plans to increase its own capacity, using the firm's proprietary chloride process technology. Sites being considered by NL for expansion are its titanium dioxide pigment plants in Canada and Europe.

Ashland Acquires Loomis Chemical

Ashland Chemical Co., Columbus, OH, has purchased the assets of Loomis Chemical Co., a Portland, OR distributor of industrial chemicals. The Loomis organization will continue to be operated by existing personnel, reporting through Ashland Chemical's Industrial Chemicals & Solvents Division.

Union Carbide Purchases Rights to UV Technology From General Electric Co.

Union Carbide Corp., Danbury, CT, has licensed rights from the General Electric Company under a broad range of patents relating to radiation curable epoxide systems.

As a result of this agreement, Union Carbide's customers for Cyracure™ cycloaliphatic epoxides will be free to practice the technology covered by approximately 200 General Electric, 3M, and Union Carbide patent rights in the U.S. and major foreign countries.

Macbeth Expands Color Control Facilities

Macbeth, a division of Kollmorgen Corp., has acquired an additional 10,000 sq ft facility in Newburgh, NY, where its headquarters are located. Personnel has been increased approximately 30% to support the firm's color control business.

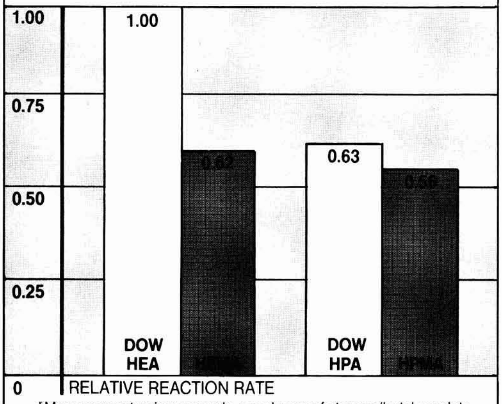
This second location will allow for expansion of Macbeth's production facility. In addition, the firm has added to its staff of optical and electrical engineering groups, as well as to its software, technical service, and field sales organization.

In a related move, the company has just established an office in Hong Kong. It also operates foreign offices in Canada, England, and Switzerland.

DOW HEA and HPA . . .

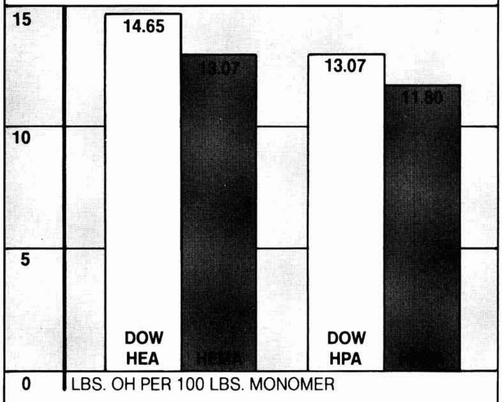
Pound for pound, more crosslinking sites than methacrylates.

Relative Reaction Rates of Copolymers Incorporating Hydroxyalkyl Acrylates vs. Hydroxyalkyl Methacrylates.†



†Measurement using example copolymer of styrene/butyl acrylate and 0.05 mole of hydroxy monomer per 100 g. polymer. Reaction rate with hexamethoxymethyl melamine at 100°C.

Comparative Hydroxyl Content — Hydroxyalkyl Acrylates vs. Hydroxyalkyl Methacrylates.



HEA: DOW hydroxyethyl acrylate
HEMA: hydroxyethyl methacrylate
HPA: DOW hydroxypropyl acrylate
HPMA: hydroxypropyl methacrylate

DOW hydroxyethyl acrylate (HEA) and hydroxypropyl acrylate (HPA) functional vinyl monomers offer substantially higher OH reactivity than hydroxyalkyl methacrylates. Plus, their lower molecular weights translate into a 12% reduction in functional monomer requirements and lower material costs.

Exceptional versatility for resin polymerization.

DOW HEA and HPA copolymerize with a wide variety of other vinyl monomers in both aqueous and non-aqueous systems. The resulting pendant hydroxyl functionality can be used in crosslinking reactions with melamine resins, polyisocyanates, polyanhydrides and epoxy resins.

Faster crosslinking and lower temperature requirements save time and money.

When HEA and HPA hydroxyl functionality is added to polymers and copolymers, higher reactivity in crosslinking reactions is the result. This permits faster curing cycles and accelerated line speeds for increased productivity.

With DOW HEA and HPA, polymers can be converted into radiation curable resins when vinyl unsaturation is introduced through reactions with backbone functional groups. And high reactivity at lower temperatures makes HEA and HPA useful in coatings for plastics and other temperature-sensitive substrates.

Favorable end-use properties.

DOW HEA and HPA hydroxyl functionality can also contribute:

- Outstanding mechanical and endurance properties in coatings enamels.
- Solubility in water and polar solvents.
- Stabilization of aqueous polymer dispersions.
- Improved adhesion to metals, wood, textiles, paper and other substrates.

Put DOW HEA and HPA to work for you.

Dow pioneered the commercial development of hydroxyalkyl acrylates. As the HEA/HPA leaders, we have in-depth product knowledge and experience to assist you with polymer development. Find out how DOW HEA and HPA can outperform methacrylates in your resin systems, pound for pound.

Write The Dow Chemical Company, Coatings and Resins Dept., Inquiry Services, 1703 S. Saginaw Road, Midland, Michigan 48640.

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Coating Compositions Based on Acrylic-Polyurethane Interpenetrating Polymer Networks

P.I. Kordomenos, K.C. Frisch, H.X. Xiao, N. Sabbah
Detroit Society for Coatings Technology
Technical Committee

Interpenetrating polymer networks were synthesized from a two-component polyurethane and an unsaturated urethane modified acrylic copolymer. The two-component polyurethane was prepared from hydroxyethylacrylate butylmethacrylate copolymer and cured with an aliphatic polyisocyanate. The unsaturated acrylic copolymer was made from the same hydroxy-functional acrylic copolymer modified with isocyanatoethyl methacrylate (IEM).

Simultaneous IPN's were prepared from the above two polymer systems at various ratios. The IPN systems were characterized by means of mechanical properties and morphology.

INTRODUCTION

Interpenetrating polymer networks (IPNs) are unique types of polymer alloys consisting of two (or more) crosslinked polymers containing essentially no covalent bonds or grafts between them.[1-3] These intimate mixtures of crosslinked polymers are held together by permanent entanglements, i.e., they are polymeric catenanes, produced by homocrosslinking of two or more polymer systems. Formation of IPNs is the only way of intimately combining crosslinked polymers with the resulting mixtures exhibiting, at worst, only limited phase separation.

IPNs can be prepared either by the "sequential" technique by swelling a crosslinked polymer with monomer and the crosslinking agent of another polymer, and curing the swollen polymer *in situ*[4] or by the "simultaneous" or "SIN" technique by blending the linear polymers, prepolymers, or monomers in some liquid form (latex, solution, or bulk) together with the respective crosslinking agents, evaporating the vehicle (if any), and curing the component polymers simultaneously.[1,5-7]

IPNs synthesized to date exhibit varying degrees of phase separation depending primarily on the compatibility of the component polymers.

The combination of various chemical types of polymer networks in different compositions, resulting frequently in controlled, different morphologies, has produced IPNs with synergistic behavior. For example, if one polymer is glassy and the other is elastomeric at room temperature, one obtains either a reinforced rubber or a high impact plastic depending upon which phase is continuous. Thus, synergistic properties may be obtained by IPNs such as enhanced tensile and impact strengths,[5-7] improved adhesion and, in some cases, significantly greater sound and shock absorption.

This paper describes novel urethane-acrylic IPNs for coating applications. Their mode of preparation used was the simultaneous or SIN technique. A schematic representation of the synthesis of these IPNs is shown in *Figures* 1 and 2.

EXPERIMENTAL

Raw Materials

The raw materials used in this study are shown in *Table* 1. The isocyanatoethyl methacrylate (IEM) was distilled before use. The other monomers were used as received.

Presented by Dr. Kordomenos at the 62nd Annual Meeting of the Federation of Societies for Coatings Technology, in Chicago, IL, October 24, 1984.

Synthesis of Hydroxy-functional Acrylic Copolymer [p(BMA-HEA)]

Into a reaction kettle equipped with a nitrogen inlet, stirrer, reflux condenser, and an addition funnel were charged 500 g of xylene. The xylene was heated to reflux and a mixture of 375 g of butyl methacrylate (BMA), 125 g of 2-hydroxyethyl acrylate (HEA) and 10 g of t-butyl-perbenzoate (t-BPB) were added over a period of five hours. After the end of the addition, the solution was *kept at 140–150°C for another hour and then 1 g of t-BPB* was added. The reaction mixture was post-reacted for two more hours and was then cooled to room temperature. The acrylic polymer solution had a viscosity of 400cps at 50% NV in xylene. The number average molecular weight of this polymer, as measured by gel permeation chromatography (GPC) was $\overline{Mn}=2076$ and had a MW distribution of 3.6.

Synthesis of BMA-HEA-IEM Terpolymer

Into a reaction kettle, equipped with nitrogen inlet, stirrer, reflux condenser, and thermometer were charged 464 g of the previously described acrylic copolymer, 171 g of IEM, 1.27 g of dibutyltin dilaurate and 0.17 g of hydroquinone. The reaction mixture was kept at 75–80°C for 2–3 hours until all of the isocyanate was fully reacted as indicated by the IR spectrum. The reaction product was then cooled to RT and the viscosity was measured to be 400 cps at 50% NV in xylene. The number average MW was 2483 and the MW distribution was 11.3.

Preparation of Coatings

PREPARATION OF P(UA)-1 COATINGS: In the 50% solution of p(BMA-HEA-IEM) were added 3% by weight of calcium naphthenate, 1% lead naphthenate, 1% cobalt naphthenate and 1% DMBEHPH (*Table* 1). Clear films were drawn on glass and steel (Bond 40) panels and were cured at 80°C for 30 min and post-cured for 30 min at 120°C.

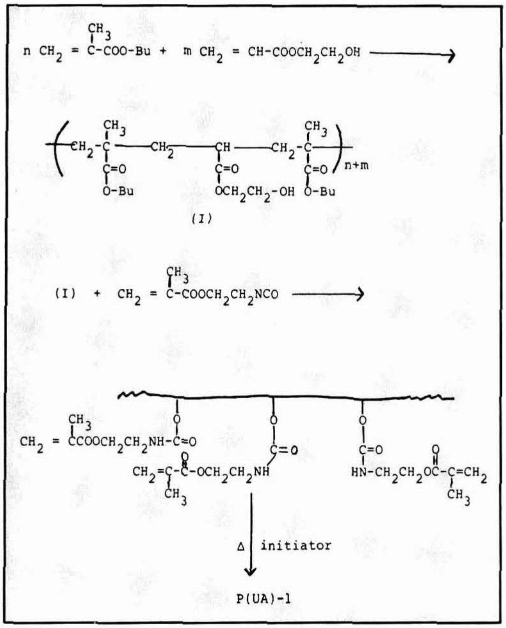

Figure 1—Synthesis of P(UA)-1

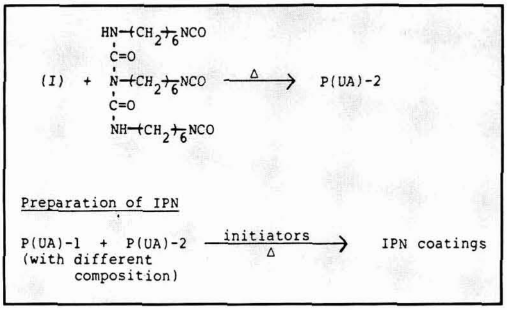

Figure 2—Synthesis of P(UA)-2 and preparation of IPN coatings

PREPARATION OF P(UA)-2 COATINGS: In 100 g of P(MBA-HEA) solution were added 45 g of Desmodur N-100 and 1 g of Dabco XDM catalyst. Films of P(UA)-2 were prepared and cured using the same conditions as for P(UA)-1.

PREPARATION OF IPN COATINGS: P(UA)-1 and P(UA)-2 were mixed at different ratios (*Table* 2) and films prepared on glass and steel panels and cured as in the case of P(UA)-1.

Testing of Coatings

The tensile strength and elongation at break were measured on an Instron tensile tester at a crosshead speed of 2 in./min; the hardness was measured by a Shore A and

Table 1—Raw Materials

Designation	Description	Supplier
HEA	2-Hydroxyethyl acrylate	Rohm and Haas Co.
BMA	Butyl methacrylate	Rohm and Haas Co.
IEM	Isocyanatoethyl methacrylate	Dow Chemical Co.
t-BPB	t-Butyl perbenzoate	Lucidol Corp.
Desmodur N-100	Biuret triisocyanate-adduct of hexamethylene diisocyanate with water	Mobay Chemical Co.
T-12	Dibutyltin dilaurate	M&T Chemical Co.
DMBEHPH	Dimethyl 2,5-bis(ethylhexanoyl peroxy) hexane (Lupersol 225)	Lucidol Corp.
Ca drier	Calcium naphthenate (4% calcium)	Mooney Chemical Co.
Pb drier	Lead naphthenate (24% lead)	Mooney Chemical Co.
Co drier	Cobalt naphthenate (6% cobalt)	Mooney Chemical Co.
—	Xylene	Aldrich Chemical Co.
Dabco XDM	N,N-(Dimethylamino-ethyl)-morpholine N,N-Dimethyl-4-morpholine-ethanamine	Air Products Co.

Table 2—Composition of Poly(urethane-acrylates) and IPNs From Them

Materials	Description	Composition
P(UA)-1	P(BMA-HEA-IEM), crosslinked terpolymer	100%
P(UA)-2	P(BMA-HEA-Desmodur N-100), reaction product of hydroxyl-containing copolymer with Desmodur N-100	100%
IPN-1	Varying compositions of blends of P(UA)-1 and P(UA)-2	P(UA)-1/P(UA)-2 = 80/20
IPN-2	″ ″ ″ ″	″ ″ 70/30
IPN-3	″ ″ ″ ″	″ ″ 60/40
IPN-4	″ ″ ″ ″	″ ″ 50/50
IPN-5	″ ″ ″ ″	″ ″ 40/60
IPN-6	″ ″ ″ ″	″ ″ 30/70
IPN-7	″ ″ ″ ″	″ ″ 20/80

D durometer. The lap shear strength in tension was measured on an Instron tester by ASTM D-3163-73 with 3×0.5 in. steel plates lapped 0.5 in. from their edges.

The glass transition temperatures (Tgs) were determined on a Perkin-Elmer TMS-2 Thermomechanical Analyzer (TMA) at temperatures ranging from −100°C to +100°C and 0.01 mm of penetrating range, 115 g of penetrating weight and at 10 degrees/min. of heating rate.

In order to determine the morphology, samples were prepared by freeze-fracturing in liquid nitrogen and applying a gold coating of approximately 200 Angstroms. The micrographs were obtained using a Phillips Scanning Electron Microscope Model SEM 505.

RESULTS AND DISCUSSION

Mechanical Properties of IPN Coatings

The IPN coatings with different composition exhibited better tensile strength than those of their component polymers P(UA)-1 and P(UA)-2, as shown in *Table* 3 and *Figure* 3. In particular the composition P(UA)-1/P(UA)-2 = 60/40 exhibited a maximum value, presumably at a high degree of interpenetration. Since the hydroxyl-containing copolymer P(BMA-HEA) was prepared by solution polymerization at high temperature and in the presence of relatively high concentration of peroxide, the reaction product possessed a rather low molecular weight and broad molecular weight distribution. After the reaction between P(BMA-HEA) and IEM, the reaction product P(UA)-1 had not undergone any great change with regard to the molecular weight and molecular weight distribution. As a result, the crosslinked P(UA)-1 possessed a relatively low cohesive energy density and rather poor mechanical properties.

P(BMA-HEA) was reacted with Desmodur N-100 which contained flexible aliphatic chains. As a result, the crosslinked P(UA)-2 made by reaction of the NCO groups with the OH groups possessed a relatively high cohesive energy density due to hydrogen bonding from the urethane structure. Both hardness and elongation of the IPN coatings exhibited a range of values between those of P(UA)-1 and P(UA)-2 due to the interpenetration between the rigid network of P(UA)-1 and flexible network of P(UA)-2.

The adhesive strength of IPN films to steel panels is shown in *Table* 4. Most IPN coatings exhibited higher lap shear strength than their original components P(UA)-1 and P(UA)-2 except at 30/70 and 20/80 compositions. Especially the three IPN coatings with 60/40, 50/50 and 40/60 of compositions, respectively, exhibited the highest lap shear strength.

Table 3—Mechanical Properties of IPN Coatings

Composition	P(UA)-1/P(UA)-2	Ts(psi)	E (%)	Hardness A	Hardness D
P(UA)-1	100/0	427	10	92	44
IPN-1	80/20	3100	11	89	41
IPN-2	70/30	3423	13	85	37
IPN-3	60/40	4351	14	84	35
IPN-4	50/50	3659	15	82	35
IPN-5	40/60	2762	16	80	33
IPN-6	30/70	2371	35	78	32
IPN-7	20/80	2303	37	76	28
P(UA)-2	0/100	2247	80	75	27

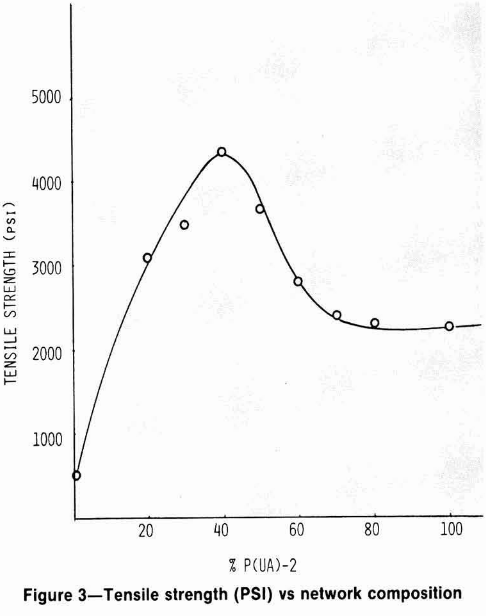

Figure 3—Tensile strength (PSI) vs network composition

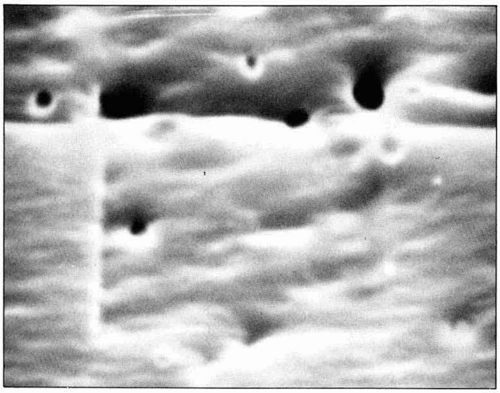

Figure 4—Micrograph of P(UA)-1

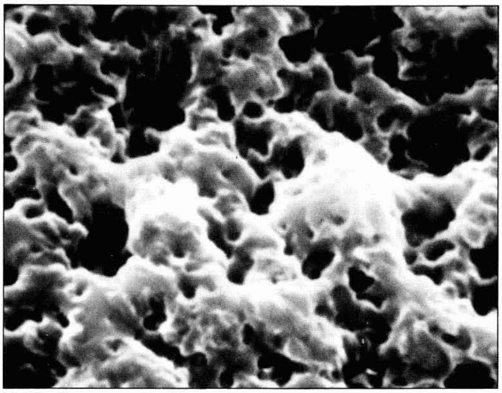

Figure 7—Micrograph of IPN-3 coating (60/40 of P(UA)-1/P(UA)-2)

Figure 5—Micrograph of IPN-1 coating (80/20 of P(UA)-1/P(UA)-2)

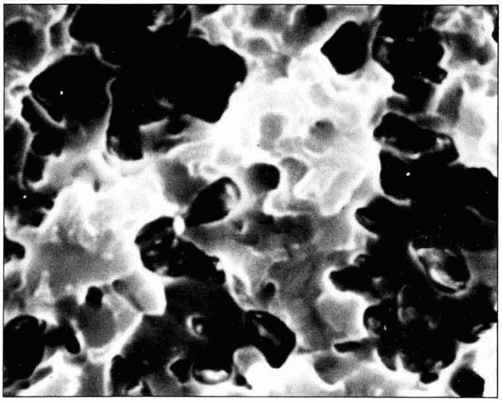

Figure 8—Micrograph of IPN-4 coating (50/50 of P(UA)-1/P(UA)-2)

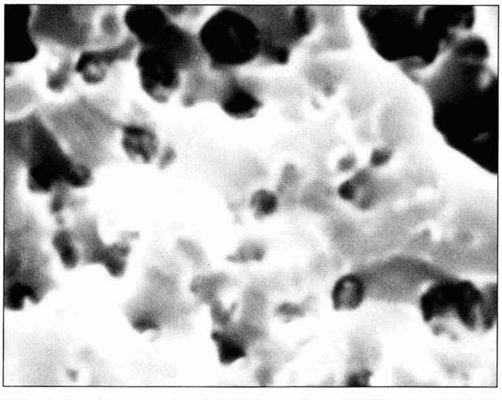

Figure 6—Micrograph of IPN-2 coating (70/30 of P(UA)-1/P(UA)-2)

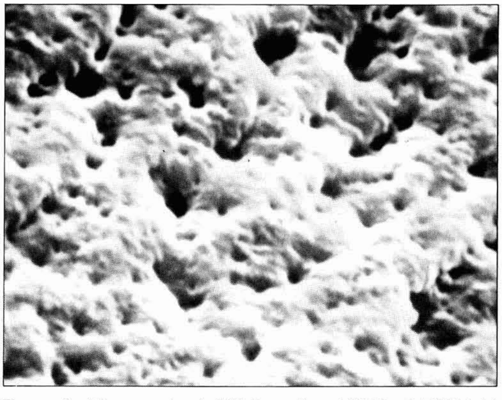

Figure 9—Micrograph of IPN-5 coating (40/60 of P(UA)-1/P(UA)-2)

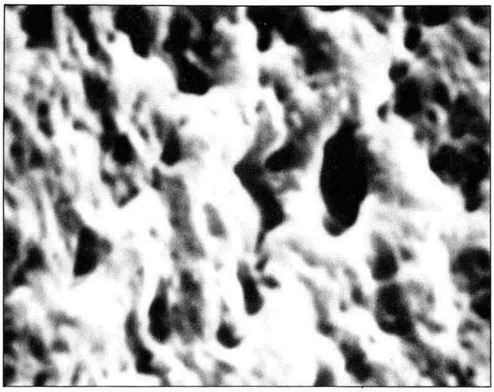

Figure 10—Micrograph of IPN-6 coating (30/70 of P(UA)-1/P(UA)-2)

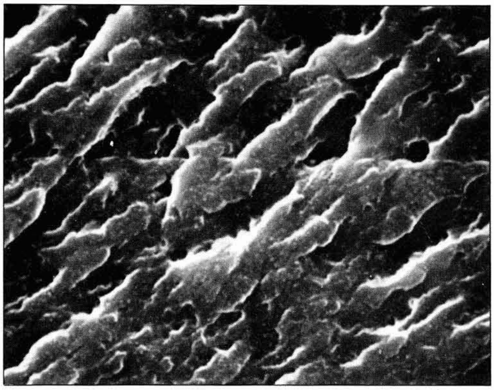

Figure 13—Micrograph of IPN-7 coating (20/80 of P(UA)-1/P(UA)-2) (with 4000X magnification)

Morphology of IPN Coatings

Glass Transition Temperatures by TMA: The Tgs for all the IPN coatings with different compositions exhibited only one Tg value which was between the Tgs of P(UA)-1 and P(UA)-2 (*Table* 5). This implies that interpenetrating polymer networks between P(UA)-1 and P(UA)-2 were formed by segmental mixing. As a result, P(UA)-1 with high crosslink density (due to many double bonds in side chains) and rigid structure gave high Tg values and P(UA)-2 with low crosslink density and flexible structure gave low Tg values. The Tg values of IPN coatings shifted toward the direction of P(UA)-2 with increasing concentration of P(UA)-2.

Micrographs by SEM: As shown in *Figures* 4-12, both components, P(UA)-1 and P(UA)-2, and the IPN coatings with different compositions exhibited only one phase at a magnification of 7000× except for *Figure* 11 (20/80). *Figure* 11 showed some small particles of P(UA)-1, dispersed into the P(UA)-2 matrix. The size of the small particles was in the range of 10Å–30Å indicating molecular mixing resulting from interpenetration between the networks. *Figure* 13 showed the morphology of the sample to be the same as for *Figure* 11 but with a 4000x magnification.

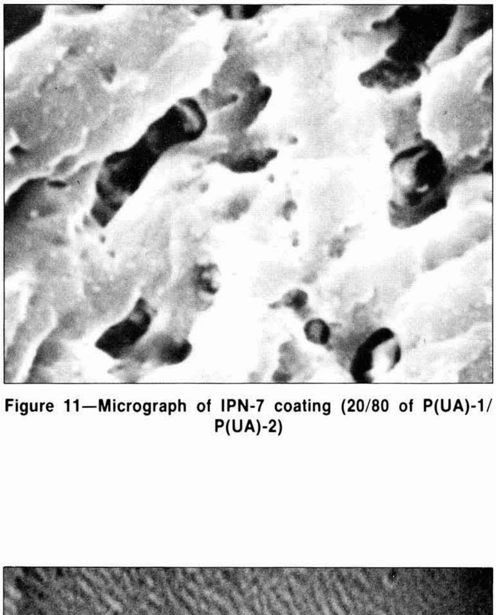

Figure 11—Micrograph of IPN-7 coating (20/80 of P(UA)-1/P(UA)-2)

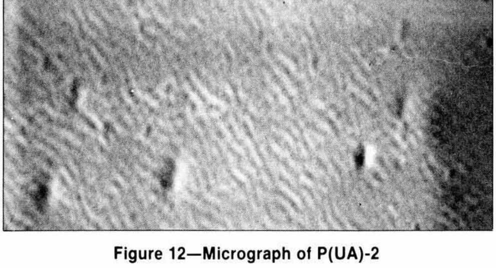

Figure 12—Micrograph of P(UA)-2

Table 4—Adhesive Strength of IPN Coating To Steel Panel

Composition	P(UA)-1/P(UA)-2	Lap Shear (psi)	Type of Failure[a]
P(UA)-1	100/0	64	Ad
IPN-1	80/20	677	Ad/Co
IPN-2	70/30	557	″
IPN-3	60/40	784	Co
IPN-4	50/50	627	″
IPN-5	40/60	636	″
IPN-6	30/70	202	″
IPN-7	20/80	272	Ad/Co
P(UA)-2	0/100	322	″

(a) Ad: Adhesive failure; Co: Cohesive failure

Table 5—Glass Transition Temperatures Of IPN Coatings (by TMA)

Composition	P(UA)-1/P(UA)-2	Tg (°C)
P(UA)-1	100/0	92
IPN-1	80/20	75
IPN-2	70/30	74
IPN-3	60/40	70
IPN-4	50/50	66
IPN-5	40/60	67
IPN-6	30/70	48
IPN-7	20/80	47
P(UA)-2	0/100	42

CONCLUSIONS

Novel IPN coatings were prepared from P(UA)-1, which contained both acrylate and urethane structures and was crosslinked by free radical polymerization of the pendant double bonds while P(UA)-2 contained both urethane and acrylate structures and was crosslinked by reacting the NCO groups with the OH groups. Both tensile strength and adhesive strength for IPN coatings exhibited higher values than those of their original components—P(UA)-1 and P(UA)-2. The morphologies of IPN coatings as determined by TMA and SEM indicated that the improved molecular mixing in these IPNs were due to interpenetration between the respective networks.

ACKNOWLEDGMENTS

The authors wish to express their appreciation to Ms. Montserrat Molins of the Polymer Institute, University of Detroit, for her assistance in the testing of the coating films and to Mr. Carmel Harkins, Department of Biology, Wayne State University, for his contributions for the SEM micrographs.

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Reactions of n-Butanol with an Alkyd Resin During Heat-Aging: An Analytical Study

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and

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Water-soluble and high-solids alkyds and polyesters are commonly sold in strong, oxygenated solvents. Primary alcohols have been used, but films with slow dry and poor water resistance resulted from unwanted reaction between the alcohol and the resin.

The purpose of this work was to identify the products formed by the reaction of n-butanol with an alkyd resin during heat-aging. An alkyd resin was dissolved in n-butanol and stored at 185°F for two weeks. The volatiles were removed by vacuum distillation and analyzed by gas chromatography. The resin was fractionated by gel permeation chromatography and analyzed by mass spectrometry.

INTRODUCTION

Water-soluble alkyds have frequently suffered from slow dry and poor water resistance. This condition accelerates on aging but may not occur when the resin is first synthesized.[1,2] Bouboulis[3] showed that storing the resin in n-butyl alcohol before the addition of any water could cause reduction in viscosity and molecular weight, as well as slower dry time and poorer water resistance of the cured film. He showed that this behavior could be avoided or minimized by replacing n-butanol with secondary butyl alcohol and he showed the effect of butoxy ethanol lay somewhere between normal butanol and secondary butanol. Engel[4] confirmed this and demonstrated a molecular weight shift from high molecular weight material to low molecular weight material during the aging of the alkyd in the presence of alcoholic solvents. He ranked certain alcohols and glycol ethers in order of their decreasing reactivity with the resin; generally from primary to secondary to tertiary.

The reaction appears to be an alcoholysis in which the alcoholic solvent exchanges places with the polyols in the ester groups of the resin to form low molecular weight species. The reaction starts when the solvent is added to the hot resin in the resin factory and continues during storage until the resin is eventually let down with water at which time water becomes the predominant solvent.

The objective of this work was to try to isolate and identify the low molecular weight species formed. The subject is timely for high-solids alkyds and polyesters as well as for the water-reducible variety because the use of strong, oxygenated solvents is an attractive way to keep viscosity low in high-solids technology.

PART I—EXPERIMENTAL

Preparation of Alkyd

A ten gallon batch of short oil alkyd was prepared from the components in *Table* 1. The Pamolyn® 200 fatty acids are approximately 20% oleic acid, 70% linoleic acid, and 10% conjugated linoleic acid. An electrically heated Brighton Reactor was used. It was equipped with a steam heated partial condenser to return refluxing glycol to the reactor while water was removed. A nitrogen stream was used throughout the reaction. Reactants A, B, C, D, E

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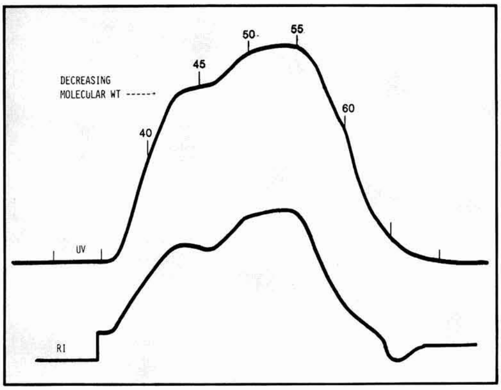

Figure 1—GPC of alkyd before heat-aging

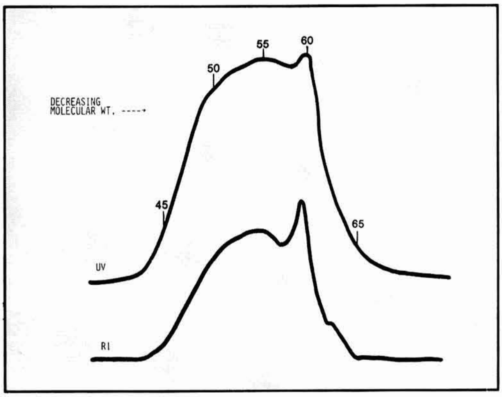

Figure 2—GPC of alkyd heat-aged at 85° C (185° F) for two weeks

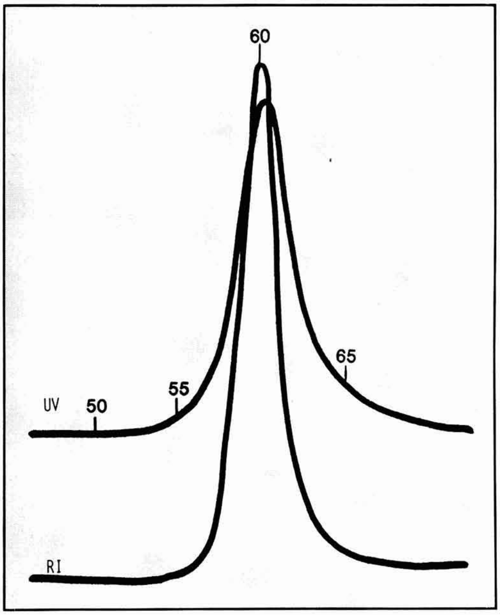

Figure 3—GPC of standard sample of butyl oleate

were loaded into the reactor and heated to a maximum temperature of 245°C (475°F). Heat input was adjusted so that the temperature at the top of the partial condenser did not exceed 100°C (212°F). The reaction was continued about six hours and then cooled to 174°C (345°F). At this point the acid value was 16.5. The trimellitic anhydride was added and the temperature was held at 168°C (335°F) for about 2½ hours without the partial condenser and then cooled. The determined acid value at this point was 43. A 1200 gram sample was removed and diluted to 73.5% solids in commercial grade normal butyl alcohol.

Accelerated Heat-Aging and Vacuum Stripping

Approximately one pint of the sample was transferred into a glass stoppered flask and the resin degassed of air by passing nitrogen through it. The sealed flask was placed in an oven at 85°C (185°F) for two weeks. After two weeks, the sample was removed and cooled to room temperature and opened. A nitrogen blanket was maintained over the resin. Fifty grams of the heat treated sample and 50 g of the control that was not heat treated were vacuum stripped. The alkyd was warmed to 85°C in an oil bath towards the conclusion of the vacuum stripping at approximately 0.5 torr. The volatiles were collected in a container cooled by CO_2 (solid) in isopropanol. Weights of volatiles and alkyd residues were determined. The volatiles were analyzed by gas chromatography.

Gas Chromatography of Volatiles

The volatiles from both fractions were analyzed using a Tracor gas chromatograph and a 3380 Å Hewlett Packard Integrator recorder. The column material was 3% OV-1 on Supelcoport,® and the carrier gas was nitrogen.

The retention time of a standard sample of butyl oleate was compared with the unknowns. Unknowns spiked with butyl oleate were also tested.

Gel Permeation Chromatography

The two samples of alkyd, the control and the heat-aged, were analyzed by gel permeation chromatography (GPC). The analyses were performed on a Waters 301 liquid chromatograph. The columns used were μ Styragel® 10^6, 10^5, 10^4, 10^3, 500, and 10^2. The solvent was tetrahydrofuran (THF) at 1mL/min. A UV detector at 254 nm and refractive index detector were employed. The

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Supelcoport is a trademark of Supelco, Inc.
Styragel is a registered trademark of Waters Associates.

Table 1—Short Oil Alkyd Resin Composition

A	Pamolyn 200 Fatty Acids
B	Trimethylolethane
C	Neopentyl glycol
D	Isophthalic acid
E	Benzoic acid
F	Trimellitic anhydride

volume counter was connected to the UV detector. The columns were calibrated with polystryene standards. Butyl oleate was included as a standard.

RESULTS AND DISCUSSION

Vacuum Stripping

The alkyd resin after heat-aging was lower in viscosity than the control. The amount of strippable solvent, however, was considerably less, 8 vs 22 wt %. This indicates that considerable alcoholysis occurred. The butyl alcohol had esterified in the polymer and was not free and removable. The stripped alkyds were hard glasses but the heat-aged alkyd was softer.

Gas Chromatography

The gas chromatography analyses of volatiles indicated essentially the same composition from the control as from the heat-treated speciman (*Table* 2). The volatiles from the control were 99.3% n-butyl alcohol while the volatiles from the alkyd after heat treatment were 97.8% n-butyl alcohol. Comparison with a standard sample of butyl oleate and the spiked samples indicated that compounds similar to butyl oleate were not obtained in the volatiles. The vacuum stripping temperature was not high enough. Butyl oleate boils at 217°C at 15 mm Hg.

Gel Permeation Chromatography

The gel permeation chromatograms of the alkyd before and after heat treatment are shown in *Figures* 1 and 2, respectively. Considerable destruction of the polymer is indicated. The weight average molecular weight decreased from 45,300 to 6,260 on heat-aging and the number average molecular weight decreased from 1670 to 960. The heterogeneity ratio $\overline{M}w/\overline{M}n$ decreased from 27.1 to 6.5. The low molecular weight peak on *Figure* 2 appears to be a fatty acid ester of butanol. Its elution volume was 59.5 mL compared to a butyl oleate standard of 60.2 mL (see *Figure* 3). The low molecular weight peak appears to be aliphatic rather than aromatic because it does not have a strong UV absorption while it does have a strong peak on the refractive index scale.

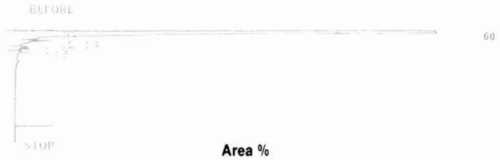
PART II—EXPERIMENTAL

GPC Fractionation and Mass Spectrometry

The low molecular weight peak on *Figure* 2 was isolated by GPC fractionation and analyzed by mass spectrometry.

A 1 g sample of the heat-aged alkyd was diluted with 2 mL of THF. The Waters 301 liquid chromatograph was used as before with the same Styragel columns. THF was used as the eluant at 1.1 mL/min. Dual detectors, refractive index, and UV at 254 nm were again used. The volume counter was connected to the UV detector. Multiple injections of the prepared sample solution were made. The volume counts between 58 mL and 66 mL were collected. Approximately half of the 1 g prepared sample was used (550 mg). The collected eluant was concentrated

Table 2—Gas Chromatographs of Volatiles From the Alkyd Before and After Heat-Aging At 85°C (180°F) for Two Weeks



RT	Type	Area	Area %	
60		172558	99.27%	= n-Butyl Alcohol
1 14	T	653	375 7	= Component A
1 53	T	429	246 8	= Component B
1 82		184	105 9	= Component C

HP 3380A
DLY OFF Stop 10 Reject 100
MV/M 30 Attn 4

RT	Type	Area	Area %	
60		177711	97.8%	= n-Butyl Alcohol
92	T	2402	1.322	= Component A′
1 14	TM	818	450 2	= Component B′
1 26	TM	288	158 5	= Component C′
1 54		468	254 8	= Component D′

HP 3380A
DLY OFF Stop 5 Reject 100
MV/M 30 Attn 4

Table 3—Identifiable Mass Ions from First Mass Spectrogram

m/e	Relative Intensity[a]	Proposed Fragment
56	100.00	$(CH_2)_4$
57	79.93	C_4H_9
73	27.05	C_4H_9O
265	14.37	$C_{17}H_{33}CO$
338	2.80	$C_{17}H_{33}COOC_4H_9$
55	70.49	Characteristic of an alkene
69	70.15	
83	81.18	
97	58.11	
111	34.60	

(a) Based on the most intense peak (m/e 56) arbitrarily set at 100.

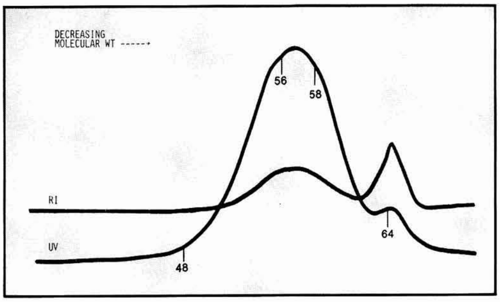


Figure 4—GPC of first fractionation of the low molecular weight peak of the heat-aged sample

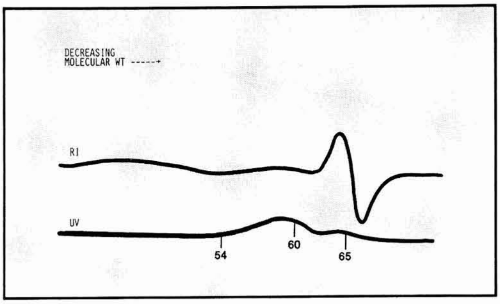


Figure 5—GPC of final fractionation of the low molecular weight peak of the heat-aged sample

by allowing the THF to evaporate in a hood draft overnight. The residue was redissolved in THF and the procedure was repeated, this time collecting eluant between 60 and 64 mL volume counts. GPC curves on the first and final fractionation are shown in *Figures* 4 and 5.

The collected fraction (*Figure* 5) was concentrated, placed in a gold cup, and analyzed in a Varian MAT Ch-4 mass spectrometer. As the temperature was increased from ambient to 400°C two distinct compound fractions were observed. These mass spectrograms are shown in *Figures* 6 and 7.

RESULTS AND DISCUSSION

By repeated injections and fractionations, a sample was obtained that contained very little high polymer and was mainly the low molecular fragment which was formed during heat-aging. In *Figure* 5 two components are seen, one at 64.5 mL and one at 59.5 mL. The mass spectrograms of these two components are shown in *Figures* 6 and 7. A listing of the major ion fragments associated with mass spectrograms is set forth in *Tables* 3 and 4. The ion fragment pattern of the low molecular weight fractions are indicative of an aliphatic hydrocarbon because many of the ions differ by a mass of 14, i.e., a CH_2 unit. In *Figure* 6 and *Table* 3, a parent ion appears at m/e = 338 which corresponds to butyl oleate. Also, the mass ion at m/e = 265 corresponds to a $C_{17}H_{33}CO$ fragment. Two of the most intense peaks in this spectrum correspond to the polymethylene fragment, $(CH_2)_4$, m/e = 56, and the butyl radical, C_4H_9 m/e = 57. The butoxide ion, C_4H_9O m/e = 73 is also a strong peak. There was only 1.06% of the linoleic butyl ester m/e = 336 and 2.87% of linoleic radical $C_{17}H_{31}CO$ m/e = 263. One might expect more linoleic because the fatty acids were 70% linoleic. A possible explanation of this is that the linoleic radical containing the diolefinic structure would be less stable and would tend to fragment more. The series of ions 55, 69, 83, 97, and 111 are characteristic of an alkene and probably originated from the linoleic portion of the alkyd. Absolute separation was not obtained and ions derived from other structures are present.

The second fraction from the mass spectrometry analysis was much more complicated (*Figure* 7). The two largest ion peaks in this fragment were benzoic acid—C_6H_5CO m/e = 105 and isophthalic acid $HO_2CC_6H_4CO$ m/e = 149. Some of the other possible ion fragments associated with the mass spectrograph of the second fragment are given in *Table* 4. Most of them are benzoic and isophthalic esters of neopentyl glycol. These structural assignments were tentatively made, but it must be remembered that the m/e peaks could be represented by several possible structures.

The mass spectrograms showed little evidence of trimethylolethane (TME) and trimellitic anhydride (TMA). One might explain the absence of TME fragments by saying that TME has three possible sites for esterification and is therefore more difficult to free from the polymer than neopentyl glycol. We find it difficult to use the same reasoning to explain the lack of trimellitic anhydride fragments because the average degree of esterification of the TMA in this polymer, as measured by

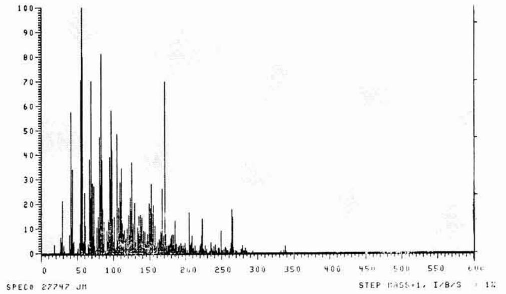


Figure 6—First mass spectrogram of the low molecular weight material

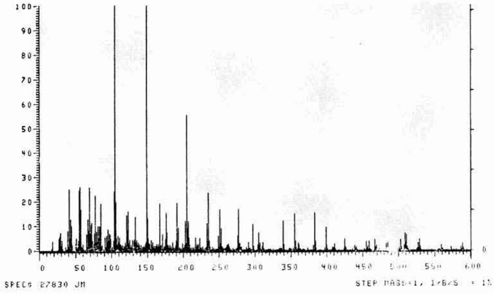
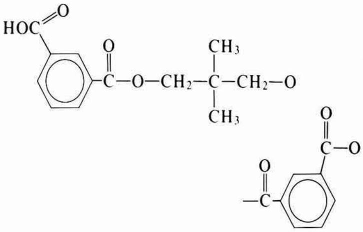
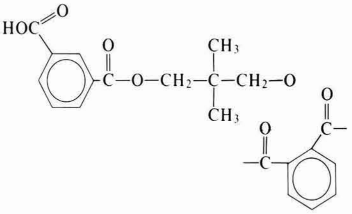
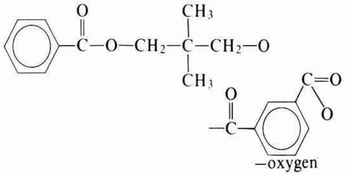
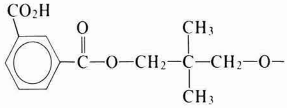
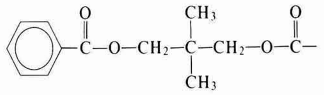
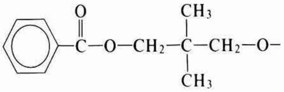
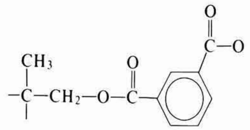
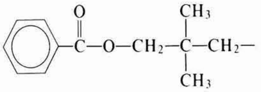
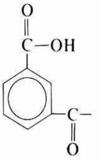
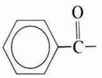


Figure 7—Second mass spectrogram of the low molecular weight material

Table 4—Possible Mass Ions from Second Mass Spectrogram

Ion Fragment	Relative Intensity[a]	m/e
	9.61	399
	15.53	383
	15.13 12.17	355 339
	16.7	251
	23.62	235
	12.31	207
	55.59	205
	19.60	191
	100.00	149
	100.00	105
C_4H_9	16.62	57
C_4H_8	26.14	56

(a) Based on the most intense peaks (m/e 105 and 149) arbitrarily set at 100%.

acid value before and after the addition of the TMA, is less than two ester groups per molecule. But TMA does constitute only five percent by weight of the polymer and only one third as many moles as neopentyl glycol. Therefore, TMA fragments would be more difficult to find. Additional experiments would be necessary to prove and explain the absence of these trifunctional materials.

CONCLUSIONS

(1) Thermal aging of an alkyd in n-butanol causes considerable degradation of the polymer as evidenced by the decrease in molecular weight.

(2) A major reaction during the thermal aging of an alkyd in n-butanol is an alcoholysis resulting in the formation of butyl esters of the fatty acids, e.g., butyl oleate, butyl linoleate, etc.

(3) The polymer fraction separated by GPC fractionation contained considerable benzoic and isophthalic acid fragments attached to neopentyl glycol.

(4) No major mass fragments associated with either of the trifunctional starting materials; trimethylolethane or trimellitic anhydride were observed.

ACKNOWLEDGMENT

Thanks to Irene Beran for preparing the manuscript, to Ed Ferlauto for editing, to Rollie Boettcher for slides, and to the Valspar Corp. for permission to publish the work.

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Hydrolytic Stability of Oligoesters In Simulated Water-Reducible Coating Formulations

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Twelve oligoesters were prepared by partial esterification of trimellitic anhydride (TMA) and of "dimer acid" with 1,6-hexanediol, neopentyl glycol, 1,4-cyclohexanedimethanol, and 1,4-cyclohexanediol. Solutions of these oligomers in water/n-propoxy isopropanol were adjusted to pH 8.2 to 8.8 with N,N-dimethylethanolamine and were aged at room temperature, at 120° F (49° C) and at 135° F (57° C). Hydrolytic stability was estimated by weekly titration of alkaline oligomer solutions to observe change in acid number. Key results were:

(1) Partial ester of TMA suffered substantial hydrolysis within a few weeks at 120° C and 135° F. At room temperature they were fairly stable, exhibiting increases in acid number of 1% to 19% in 10 weeks, and of 5% to 23% in 30 weeks.

(2) Partial esters of "dimer acid" hydrolyzed much more slowly than partial esters of TMA. Acid numbers increased only 6% to 12% after 8 to 10 weeks at 135° F.

(3) The data did not yield clear-cut correlations of alcohol structure with stability.

Model compounds were studied to gain insight into the effect of alcohol structure on hydrolytic stability. Monoesters were prepared by partial esterification of phthalic anhydride with four monoalcohols. Solutions of these monoesters in water/n-propoxy isopropanol were adjusted to pH 8.1 to 8.3 with N,N-dimethylethanolamine and aged at 135° F. Hydrolytic stability was strongly influenced by alcohol structure in the following order: cyclohexanol (forms the most stable phthalate monoester); neopentyl alcohol; cyclohexyl methanol; and n-hexanol (forms the least stable phthalate monoester).

INTRODUCTION

Improved water-reducible coatings are a top priority research objective of the coatings industry.[1] Carboxy/hydroxy functional polyester resins are widely used as binders for such coatings. The basic chemistry by which such resins are "solubilized" with amines is well known.[2]

It is also well known that coatings formulated from such resins often have poor package stability, a problem generally attributed to slow hydrolysis of the ester linkages of the resin. In 1975 Turpin[3] provided useful guidelines for relating hydrolytic stability to steric factors in resin structure. His conclusions were based mainly on theoretical considerations and studies of model com-

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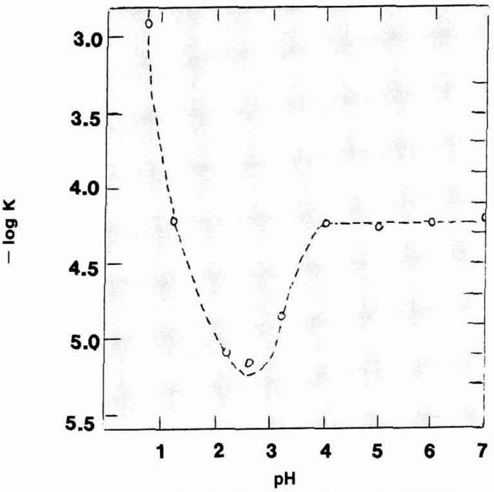


Figure 1—Hydrolysis of methyl hydrogen phthalate at 109° C as a function of pH

pounds. In view of the growing use of carboxy/hydroxy functional resins and of the known potential for problems, surprisingly few studies of hydrolytic stability of these resins in simulated coatings formulations have been published since. This is especially surprising because ester hydrolysis has been one of the most intensively studied organic reactions.[4]

The intent of this research was to estimate how much hydrolysis can actually be expected under paint-drum conditions and to confirm Turpin's predictions of how resin structure affects stability. Initially the scope of this study was limited to oligoesters prepared by partial esterification of trimellitic anhydride (TMA) and of "dimer acid" with four representative diols. Later the scope was expanded to include study of model compounds prepared from phthalic anhydride and monoalcohols.

Table 1—Properties of Oligoesters

TMA Monoesters

Diol Type	Percent Solids	Acid Number[a]
Neopentyl	69	297
1,6-Hexane	59	297
1,4-Cyclohexanedimethyl	60	285
1,4-Cyclohexyl	50	292

TMA Diesters

Diol Type	Percent Solids	Acid Number
Neopentyl	68	132
1,6-Hexane	57	146
1,4-Cyclohexanedimethyl	73	97
1,4-Cyclohexyl	76	115

Dimer Acid Monoesters

Diol Type	Percent Solids	Acid Number
Neopentyl	79	53
1,6-Hexane	77	51
1,4-Cyclohexanedimethyl	77	71
1,4-Cyclohexyl	78	66

(a) Acid numbers at 100% solids

Turpin identified two types of structural features that favor hydrolytic stability of esters:

(1) *Presence of Steric Hindrance*—"As much alkyl substitution as possible at the alpha and beta carbons on either side of the ester group" improve hydrolysis resistance. Turpin's thinking was based on Newman's rule of six[5] and on his own studies of model compounds.

(2) *Absence of Anchimeric Effects*—Nearby nucleophiles such as carboxy groups can act as intramolecular catalysts for ester group hydrolysis. An example was provided by Bender, et al.,[6] who demonstrated that the rate of hydrolysis of methyl hydrogen phthalate at 109° is much faster at pH 7 than at pH 2.5 (*Figure* 1).

Based on extrapolations of Bender's data and his own study of phthalate half esters, Turpin concluded that the anchimeric effect can have a strong adverse effect on hydrolytic stability in the pH range 7 to 8.

Water-reducible coatings are often formulated at pH 8.2 to 8.6. A small degree of hydrolysis creates —COOH groups, causing a downward drift of pH. Normally the rate of hydrolysis would drop as pH approaches 7. However, as Turpin points out, the anchimeric effect can keep it going throughout the pH range 8 to 4, causing massive deterioration of the resin.

A more detailed literature survey and description of this work can be found in a recently published thesis.[7]

EXPERIMENTAL

Materials

Reactants were obtained from laboratory chemical supply houses, except for 1,4-cyclohexanediol (SSF Dottikon) and 1,6-hexanediol (Celanese). Solvents were commercial grades.

Synthesis of TMA and "Dimer Acid" Esters

All reactions were carried out in a one-liter breakaway flask equipped with N_2 inlet, thermometer, mechanical stirrer, and Dean-Stark trap condenser combination. The flask was charged with equimolar amounts of diol and "dimer acid" for dimer acid monoesters, with 1.04:1.00 molar ratios of diols to TMA for TMA monoesters, and a 2.75:1.00 molar ratio of diol to TMA for TMA diesters.

The charging procedure for TMA was to preheat the reactor to 120°C and to add the ingredients gradually with stirring at 120°C to keep them molten. Dimer acid cooks were charged at 25°C. In both cases the reactors were purged with N_2 for 30 minutes before heat up.

All reactions were slowly warmed to 175–180°C and held at this temperature for one hour. The Dean-Stark trap was filled with xylene, and a small amount of xylene, 3% of the total charge, was added to the reaction mixture to regulate temperature and assist water removal.

The resulting mixture was warmed to 190°C and refluxed. Approximately 1.0 g samples of the resin were removed from the reaction pot using a deflagration spoon, dissolved in 60 mL of xylene and titrated to the phenolphthalein end point with alcoholic standardized potassium hydroxide solution. After achieving an acid number which was close to the theoretical acid number for mono- or diester formation the reaction was cooled to about 140°C and then thinned to 50–80% solids with n-propoxy isopropanol (*Table* 1).

Acid Number Determination of Oligoesters

Samples containing TMA were dissolved in methanol (60 mL) while samples containing dimer acid were dissolved in acetone. The resulting solutions were titrated to a phenolphthalein endpoint using standardized potassium hydroxide in methanol for the TMA samples and standardized potassium hydroxide in distilled water for the dimer acid samples.

For the TMA samples acid number calculations were established by back titration with hydrochloric acid solution.

Synthesis of Phthalate Esters

Fifty grams of mono-alcohol, 57 g of phthalic anhydride, and 20 g of anh. pyridine were combined in a 500 mL 3-neck round-bottom flask equipped with mechanical stirrer, thermometer, and a reflux condenser. Reagents were warmed to a mild reflux (90–95°C) using a water bath. The reaction was complete in one hour. The reaction mixture was allowed to cool slowly to 40°C. One hundred mL of spectro-grade acetone were added to the reaction mixture which was stirred for 5 mins followed by the slow addition of 55 mL of concentrated hydrochloric acid. Approximately 300 g of ice were added to the mixture which was stirred for two hours at room temperature.

Alicyclic mono-alcohols gave a white precipitate which was collected by suction filtration, air dried, and recrystallized from glacial acetic acid. Aliphatic mono-alcohols gave an oily suspension. These phthalate esters were recovered by:

(1) Extracting the suspension 3X with 500 mL of chloroform
(2) Combining chloroform solutions and drying with anh. $MgSO_4$ and filtering
(3) Vacuum stripping
(4) Dissolving residue in petroleum ether, drying with anh. $MgSO_4$, and filtering
(5) Vacuum stripping.

The structures of the phthalate esters were verified by NMR (see Appendix). The spectra indicated that the products were over 90% pure.

Preparation of Oligomer Solutions for Hydrolysis Study

Approximately 600 g samples of each oligoester and 200 g samples of each phthalate ester were prepared such that each oligomer solution contained 20% solids and 80% volatiles, of which 20% was n-propoxy isopropanol and 80% was water. The solutions were mixed for 5–15 mins to achieve homogeneity. At this stage each solution was adjusted to a pH of approximately 8.2 to 8.8 by adding N,N-dimethylethanolamine. The properties of the solutions are shown in *Table* 2.

Hydrolysis Study

Oligomer solutions were divided into three 200 g portions. One set was aged in brown bottles at room temperature and the other two sets were aged in pressure bottles stored in ovens at 120° F (49° C) and 135° F (57° C). Solutions containing phthalate esters were aged in pressure bottles stored in a 135° F oven.

Hydrolysis of Dimer Acid-NPG Monoester Aged at 135° F as a Function of Diluent Concentrations

Oligomer solutions containing 20% dimer acid-NPG monoester and 80% volatiles were prepared such that the percent volatiles was adjusted to give ratios of n-propoxy isopropanol:water of 20:80, 50:50, and 80:20, respectively. The pH of each oligomer solution was adjusted to 8.2–8.5 by adding N,N-dimethylethanolamine. The solutions were then aged in pressure bottles stored in 135° F oven.

Stability Testing

Acid numbers were determined in triplicate by taking 2.5 g of oligomer solutions, adding 10 drops of 1.0%

Table 2—Properties of Oligomer Solutions at Start of Hydrolysis

Sample #	% Solids	% Neutralization	pH	Acid Number
1	18.1	129	8.5	288
2	18.2	118	8.2	300
3	18.3	120	8.2	280
4	18.0	133	8.8	297
5	19.0	131	8.8	127
6	19.1	117	8.2	137
7	19.4	119	8.2	86
8	19.3	121	8.3	115
9	19.7	56	8.5	50
10	19.7	62	8.6	49
11	19.6	81	8.2	70
12	19.6	58	8.2	66
13	18.7	112	8.2	186
14	18.6	110	8.3	224
15	18.6	102	8.2	223
16	18.6	109	8.3	217

Sample #	Oligoester
1	TMA-Neopentyl glycol monoester
2	TMA-1,6-Hexanediol monoester
3	TMA-1,4-Cyclohexanedimethanol monoester
4	TMA-1,4-Cyclohexanediol monoester
5	TMA-Neopentyl glycol diester
6	TMA-1,6-Hexanediol diester
7	TMA-1,4-Cyclohexanedimethanol diester
8	TMA-1,4-Cyclohexanediol diester
9	Dimer Acid-Neopentyl glycol monoester
10	Dimer Acid-1,6-Hexanediol monoester
11	Dimer Acid-1,4-Cyclohexanedimethanol monoester
12	Dimer Acid-1,4-Cyclohexanediol monoester
13	n-hexyl phthalate
14	Neopentyl phthalate
15	Cyclohexyl phthalate
16	Cyclohexylmethyl phthalate

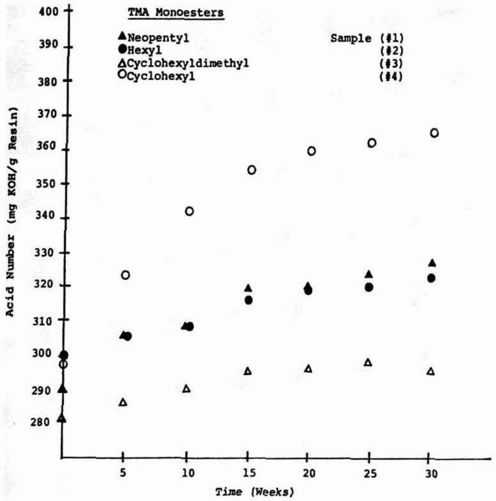


Figure 2—Rate of hydrolysis of oligoesters (samples 1-4) aged at room temperature

phenolphthalein in methanol and titrating to a pink end point with standardized methanolic potassium hydroxide. Reproducibility was good.

Hydrolytic stability was observed by measuring the change in the acid number. Oligomer solutions derived from TMA and dimer acid oligoesters were aged at room temperature for 30 weeks, and at elevated temperatures for 10 weeks. Acid numbers were determined weekly for the solutions aged at elevated temperatures and at five week intervals for those aged at room temperature.

Solutions containing phthalate half-esters were aged at 135°F for approximately 1000 hours. Acid numbers were determined at regular intervals not exceeding 94 hours. The results are presented graphically in *Figures* 2 through 12. Raw data were published elsewhere.[7]

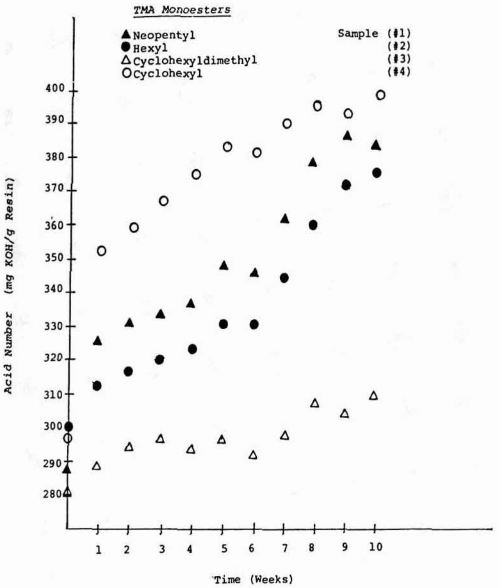


Figure 3—Rate of hydrolysis of oligoesters (samples 1-4) aged at 120°F

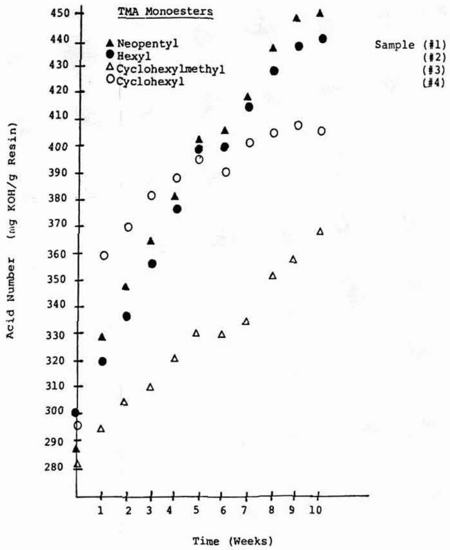


Figure 4—Rate of hydrolysis of oligoesters (samples 1-4) aged at 135°F

In view of the surprising results obtained from mono- and diesters of TMA-1,4-cyclohexanediol and TMA-1,4-cyclohexanedimethanol, the hydrolysis of these samples was repeated for six weeks at 135°F. The results were similar to those obtained earlier.

DISCUSSION

The oligomers studied here contain structures typical of polyester and alkyd resins used in water-reducible coatings. Molecular weights were made lower so that fragments tend to stay in solution throughout hydrolysis

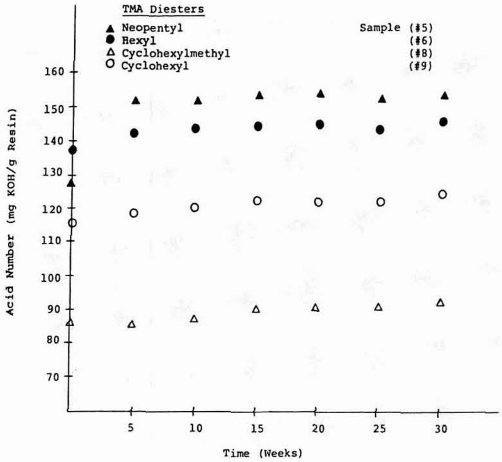


Figure 5—Rate of hydrolysis of oligoesters (samples 5-9) aged at *room temperature*

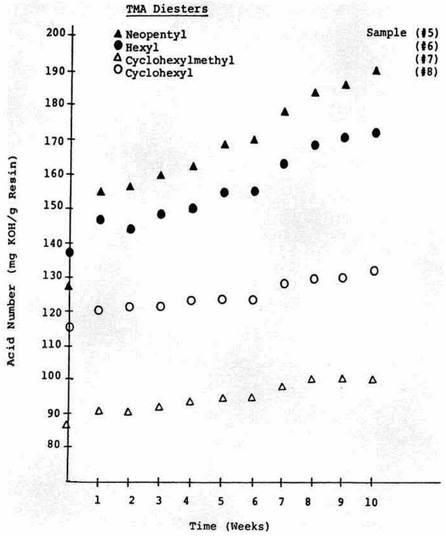


Figure 6—Rate of hydrolysis of oligoesters (samples 5-8) aged at 120° F

testing. Test conditions were chosen to simulate the conditions to which such resins are exposed in formulated water-reducible industrial coatings. It is believed that the choice of solvents (20/80 water/n-propoxy isopropanol), amine (dimethylaminoethanol) and pH (8.2 to 8.8) is typical of many such formulations, and the temperatures (room temperature, 120° and 135°F) represent typical paint storage and accelerated stability testing conditions.

At the onset of this study it was necessary to decide whether to try to neutralize the oligomers to a target pH,

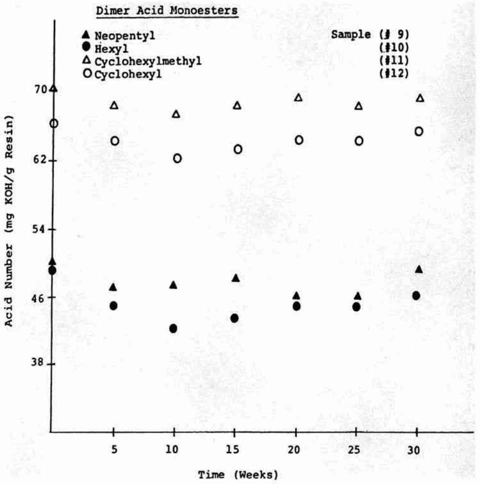


Figure 8—Rate of hydrolysis of oligoesters (samples 9-12) aged at room temperature

to a target degree of neutralization, or both. The target pH was chosen. The consequences of this choice were that the degree of neutralization varied widely, as shown in *Table* 2. It was necessary to add about 1.2 equivalents of amine per equivalent of —COOH to raise the pH of the TMA oligomers to 8.2. However, the dimer acid oligomers, being weaker acids, needed only 0.55 to 0.8 equivalents of amine to reach this pH. As pointed out by a reviewer, it is widely believed that the rate of polyester hydrolysis increases sharply if degree of neutralization exceeds 100%. Therefore, it is possible that if the experiments had been done using a predetermined degree

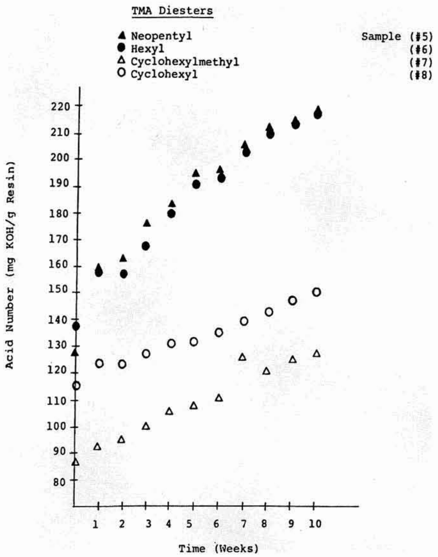


Figure 7—Rate of hydrolysis of oligoesters (samples 5-8) aged at 135° F

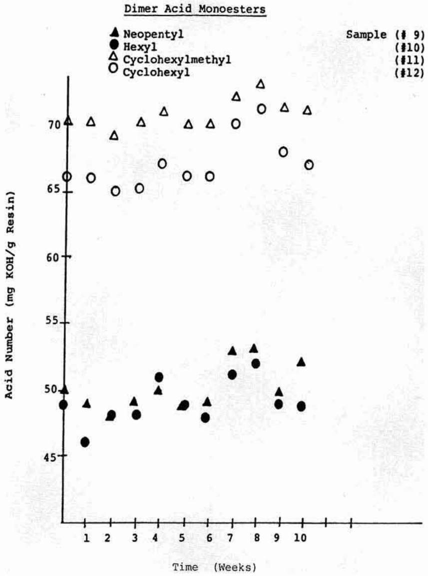


Figure 9—Rate of hydrolysis of oligoesters (samples 9-12) aged at 120° F

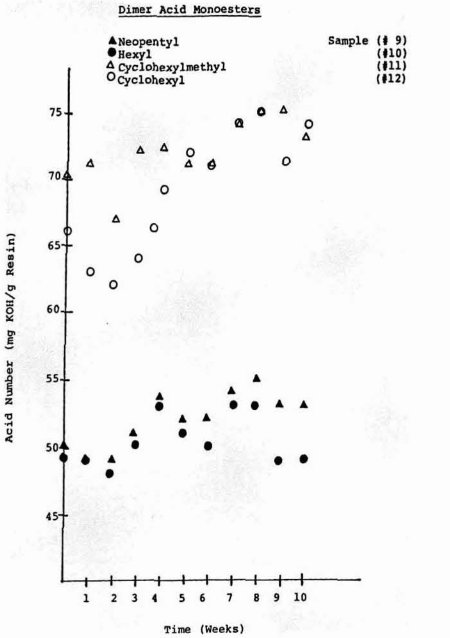


Figure 10—Rate of hydrolysis of oligoesters (samples 9-12) aged at 135°F

of neutralization and a variable pH the very large differences in stability between TMA and dimer acid oligomers might have been reduced.

Thus the data in *Figures* 2 through *10* provide a broad indication of how rapidly polyester and alkyd resins might deteriorate in coatings formulated to an initial pH of 8.2–8.8. The trends which emerge are:

(1) Dimer acid esters undergo no hydrolysis (within experimental error) at temperatures up to 120°F (*Figures* 8 and 9) and little hydrolysis at 135°F (*Figure* 10). Even those made from primary alcohols are stable.

(2) TMA partial esters suffer substantial hydrolysis at 120° and at 135°F (*Figures* 3, 4, 6, and 7). For example the acid number of Sample #1 (TMA/NPG monoester) increased from 288 to 380 in ten weeks at 120°F. This increase is estimated to correspond to hydrolysis of roughly 2/3 of all ester groups present. This indicates that water-reducible coatings containing NPG polymers tipped with TMA monoester may undergo massive deterioration within a few weeks at 120°F or above.

(3) TMA diesters appear appreciably more stable than TMA monoesters (compare *Figures* 3 and 6 or 4 and 7), but are still vulnerable.

(4) Two samples, #4 and #5, displayed a rapid early increase in acid number and then leveled off as the test progressed. This behavior can be attributed to the high initial pH, 8.8, of these samples, although other factors may be at work. Presumably the rate of hydrolysis slows because the carboxy groups formed cause pH to drop. It appears that the rate of hydrolysis in very sensitive to pH in the 8 to 9 range, as predicted by Turpin.[3]

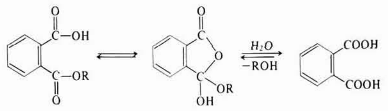
(5) Disregarding Samples #4 and #5, it appears that TMA diesters undergo very little hydrolysis in 30 weeks at room temperature (*Figure* 5). TMA monoesters undergo appreciable hydrolysis at room temperature (*Figure* 2), but the rate is slow enough that package stability might be marginally adequate for practical purposes.

In summary it appears that current formulating practices for water-reducible industrial coatings may provide acceptable package stability *providing* pH is held below 8.4 and elevated temperatures are avoided.

The cost of controlling shipping and storage temperatures is often prohibitive, and the need for exacting control of pH is an undesirable complication. Therefore, it is desirable to build as much hydrolytic stability into the resins as possible. Stable resins will yield better package stability under adverse conditions and, quite probably, more durable coatings films.

Turpin's guidelines (see "Background") stressed the importance of steric and anchimeric effects. The stability of all dimer acid partial esters, including those made from unhindered alcohols such as hexanediol, indicates that avoidance of structures in which anchimeric effects operate is sufficient to impart good package stability.

However, use of acid anhydrides to tip polyester and alkyd resins with carboxy groups has become a standard method for placing hydrophylic sites in the resin.[8] This method yields structures that are almost ideal for the anchimeric effect [equation (1)].



(1)

TMA is a popular reactant for the purpose because each mole is capable of adding two carboxy groups [equation (2)].

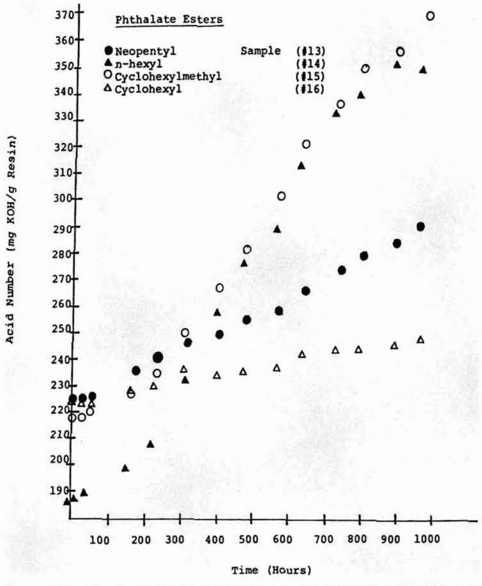


Figure 11—Hydrolysis of phthalate esters (samples 13-16) aged at 135°F

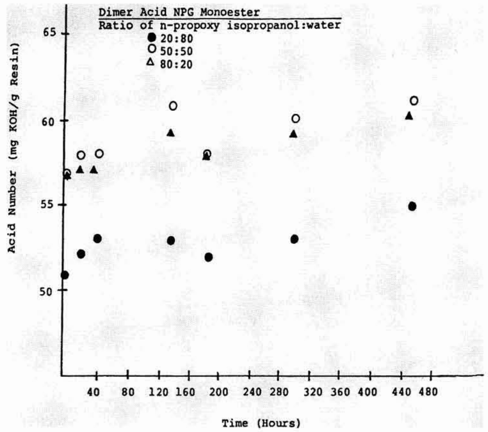
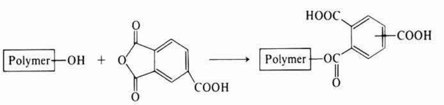


Figure 12—Rate of hydrolysis of dimer acid-NPG monoester aged at 135° F



(2)

Thus the structure which resin chemists find most useful for promoting water miscibility is quite unattractive from the standpoint of package stability.

Because there are few attractive alternatives to the use of anhydrides, other means must be sought to build hydrolytic stability into the resin. Use of sterically hindered polyols is a logical step. The question is, "how well will it work?"

The results, summarized in *Table* 3, with phthalate half-ester model compounds offer hope that it will work well. In this Table an average rate of increase in acid number is shown for each half-ester. The time interval from 160 to 895 hours was selected to minimize irregularities in the early stages. The numbers presented correspond to the slopes of the lines in *Figure* 11. They provide an estimate of the relative hydrolytic stability of each half-ester.

Table 3 shows that the hydrolytic stability of these model compounds falls in the order predicted by steric considerations, the most hindered being the most stable. The ten-fold difference in rate between cyclohexanol and n-hexanol is not enormous by the standards of chemical kinetics, but it could be enough to make the difference between satisfactory and unsatisfactory package stability.

However, results with oligomers are much less clear-cut. Examples are shown in *Table* 4.

As shown in *Table* 4, steric hinderance did not correlate with hydrolytic stability of TMA monoesters during the first three weeks at 120°F. Later in the experiment the rates fell closer to the expected order, although some irregularities persisted. The magnitude of the differences was smaller for oligomers than for model compounds. Examination of our data for other TMA oligomers (*Figures* 2 and 4 through 7) lead to similar conclusions. In general it appears that there is little correlation with steric factors during the early stages of the study and only a weak one during the latter stages.

Table 3—Relative Rate of Acid Number Increase Of Phthalate Half-Esters at 139° F

Alcohol	Relative Rate of Acid # Increase in the Interval 160-895 Hrs., mg KOH/g Resin-Week
Cyclohexanol (most hindered)	3.4
Neopentyl alcohol	12
Cyclohexyl methanol	30
n-Hexanol (least hindered)	35

Experimental error may play a role in the irregular results observed, but it is not the whole story. The results were repeatable. Apparently factors other than steric hinderance are at work. While is has not been possible to isolate all the factors which may affect hydrolytic stability of oligomers, it is thought that they may include:

(1) As demonstrated above, hydrolysis rates are quite sensitive to pH in the 8.2 to 8.8 range. The variable degree of neutralization may also play a role. This sensitivity may largely account for the early deterioration of samples #4 and #5, which had the highest pH at the outset. Sample #1 also had higher than average pH and displayed a similar effect.

(2) Small differences in oligomer process or composition might lead to differences in oligomer structure that could significantly affect hydrolytic stability. There are at least two possibilities:

- During polymer preparation, thinning of the melt with hydroxy containing solvents at elevated temperatures might lead to formation of ester groups incorporating solvent. Such ester groups would be less hindered than ester groups derived from hindered diols and might hydrolyze faster, diluting the effect of using hindered monomers. For example, commercial n-propoxy isopropanol contains a modest percentage of 2-(n-propoxy)-1-propanol having relatively reactive primary OH groups. In this study an attempt was made to detect esters derived from solvent spectroscopically, but the results were inconclusive.

Table 4—Rates of Increase in Acid Number Of TMA Monoesters during Different Time Intervals, 120° F

	Rate of Increase in Acid Number	
Diol in Monoesters	**0 to 3 weeks**	**5 to 8 Weeks**
1,4-Cyclohexanediol (most hindered)	23	3.7
1,4-Cyclohexanedimethanol	5.3	3.7
Neopentyl glycol	15	9
1,6-Hexanediol (least hindered)	6.7	10
Diol in Diesters		
1,4-Cyclohexanediol (most hindered)	2.0	2.3
1,4-Cyclohexanedimethanol	2.0	2.0
Neopentyl glycol	11	5.0
1,6-Hexanediol (least hindered)	3.7	4.3

- In preparation of TMA diesters the more hindered diols would be more likely to form 1,4- or 2,4-diesters and less likely to form 1,2-diesters. If this selectivity occurs it would reduce the advantage of using hindered diols because they would have the greatest propensity to form structures vulnerable to the anchimeric effect. The complex subject of esterification rates of TMA has been studied.[8]

(3) Water-reducible polyester and alkyd resins usually form micelles rather than true solutions in 20/80 organic solvent/water blends.[9] This factor could affect the hydrolytic stability because local concentrations of water and of amine in these micelles could be quite different than in the solution as a whole. In this study an attempt was made to determine whether such an effect explains the stability of dimer acid partial esters. It was hypothesized that switching to 50/50 or 80/20 organic solvent/water blends might dissolve the (in this case hypothetical) micelles and increase the rate of acid number build up. However, no such effect was observed (see *Figure* 12).

SUMMARY

Our results and published information lead us to believe that water-reducible polyester and alkyd coatings are now formulated on the borderline of serious package instability problems. Deterioration in the package can be minimized by three expedients:

- Careful control of pH and of degree of neutralization
- Avoidance of even mildly elevated temperatures
- Use of resins in which intramolecular catalysis of ester group hydrolysis (the anchimeric effect) is not possible.

Unfortunately, these expedients are often not feasible in present-day commercial practice. A fourth expedient, which is more economically feasible, is synthesis of resins exclusively from sterically hindered polyols. Our results indicate that this step may modestly improve package stability, but cannot dependably assure satisfactory stability at high pH or elevated temperatures.

ACKNOWLEDGMENT

We wish to thank Valspar, Inc. for a grant which supported this work.

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APPENDIX

NMR SPECTROSCOPY

Neopentyl phthalate—
($CDCl_3$) 1.03 (s,9H,CH_3) 4.07 (s,2H,CH_2) 7.49 (d,2H.Ph) 7.84–8.83 (d,2H,Ph).

n-Hexyl phthalate—
($CDCl_3$) 0.86 (t,3H,CH_3) 1.30 (m,6H,CH_2) 1.66 (m,2H,CH_2) 4.26 (t,2H,CH_2) 7.53 (d,2H,Ph) 7.76 (d,2H,Ph).

Cyclohexyl phthalate—
($CDCl_3$) 0.46–2.13 (m,10H,C_6H_{11}) 4.86 (m,1H,C_6H_{11}) 7.46 (d,2H,Ph) 7.99 (d,2H,Ph).

Cyclohexylmethyl phthalate—
($CDCl_3$) 0.46–1.13 (m,10H,C_6H_{11}) 1.73 (m,1H,C_6H_{11}) 4.17 (d,2H,CH_2) 7.66 (d,2H,Ph) 7.89 (d,2H,Ph).

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Du PONT DBE	–	15	–	12	–	5
PM ACETATE	–	25	–	38	–	8
MEK	–	–	47	50	26	26
N-BUTANOL	–	10	–	–	–	–
TOLUENE	–	–	–	–	61	61
XYLENE	50	50	–	–	–	–
EE ACETATE	50	–	53	–	13	–
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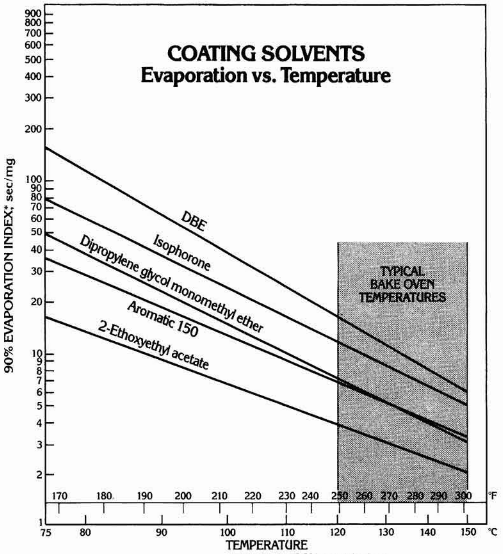

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Chromatographic Characterization Of Epoxy Resins

D.R. Scheuing
The Dexter Corporation*

High performance size exclusion and reverse phase liquid chromatography can be applied to the characterization of epoxy resins of a wide molecular weight range. Oligomer separations differentiate "advancement" epoxy resins, formed by reaction of bisphenol A with a liquid epoxy resin, from "taffy" types manufactured by direct addition of epichlorohydrin to bisphenol A. Reactions of various molecular weight oligomers can be monitored by reverse phase LC. Several types of reverse phase packings are compared in terms of analysis time and selectivity.

INTRODUCTION

Epoxy resins are being used as major components of high performance coatings systems. Modifications of epoxy resins to provide water dispersibility have been reported and use of epoxy resins in solvent based primers continues. Epoxy resins also are important in other areas as structural thermosets such as in the production of "prepregs" for circuit boards.

In recent years, advances in both instrument design and column technology have made liquid chromatography (LC) an increasingly rapid and efficient technique for the characterization of complex oligomeric mixtures such as epoxy resins and other polymers of interest to the coatings industry.[1] The goal of this study was to provide separations of varying degrees of resolution of epoxy resin components for support of research on the reactions of the oxirane groups with hydroxyl and carboxyl functional molecules. Similar studies of the reaction of liquid epoxy resins with amine curing agents in prepreg production have been reported.[2]

EXPERIMENTAL

A Waters Model 244 liquid chromatograph, equipped for gradient elution, having a fixed wavelength ultraviolet detector operating at 280 nm (Model 440) and a differential refractometer (Model R401) were used (Waters Associates, Milford, PA). Tetrahydrofuran (THF) was Omnisolve® chromatography grade, unstabilized.* Water was deionized, filtered through a 0.45 μm pore filter, then passed through a Norganex cartridge for removal of trace organics (Millipore Corp., Bedford, MA). Several different commercially available columns were employed, as described in the text.

The epoxy resins investigated were commercial products, modified as described below. Samples for LC analysis were dissolved in THF at 10 mg/mL concentration. Depending on the sensitivity required, from 5 to 20 microliters were injected.

Chromatograms were plotted, and peak retention times and areas determined by use of a Waters Data Module.

Infrared spectroscopy was used for the identification of peaks separated by reverse phase LC.

A short section of capillary tubing was installed at the exit of the ultraviolet detector for collection of components of interest as they eluted from the chromatograph.

The peak fractions thus collected are reduced in volume to about 100 microliters with a stream of inert gas, then deposited and dried on the faces of a micro-ATR crystal (KRS-5 type). Several micrograms of material analyzed

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Presented at the 61st Annual Meeting of the Federation of Societies for Coatings Technology in Montreal, Que., Canada, October 12, 1983.

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*Omnisolve is a registered tradename of MCB, Inc.

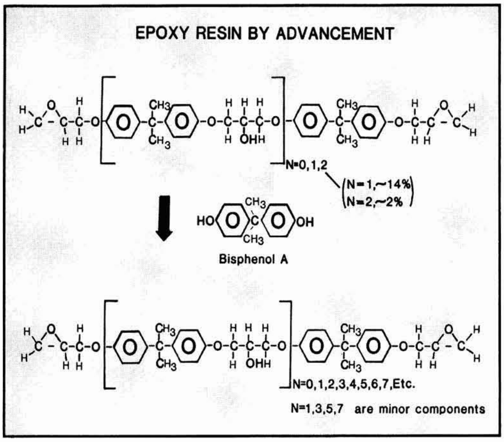

Figure 1—Reactions of the advancement process

in this manner are sufficient to obtain an excellent spectrum on a dispersive spectrophotometer.

A Perkin-Elmer Model 283 Infrared Spectrometer with a Model 3600 Data Station was used for recording and storage of spectra.

CHARACTERIZATION OF EPOXY RESINS BY SIZE EXCLUSION CHROMATOGRAPHY

"Advancement" type epoxy resins, formed by reaction of bisphenol A with a di-epoxide functional resin are common today, compared with the older "taffy" process types, which were manufactured by the direct addition of epichlorohydrin to bisphenol A. Differences in saponifiable chlorine, phenolic hydroxyl content, and other trace impurities between these resin types are well known. A substantial difference in the oligomer distributions also exists.

The formation of "advancement" epoxy resins is shown in *Figure* 1. A low molecular weight liquid epoxy resin is reacted with bisphenol A to advance the molecular weight. From the stoichiometry of this reaction, formation of only even numbered oligomers ($n = 0,2,4,6$) is theoretically possible. Due to the small amount of $n = 1$ oligomer present in the starting liquid resin, a small amount of odd oligomers are found in advancement resins. Advancement resins also are known to contain phenolic hydroxyl terminated species which are commonly determined with titrimetric techniques.[3,4,5]

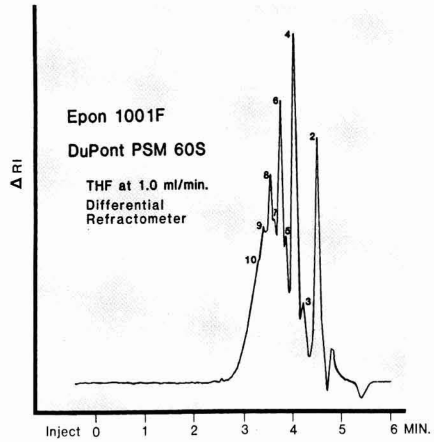

Figure 2—S.E.C. chromatogram of Epon 1001F. DuPont PSM 60S with THF at 1.0 mL/min. Differential refractometer

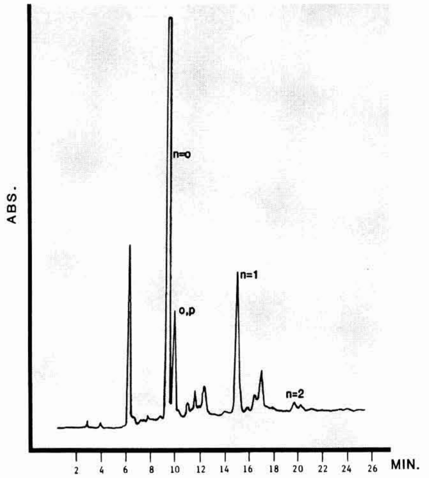

Figure 3—Reverse phase chromatogram of liquid epoxy resin (low molecular weight epoxy) obtained with 10 μm diameter packing. Micro-Bondapak C-18. Gradient: 40-60% THF in H_2O (60 min); Flow rate: 2.0 mL/min. Curve 5. Detection at 280 nm

Chromatographic characterization of advancement resins can be accomplished in a variety of ways depending on the information required. *Figure* 2 shows a size exclusion chromatogram of Epon® 1001F (Shell Chemical).

This chromatogram was obtained using a silica-based "deactivated" size exclusion column from DuPont (PSM 60S) with THF at a flow rate of 1.0 mL/min. Adequate resolution to identify the typical pattern of oligomers exhibited by advancement resins (enriched in even n-mers) is achieved with a single PSM 60S column, rated by the manufacturer as having an exclusion limit of approximately 1×10^4 relative to narrow dispersity polystyrene standards. Hagnauer,[6] Batzer and Zahir,[7] and Antal, et al.[8] have published size exclusion chromatograms in connection with kinetic and quality assurance studies of resins of similar molecular weight. The availability of rigid particle packed columns such as the "PSM" types employed in this study make it possible to rapidly obtain the oligomer distribution of lower molecular weight resins.

CHARACTERIZATION OF EPOXY RESINS— REVERSE PHASE LIQUID CHROMATOGRAPHY

In addition to the high efficiency size exclusion separations possible, the application of reverse phase LC to the characterization of epoxy resins is also very useful.

Reverse phase LC has continued to grow in popularity in recent years because of improvements in column technology which have provided high efficiency columns and reduced analysis times. This mode of LC achieves separation of molecules based on chemical rather than size differences. The differences in partition coefficients of related species between the stationary phase and a mobile phase usually consisting of water and an organic solvent provides the separation. The stationary phase consists of a hydrocarbon chain from 8 to 18 carbon atoms in length, permanently bonded to silica particles ranging from 3 to 10 μm in diameter.

The stationary phase is non-polar while the mobile phase is a very polar water-solvent blend. These conditions are the "reverse" of older chromatographic techniques using silica gel packings (polar) and hexane-chlorinated solvents (non-polar) as mobile phases, hence the term "reverse-phase" LC. Readers new to the technique have a choice of several texts describing liquid chromatography in detail. That of Snyder and Kirkland is particularly complete.[9]

Gradient elution in liquid chromatography involves programming the mobile phase composition, and hence, polarity with time. In the reverse phase separations discussed below, this means that the mobile phase is changed from a more polar, higher water content blend to a less polar, higher solvent content blend.

The chromatograph employed can produce gradient profiles which are linear, concave, or convex. A slightly convex gradient was used to produce the reverse phase separations shown. This gradient features a faster rate of increase in THF concentration early in the run, and a slower rate of increase later, relative to a linear change over the same time period. Curve 5 on the Model 660 solvent programmer was used to generate this gradient.

Changes in gradient shape are powerful tools for optimizing chromatograms once the range of mobile phase composition necessary to elute all components of interest has been established through experiments with linear changes.

Compared with the other common organic mobile phase modifiers, methanol and acetonitrile, THF offers the least amount of selectivity.[10] The separations of impurities in liquid epoxy resins may be improved somewhat if acetonitrile is used instead of THF.[11] However, acetonitrile is not suitable for the analysis of higher epoxy resins due to partial resin insolubility. Since higher molecular weight resins were of primary interest in the studies summarized below, THF was used as the "strong solvent" in the separations developed.

Figures 3 and 4 are the best separations of the components of a liquid epoxy resin and a higher average molecular weight solid epoxy resin (Epon 1001F) that were developed using the column packed with 10 μm diameter particles. *Figures* 5 and 6 show the separations achieved with a higher efficiency column containing 5 μm diameter particles.

The assignments of the major peaks followed that of Schenck and Kayen. The retention time of the peak assigned to n = 0 can be compared with that of a DGEBA

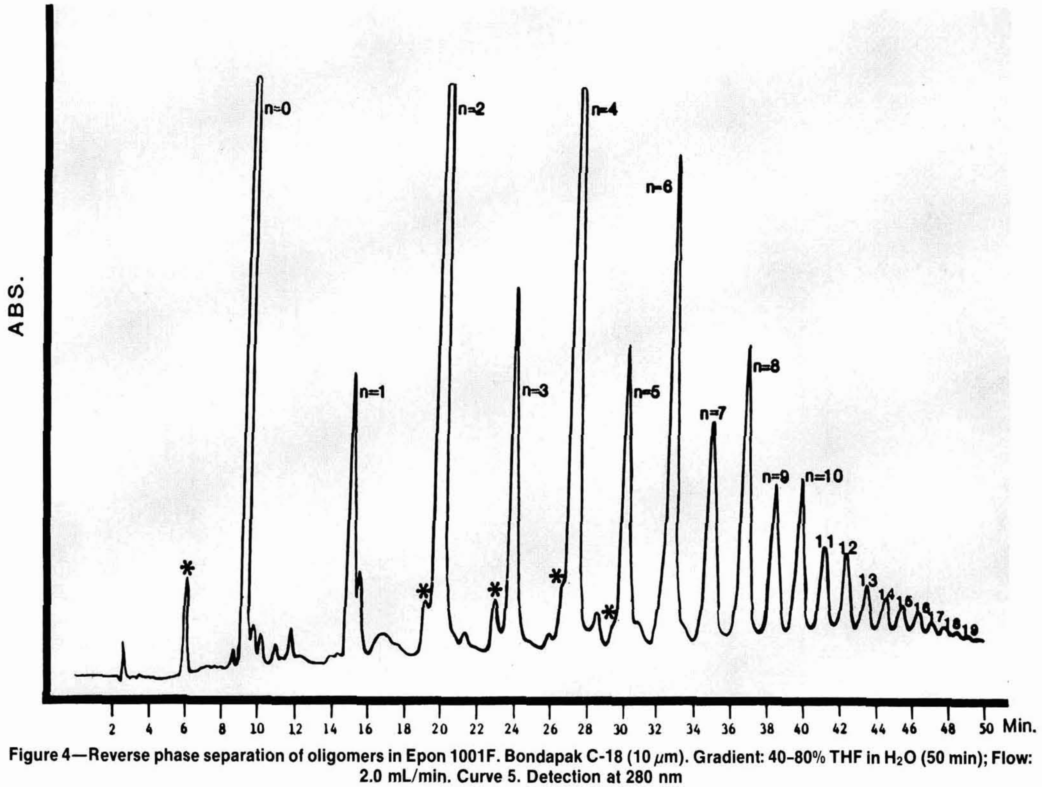

Figure 4—Reverse phase separation of oligomers in Epon 1001F. Bondapak C-18 (10 μm). Gradient: 40–80% THF in H_2O (50 min); Flow: 2.0 mL/min. Curve 5. Detection at 280 nm

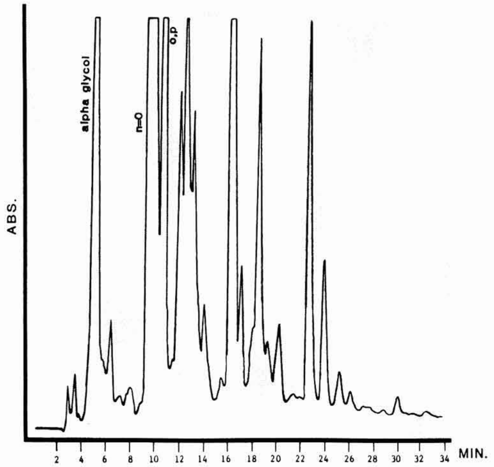

Figure 5—Reverse phase separation of liquid epoxy (low molecular weight epoxy) obtained with 5 μm diameter packing. Ultrasphere ODS. Gradient: 50-70% THF in H_2O (60 min); Flow: 1.0 mL/min. Curve 5. Detection at 280 nm

standard. Micro-ATR infrared spectroscopy was used to confirm the identities of the n = 1 and n = 2 peaks. Since the Epon 1001F is manufactured via the advancement process, the even numbered oligomers are expected to be predominant. Thus, the higher homologues are assigned indirectly to the major, later-eluting peaks.

The peak designated "o,p" in *Figures* 3 and 5 was identified spectroscopically as the diglycidyl ether of o,p bisphenol A, derived from that impurity in the bisphenol.

In *Figure* 5, the earliest eluting major peak in this chromatogram, produced at high detector sensitivity, exhibited hydroxyl as well as oxirane absorptions in its infrared spectrum. This peak is probably the n = 0 oligomer with one epoxide and one alpha-glycol group formed by hydrolysis of an oxirane group. Since the general elution pattern in reverse phase LC is from most hydrophilic to most hydrophobic, an epoxy-glycol would be expected to elute before a di-epoxide. Schenck discusses the elution order of other impurities in liquid epoxy resins and presents kinetic data on their formation during production of these resins.

The small peaks marked with asterisks in *Figure* 4 were found by infrared spectroscopy to be mono-epoxide mono-alpha-glycol functional. The decrease in resolution between these by-products and the parent oligomers with increasing molecular weight is readily apparent.

As a tool for following the production or modification of such resins, separations such as these can be quite useful. This is discussed further below.

Another type of column (RCM-100, Waters Associates), which employs a radially compressed flexible cartridge instead of rigid steel tubing, can yield separations similar to those obtainable with the Bondapak column. However, the RCM can be operated at higher flow rates than its steel tubing counterpart and hence offers reduced run times. Rapid shifts from final gradient mobile phase conditions to initial conditions are also possible because the flexible radially compressed column walls are more tolerant of rapid pressure changes, and hence these columns do not void as readily as steel columns.

A disadvantage to the use of the RCM is the pressure limitation of 2000 psi, compared with the 6000 psi limit of steel columns. Mixtures of water and THF, especially near a 1:1 ratio, are more viscous than water or THF alone, and can lead to back pressures at or near the limits recommended for the RCM at flow rates typically used to achieve short run times (4.0 mL/min). Nevertheless, a separation of low molecular weight epoxy oligomers and reaction products was possible, and was used in monitoring a reaction, as will be described below.

It has been shown[11] that the extinction coefficient of epoxy resins will change by less than 13% between the n = 0 and n = ∞ oligomers, at a wavelength of 278 nm. The extinction coefficients of asymmetrical intermediates such as the mono-epoxide-alpha glycol are found to be the mean of the symmetrical homologues. Thus, quantitation of the various di-epoxide oligomers as well as several of the minor by-products is possible.

For oligomers around n = 22 and larger, reverse phase resolution deteriorates rapidly with all columns employed

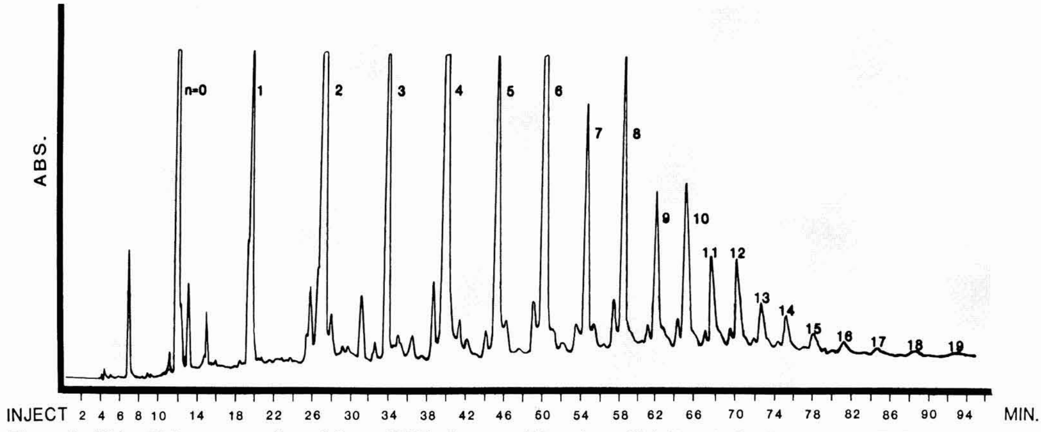

Figure 6—High efficiency separation of Epon 1001F oligomers. Ultrasphere ODS (5 μm). Gradient: 50-70% THF (60 min). Flow: 1.0 mL/min. Curve 5. Detection at 280 nm

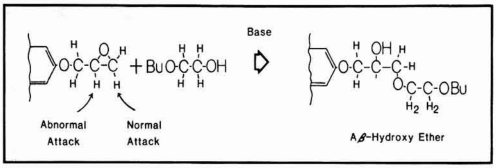

Figure 7—Reaction sites of Butyl Cellosolve in formation of β-hydroxy ethers

in this study. Although the high molecular weight species are retained by the column, they are poorly resolved regardless of gradient conditions used. Several factors are responsible for this behavior. First, the relative change in hydrophobocity between higher oligomers is much less than that between lower oligomers. Secondly, for higher oligomers there is a decreased amount of packing accessible to these molecules due to steric exclusion.[12] Thus, for improved resolution of larger molecules by reverse phase LC, larger pores in the packings would be desirable. Packings of this type have recently been introduced for separation of large biological compounds.[13]

MONITORING OF PRODUCTS OF OXIRANE RING OPENING

The reactions of the terminal oxirane groups on epoxy resins of higher molecular weight (epoxy equivalent weight, EEW ≈ 3000) are of interest in studies of the curing of these resins, changes occurring during storage, and dissolution in potentially reactive solvents such as the glycol ethers.

Figure 7 shows the sites of "normal" and "abnormal" attack[14] of the hydroxyl group of a glycol ether on an epoxy oligomer. Reduction at the "normal" site is favored due to steric influences.

Heating of higher molecular weight epoxy resins in Butyl Cellosolve® to accelerate dissolution has been found to cause increases in the EEW. Formation of β-hydroxy ethers is indicated as described below. Since the relative oxirane content of higher molecular weight resins is much less than that of lower homologues, only small changes in the resins (1% change in % N.V.M.) are observed as Butyl Cellosolve reacts.

The loss of even a fraction of the relatively few oxirane groups in higher epoxy resins can affect subsequent reactivity of the polymer with amino or carboxyl functional species during cure. Thus, detection of β-hydroxy ether formation by a means other than solids determination or infrared spectroscopy is desirable. Reverse phase LC can provide a sensitive, selective measurement of ether formation.

This study began with the preparation of the β-hydroxy ethers of a model system, the low molecular weight liquid resin Epon 828. This epoxy was reacted at 340°F with Butyl Cellosolve at 25% epoxy resin solids. The EEW of this resin changed from 765 to 1451. The reverse phase chromatogram obtained on these products is shown in *Figure* 8. The major peaks 1, 3, and 4 were thought to be reaction products since they were absent from the chromatogram of the epoxy resin itself.

Peak #1 (in *Figure* 8) was found to be a bisphenol oligomer devoid of oxirane functionality and is thought to be the di-alpha glycol of DGEBA. Peak #2 lacks any aliphatic C-O-C ether stretch absorptions in its infrared spectrum and, with a significant oxirane content, is identified as DGEBA.

Peaks #3 and #4 exhibited ether stretching absorptions in the infrared near 1100 cm^{-1} with an oxirane absorption in the spectrum of peak #3 but not peak #4. It appears that peak #3 is the monosubstituted β-hydroxy ether of the n = 0 oligomer (DGEBA) and peak #4 is the disubstituted isomer. Peaks thought to be Butyl Cellosolve adducts of n = 1 are also labeled "BC" in the Figure.

A detector wavelength of 280 nm which is near the maximum absorption of the bisphenol rings on the epoxy backbone, confers a degree of selectivity on this analysis. The glycol ether, water, and most non-aromatic impurities do not absorb at this wavelength and go undetected, while oligomeric β-hydroxy ethers are detected because they retain a bisphenol ring as a label.

The reaction of a higher molecular weight advancement epoxy (Epon 1001F) was studied next. Reaction of this resin with Butyl Cellosolve at reflux was conducted to change the EEW from 1100 to 1450, approximately a 24% loss of oxirane content. *Figure* 9 shows the reverse phase chromatograms of the resin and reaction products. Reduced retention times of the components of interest were obtained with the use of the Ultrasphere "Octyl"

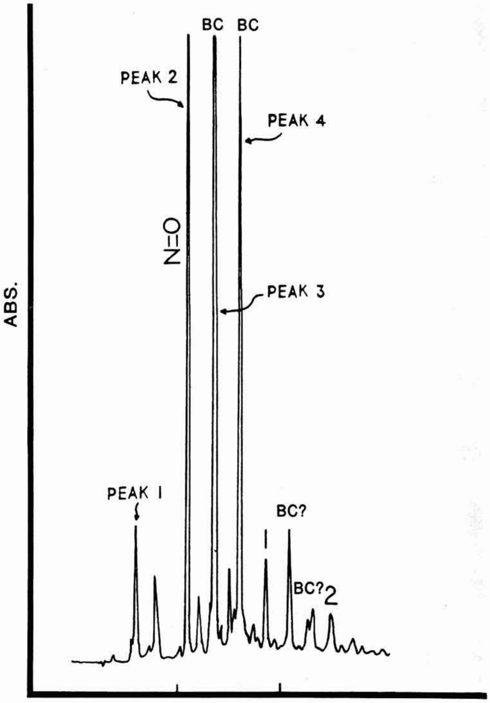

Figure 8—Reverse phase separation of adducts of Butyl Cellosolve and liquid epoxy resin. Column: 15 CM Ultrasphere "Octyl"; Gradient: 50-70% THF in H_2O (30 min); Flow: 1.0 mL/min; Detection at 280 nm

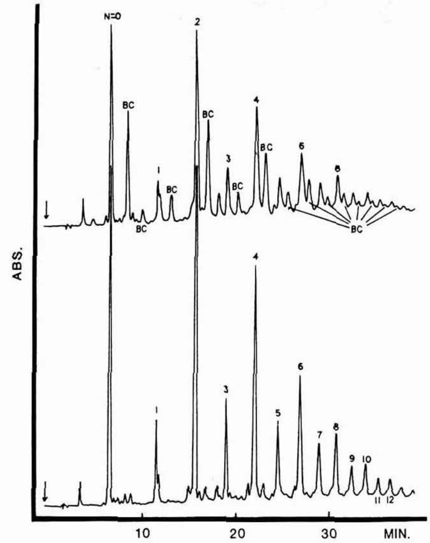

Figure 9—Reverse phase separation of adducts of Butyl Cellosolve and Epon 1001F. Column: 15 CM Ultrasphere "Octyl": Gradient: 50-70% THF in H_2O (30 min); Flow: 1.0 mL/min; Detection at 280 nm

column (Beckman Instruments), which was 15 cm in length, and contained 5 μm diameter particles with an eight carbon chain bonded phase.

The apparent product peaks formed by reaction of each oligomer with the glycol ether are labeled "BC" in the upper chromatogram in *Figure* 9. The retention times of the two peaks labeled as products which elute after the n = 0 peak matched those of the products found in the chromatogram of the liquid resin reaction mixture (*Figure* 8). Assignment of the β-hydroxy ethers of the higher homologues is based on the elution order established with the reaction of the liquid epoxy resin. The β-hydroxy ether products are expected to elute after the parent di-epoxides, with decreasing resolution from the di-epoxide as chain length increases, which is consistent with the pattern observed.

Reaction of the oxirane group with carboxylate ions is another potentially important route to loss of functionality or "curing." Studies of ring opening by carboxylic acids with the formation of β-hydroxy esters are also possible with LC.

A liquid resin was again employed as a model to study this reaction. Equimolar amounts of this epoxy and acetic acid were reacted in Butyl Cellosolve at 240° F with base catalysts at approximately 50% resin solids. Analogous to the case of ether formation discussed above, detection at 280 nm was employed. Since this reaction is considerably more rapid, samples were taken from the reactor during processing. The reverse phase liquid chromatograms obtained with gradient elution on the Waters RCM column are shown in *Figures* 10 and 11. Peaks #1 through #4 were collected from the final sample for infrared spectroscopic analysis.

Peaks #2 and #3 exhibited infrared spectra containing ester carbonyl, bisphenol rings, and secondary hydroxyl absorptions. Peak #2 lacked any oxirane absorption and had a relatively larger C-O-C absorption in its spectrum compared to peak #3 which was oxirane functional. These observations are consistent with assignment of peak #2 as the di-acetate of the n = 0 oligomer, and peak #3 as the mono-acetate.

The infrared spectrum of peak #1 differs from that of the others in that it exhibits primary hydroxyl bands in addition to ester carbonyl and the absence of oxirane. It appears to be the mono-ester of the mono-alpha glycol of the n = 0 oligomer. The spectrum of peak #4 was found to be very similar but not identical to that of peak #3. It is thought to be the mono-acetate of the n = 1 oligomer.

It is interesting to note that the di-acetate apparently elutes before the mono-acetate even though it possesses relatively more hydrocarbon character with two acetate chains compared with one. This reversal of elution order, relative to that of the ether derivatives, is probably a consequence of secondary hydroxyl groups. The di-acetate contains two of these hydrophilic groups while the mono-acetate has only one. The addition of the second hydroxyl group to the molecule apparently outweighs any increase in hydrophobic character imparted by the second acetate chain. Thus, the di-acetate, being more hydrophilic, elutes before the mono-acetate and the parent di-epoxide. This elution order is also consistent with the fact that the mono-ester, mono-alpha glycol derivative, which has both primary and secondary hydroxyl groups, elutes even before the di-acetate.

The progress of the reaction of acetic acid with the epoxide groups can easily be followed with LC. *Figure* 10 shows the separation of components five minutes into the reaction (lower chromatogram) and 30 minutes into the reaction (upper chromatogram). The formation of a significant amount of the mono-ester, peak #3, within five minutes is observed. After 30 minutes of reaction, the di-ester peak has grown substantially (peak #2).

In *Figure* 11, the significant reduction in the n = 0 di-epoxide becomes obvious at 15 minutes of reaction (upper chromatogram). The final sample, taken at 140

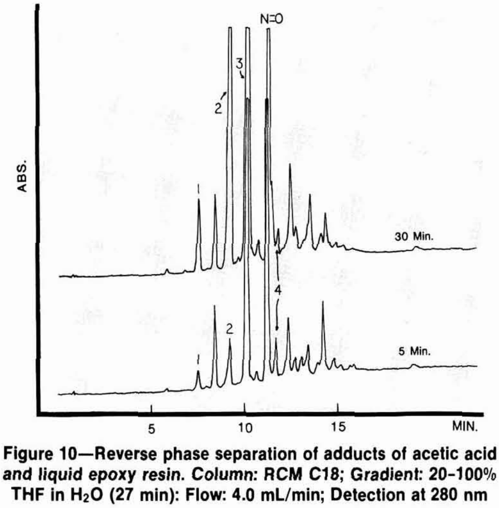

Figure 10—Reverse phase separation of adducts of acetic acid and liquid epoxy resin. Column: RCM C18; Gradient: 20-100% THF in H_2O (27 min): Flow: 4.0 mL/min; Detection at 280 nm

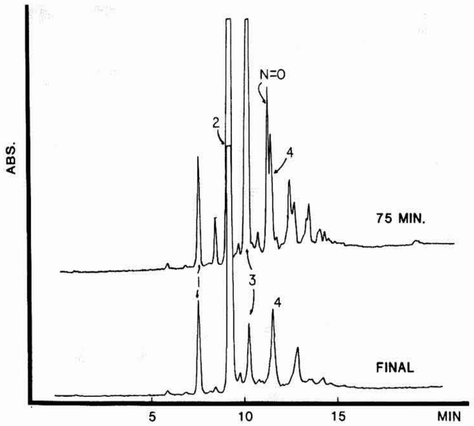

Figure 11—Subsequent analyses of reaction mixture in *Figure* 10. Column: RCM C18; Gradient: 20-100% THF in H_2O (27 min); Flow: 4.0 mL/min; Detection at 280 nm

minutes of reaction, is found to contain practically no n = 0 oligomer, and very little mono-ester (peak #3) due to its conversion to di-ester (peak #2). Titrimetric analysis of the mixture indicated at least 92% of the oxirane groups had reacted over this time period, while this series of chromatograms yields information about formation of intermediates and "by-products" such as the mono-ester mono-alpha glycol (peak #1).

SUMMARY

Modern liquid chromatographic techniques can yield rapid characterization of epoxy resins of a wide molecular weight range. Size exclusion chromatography utilizing silica based mixed pore size columns can provide excellent resolution in minimum time. Reverse phase liquid chromatography is useful in separating oligomers up to approximately n = 18 as well as the ether and ester reaction products of these oligomers. Depending on the application, 10 μm diameter reverse phase packings in steel or radially compressed columns, or 5 μm packings with various carbon loadings and column length can be employed.

Improvements in column technology can provide separations of the components of complex raw materials and intermediates used in the coatings industry. As an example, reactions of the oxirane groups of epoxy resins of various molecular weights can be monitored. Reaction of glycol ethers with oxirane groups can be detected, which allows monitoring of changes in epoxy resin solutions.

The more rapid reaction of carboxylate groups with oxiranes can also be followed with LC. Studies of the kinetics of this reaction and characterization of the products through the use of model compounds is possible. Information about the formation, curing, and degradation of novel polymers based on epoxy resins would aid research efforts in the design of vehicles for modern coatings systems requiring the performance characteristics of these resins.

ACKNOWLEDGMENTS

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The Seven Stages of an Invention

George E.F. Brewer
Coating Consultants*

In the late 1950's, as a member of a Research & Development team at Ford Motor Company, I was struggling with a project,[1] which a thought of mine had initiated, on *electrocoating*—an extension of several scientific disciplines into then uncharted waters. I didn't know about the seven stages every invention goes through, but I know now that the awareness of these successive steps would have guided me, and I want to share my experiences with those who embark on a similar journey. This report may be of particular interest to those whose responsibility it is to authorize and to supervise Research and Development.

The First Stage: The Prepared Mind

"Relevant" courses are a good preparation for an artisan, but for a creative scientist any fact may become relevant at any unexpected time.

Sherlock Holmes said once: "If I had collected stamps when I was a boy, I would be a better detective now!"

I had gone through some 40 years of my life without realizing that I was preparing for the inception of one single fruitful thought—for the moment of knowing that there is a need for a new and better way. This moment comes at an unexpected time, in an unexpected shape. It is something like meeting your future spouse.

The Second Stage: The Inception

Howard L. Gerhart[2] mentioned frequently that being eager for, listening, and picking up signals, if used wisely, is an asset for a coatings engineer.

For me, the signal which eventually led to an invention came in 1958 when my supervisor, Gilbert L. Burnside, showed me a huge pile of junked cars (*Figure* 1) and asked, "What do you see?"

I knew I had reached an important juncture, to "see" something that had never before come to my mind.

After studying the pile, I said: "The hoods, the tops and the trunk lids look almost new. The paint is good. It must be a combination of factors which under the doors and in other places causes corrosion."

Subsequent study showed that incomplete paint coats exist in poorly ventilated areas such as doors. During the paint bake operation, entrapped solvent vapor condenses and washes down uncured paint. The then unpainted areas give rise to early corrosion.

Gil Burnside then asked: "What do you want to do?"

Now I spoke too fast: "We will have to paint without solvent."

He looked at me and said: "You better sit down on your brain and figure out what kind of paint you want."

I realized that I had better solve the problem in order to wipe out the poor impression I had made. I had trapped myself into solving the problem.

First, I came up with a list of desired properties which contained items such as no hazard, no sagging, complete coverage, high corrosion resistance, and a gem—no solvent and waterlike viscosity![3]

The Third Stage: The Gestation Period

If the inception of an invention is combined with a compelling need, nay, an obsession to bring it to fruition, then it has a good chance to result in a working model. I knew that every invention is based on a new point of view. For instance, Fred Stieg[4] states that he looked at "paint" in a novel way, namely as a bed of pigment into which resin has been infused. This new approach resulted in improved coatings, due to the understanding of the Critical Pigment Volume Concentration.

Presented before the Paint Research Association, Teddington, Middlesex, England, March 12, 1984, and as the Reg. Gower Memorial Lecture of the Institute of Metal Finishing in Birmingham, England, March 15, 1984.

*6135 Wing Lake Rd., Birmingham, MI 48010.

I spent several months trying to find a new outlook, questioning every phase of the conventional painting processes. Then I began to question the validity of the requirements for an ideal paint which I had demanded. I decided I could not dispense with waterlike viscosity since I needed fast dipping and draining, and I could not tolerate solvent or water vapor in cavities. Yet, there had to be a solvent, and water at that, to meet the other requirements.

I was ready to give up.

The Fourth Stage: The Breakthrough

I distinguish between a breakthrough in the theory, which brings a different and and new concept of the goal, and a technical breakthrough which leads to the embodiment of the invention.

The theoretical breakthrough came when I realized that it was not really the solvent which had to be avoided, it was the bake in presence of solvent which damaged the paint film. I did not see how to make use of this finding, yet in the end it proved to be the theoretical breakthrough.

The technical breakthrough came when I read a paper on the electrophoretic deposition of inorganic salts on the filaments which are used in an electric bulb. All of a sudden I remembered two classmates in graduate school: Their experiments caused explosions every few days. They tried to make rubber boots and gloves from natural latex by use of a curious process called "electrophoretic deposition." They wanted a 100 mil thick film and were only able to form 5 mil films since the deposit showed high electrical resistance. The high voltage needed to overcome the resistance was quite hazardous.

Now, I immediately realized that they were doing exactly what I wanted to do: They were forming a polymeric film from an aqueous dispersion at the approximate viscosity of water! Their problem was the film thickness. All I needed was one mil, which was no problem for my friends. The solvent stayed in the bath, so I could bake in absence of solvent! Everything fell into place. I understand now why Aristotle cried out "*Eureka!*"

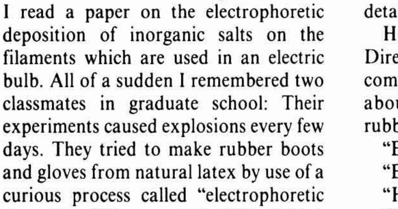

Figure 1—"What do you see?"

The Fifth Stage: "The man with a new idea is a crank until the idea succeeds" (Mark Twain)

I was not aware of this quote, and with unbelievable luck, the new idea did succeed. Actually, the main reason for writing this treatise is to warn future inventors to be very careful to disclose their ideas only to a patent attorney if they have access to one, and not to anybody else until they are reasonably sure to get support. I did not know that. I just barged into my supervisor's office and said: "Gil, now I know how to paint cars."

Gil Burnside said: "How will you do it?"

"By electrophoretic deposition."

He reached for the chemical dictionary while I went on:

"A method by which the rubber industry tried to make boots and gloves in the 1920's," and continued with a lot of other details.

He dialed a classmate of his, now the Director of Research of a large rubber company and asked: "What do you know about electrophoretic deposition of rubber?"

"Electro-what?"

"Electrophoresis"

"How do you spell it?"

"Never mind, you answered my question."

Then Gil thought for a while and said that I should go to the city library and read about electrophoresis, without letting anybody know what we were investigating.

Looking back, I should have expected a noncommittal "let's think about it" as an answer, and that would probably have ended the project, except for the alertness of Gil Burnside.

The literature revealed a first observation of the phenomenon in 1808 plus five milestones in the field of analytical chemistry, culminating with Svedberg's Nobel Prize in 1937, and four patents or groups of patents, ending in 1936 with Clayton's work for Crosse & Blackwell on the coating of food cans with bees wax emulsions.[5]

The next step in the fifth stage is the *demonstration of feasibility*. Actually, the inventor should demonstrate the feasibility before disclosing it to anybody. Yet, this particular invention had already found two believers: Gil Burnside and me. So, I was told to experiment with car batteries and waterborne house paints. We got some deposits that could be called coatings, and also uniformly thick deposits on all surfaces of a formed piece.

The most important step in the life of an invention is the *funding* for development of an embodiment or embodiments. Now, several or many persons have to agree on the viability of the idea. Some corporations have a Review Board to select promising ideas. The Board looks for compliance with certain rules. For instance, the invention should be the extension of a familiar principle, the raw material should be plentiful, the operation should require little maintenance and few repairs, a short training period without requiring special skills, etc.

The Director of Research of a large corporation said that only once has he seen an invention which complied with all the requirements of his Review Board: *The Pet Rock*—a handy sized, fairly round rock, sold in velvet lined boxes.

Be it a committee or a random number of members of an organization, an unlimited number of points can be held against any invention. It is amazing that every year hundreds of thousands of ideas are followed far enough to be granted patents. In this fifth stage *everybody is the judge*—and usually without a court of appeals. Through long and hard experience, I learned to classify the judges into five groups:

- The yes-type: their "yes" may or may not mean active support
- The experienced person, who proposed something similar years ago and is usually helpful
- The strong silent type, who may be supportive
- The pessimist: "It can't possibly work"
- The completely un-understanding person. Typical of these is the newspaper editor who, when hearing of the successful flights of the Wright Brothers, put into his Society column that they will be home for Christmas.

I know only one way to keep a project going and funded and this is through *presentation in the simplest terms,* using words common to all kinds of professionals.

I was overjoyed when finally a genius of a Patent Attorney found a way to work a basic patent for electrocoating, claiming the use of "synthetic, film-forming, electrodepositable macroions," but I was saddened by the thought of obtaining funds for these ions from Business Administrators, Finance Experts, Market Analysts, and hosts of other specialists. We did the best we could through talking about the similarities of electrocoating and metal plating. In fact, we tried to place the load of understanding into their lap through saying: "Well, you know what nickel plating does. We learned to plate-out paint."

The most determined resistance against a new idea comes from those who are working on developmental projects in related fields. Their objections are highly emotional, for instance: "Just now, when I am almost ready with liquid coal as a fuel, he wants solid hydrogen! *Der Dummkopf*!" Logic and arguments of this type force us into the next phase in the development of an invention.

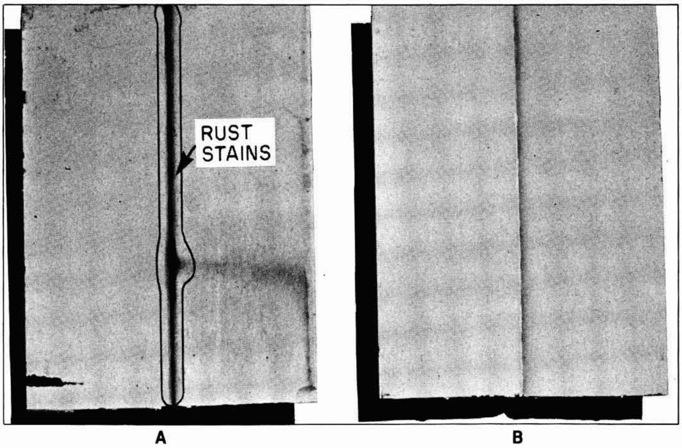

Figure 2—Coach joints (butted joints. A—spray painted; B—electro painted)

The Sixth Stage: Demonstration of Advantages

Samples made by use of the invention are needed. This includes all kinds of samples, not only those samples for which the invention was originally targeted. Although "... the largest market for a successful idea is unexpected ...,"[6] this fact had not been known to us. But we put our electrocoating paint on almost anything we could get our hands on: toys, particularly toy cars; nuts and bolts; hardware; expanded metal and wire mesh; wheels; doors; flanges; joints; even entire automotive bodies. All these pieces went through solidity tests like salt spray, gravelometer, Florida exposure, etc.

These samples definitely aroused interest, and several years later we found that one sample had secured the interest of the most important person: Mr. John Dykstra, who retired as President of Ford Motor Co. His secretary returned to us samples of coach-joints (*Figure* 2) which he had kept in his desk and used as examples for the only painting process capable of protecting such structures.

Part of the sixth stage of an invention is cost estimation. Again, we were lucky. The figures showed that the direct cost for the new painting process is approximately one half of conventional coating methods —a ball-park figure, still valid after 20 years! Also, our data showed that electrocoating uses extremely small quantities of Volatile Organic Compounds and is, therefore, environmentally desirable. I describe such random efforts as important since the inventor is groping during the sixth stage of an invention to find advantages and unexpected applications.

It is true that the successful demonstration of the advantages completes or even crowns the inventor's work, but it is a frequently-made mistake to think that the monetary fruits of the investment are now supposed to come in. Nothing can be farther from the truth. Actually we are facing the last, and sometimes most expensive phase of an invention.

The Seventh Stage: Innovation

Innovation has been defined as "... the translation of scientific findings into products and sevices ...."[7] I think this was included in Edison's words when he defined an invention as "... one percent inspiration and ninety-nine percent perspiration. ..."

In our case, we were sure that the first few weeks of operation of an electropainting installation would be a great success. In fact, we had already painted several bodies on a conveyor and they had shown superiority to dipped or zinc rich primed bodies on the test track. Yet, we were not sure that we could keep an electrocoating paint in top shape for at least one year of operation. We had computed that, during one year, 20 times the "fill" of the tank has to be added as "feed." Thus, if we had to discard the tank fill after one year, the loss would amount to only 5% of the total cost of paint. We realized, of course, that we can not experiment with one year production of bodies. The warranty cost could be astronomical.

We looked for a comparatively cheap part, easily replaceable in case of complaints. Thus, we decided to paint wheels with body primers. In fact, we painted 10,000 wheels per shift for a total of 5.6 million wheels. Over 75,000 panels were prepared and tested in our laboratories and subjected to Florida exposure, etc. We also prime coated several hundred automotive bodies. Through this work we learned how to run not only one, but several automotive paints in production tanks.

It can easily be seen that the seventh stage of an invention—the translation of an idea into goods or services—can demand a heavier investment than all earlier stages together.

Summary

The road from ideas to goods or services is long, hard, and expensive, but the rewards—satisfaction and tangible benefits—are sufficient to secure a nondiminishing stream of inventions.

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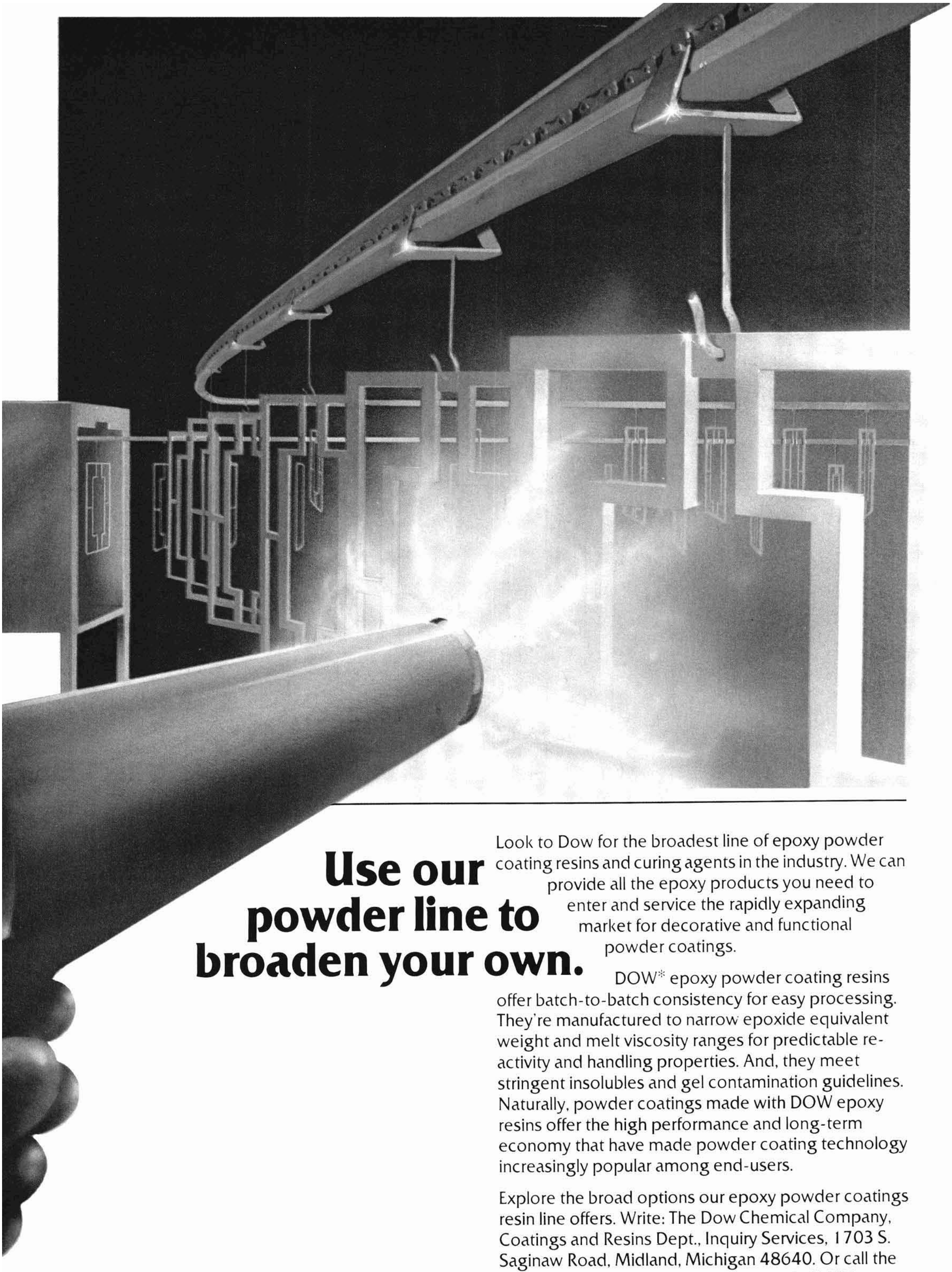


BIRMINGHAM .... NOV.

"Powder Coatings"

In recognition of their contributions to the Club, Distinguished Service Certificates were awarded to John Green, Roy Arnold, and David Heath.

Mr. Green, a member of the Club since 1955, has served on the Program and Publicity Committees. President from 1981-82, he now holds the position of Social Officer.

Mr. Arnold joined the Club in 1962 and served as Treasurer from 1971-73. Elected President in 1975, Mr. Arnold also served on the Federation Executive Committee.

A member since 1970, Mr. Heath was Treasurer from 1974-77. He served as President in 1978-79 and was Social Officer in 1981-84. Mr. Heath now holds the office of Hon. Secretary.

A presentation on "REACTIVITY AND RHEOLOGY OF POWDER COATINGS" was given by A. van der Werff, of DSC Resins, Holland.

Mr. van der Werff based his discussion upon an intricate study of the catalysis of powder coatings. The presence of catalysts was said to be essential in decreasing curing time and increasing performance.

According to Mr. van der Werff, the three catalysts considered in epoxy TGIC and polyester systems were 2-phenylimidazoline; tetramethylammonium chloride; and benzyl-N,N-dimethylamine. For the catalyst to be efficient, it must first be soluble in the molten resin, he said. Catalysts are not consumed by the curing process, and therefore may remain in the cured film unless they are volatile.

Mr. van der Werff described the test scheme used to assess the catalyst performance which included mechanical testing, flow, gloss, QUV, boiling water resistance, and differential thermal analysis of fresh and aged powders. He said that all three catalysts proved excellent for mechanical properties, gloss, and accelerated weathering. There was good correlation between flow and gel times, which showed 2-phenylimidazoline to have the best characteristics; benzyl-N,N-dimethylamine slightly worse; and tetramethylammonium chloride to be very poor for flow and gel time.

Salt spray resistance showed tetramethylammonium chloride, at a high concentration, to impart severe film creepage from scribed edges in both epoxy and polyester systems, he continued. Differential scanning calorimetry results indicated this technique to be of particular use in determining the percentage of pre-reaction and gel times. At both higher and lower temperatures, the percentage reaction was found to be higher in catalyzed systems than in non-catalyzed.

In conclusion, Mr. van der Werff said that there was good correlation between gel times and flow. Epoxy TGIC systems had good water resistance. In addition, tetramethylammonium chloride reduced water resistance and salt spray resistance, even at lower temperatures. He maintained that conversion at gel is lower with uncatalyzed systems and conversion at gel is increased by higher temperatures. Finally, increases in gel time with aging are not related to the amount of pre-reaction, he said.

DAVID M. HEATH, *Secretary*

CHICAGO .... JAN.

"Aluminum Pigments" and *"Superfund Legislation"*

Russell Furguson, of Silberline Manufacturing Co., spoke on "ALUMINUM PIGMENTS: PAST, PRESENT, AND FUTURE."

In addition, "SUPERFUND LEGISLATION" was discussed by Margaret McCue, of U.S. Environmental Protection Agency.

Wally Krason, of the Enterprise Cos., announced the establishment of an ACS-sponsored Roy Tess Award honoring significant scientific achievement in polymers and coatings. The award will be presented at the 192nd ACS meeting in September 1986.

R.L. KLEINLEIN, *Secretary*

KANSAS CITY .... JAN.

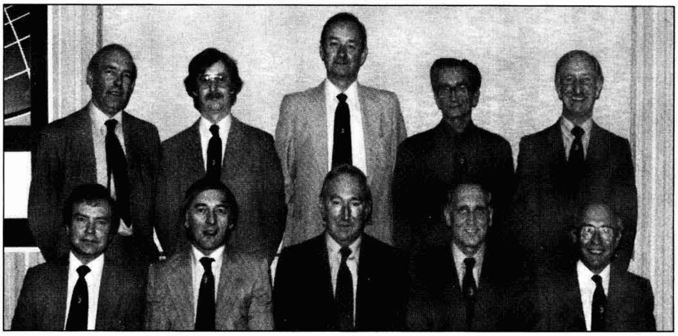
"Industrial Lacquers"

Society President Steve Bussjaeger reported that the Federation "Spring Week" will be held in Baltimore from May 14-17.

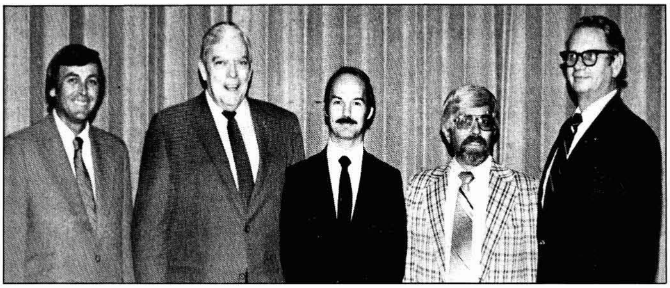
Mr. Bussjaeger told members that a Hazardous Materials Identification System (HMIS) seminar was held recently by NPCA. It covered new regulations for Material Safety Data Sheets and Material Identification. He urged members to become familiar with HMIS.

Philip Reitano, of Dynamit Nobel of America, gave a talk on "INDUSTRIAL LACQUERS BASED ON HM-LINEAR SATURATED POLYESTERS."

JERRY P. HEFLING, *Secretary*



EXECUTIVE COMMITTEE MEMBERS of the Birmingham Club for 1984-85. (Seated, left to right): Secretary—David Heath, of Holden Surface Coatings Ltd.; Past-President—Harry J. Griffiths, of Postans Ltd.; President—Roland L. Staples, of Midland Specialty Powders; President-Elect—J.R. Jukes, of Croda Paints Ltd; and Treasurer—Stan Brettell, of Llewellyn Ryland Ltd. (Standing, left to right): Graham Fowkes; Gerry Gough, John Green, John Hitchin, and Ray Tennant



1984-85 HOUSTON SOCIETY OFFICERS are (left to right): President—Richard Batchelor, of Valspar Corp.; Vice-President—Arthur McDermott, of Nalco Chemical Co.; Secretary—David Satzger, of The O'Brien Corp.; Treasurer—James Judlin, of Devoe & Reynolds Co.; and Society Representative—Willy C.P. Busch, of PPG Industries, Inc.

HOUSTON .... DEC.

"Diatomite in Coatings"

Tom Remmers, of Manville Products, discussed "THE USE OF DIATOMITE IN COATINGS."

Diatomites are the skeletal remains of aquatic plants, Mr. Remmers explained. With their unique hollow structures and shapes, these plants can impart a variety of properties. In coatings, diatomite serves principally as a flattening agent.

He continued by describing how the irregularity of the particle shapes contribute to an improved intercoat adhesion, better sandibility, and faster solvent release. The irregularity provides for a more uniform gloss and sheen and when compared with talcs, shows a greater flattening efficiency, he concluded.

DAVID SATZGER, *Secretary*

LOS ANGELES .... JAN.

"Exterior Trade Sales Paints"

Society President Earl Smith asked Trev Whittington, Textbook Committee Chairman, to give a report on the Committee's project. The contents of the book will be governed by employers, he explained. Mr. Whittington also stated that three years would be needed to complete the textbook.

Lloyd Haanstra gave an environmental report. Mr. Haanstra asked the membership for help in notifying everyone interested in Air Resources business. He also explained that Rule 1113, which calls for 380 grams per liter for non-flat coatings, will run out in September 1985. Mr. Haanstra added that the rule will probably be extended until September 1986, but it will take a fight to try and have it extended beyond that time. A bulletin will be released which requests industry people to attend a workshop to come up with new ideas to fight for the extension.

Martin Freedman, of Rohm and Haas Co., spoke on "EXTERIOR TRADE SALES PAINTS—PROBLEMS AND CORRECTIONS."

Mr. Freedman addressed the problems of adhesion, cracking and flaking, yellowing, flexibility, dirt accumulation, chalking, staining, fading, tint retention, gloss retention, frosting, and efflorescence/hydrolysis.

Q. What are the effects of excess coalescent on weatherability?

A. The only effect that we have seen is the increase of dirt pickup. This is due to the coalescents staying in the film.

Q. What is the chemical principal that causes the different titanium dioxides to yield different initial glosses?

A. Primarily, the gloss differences are due to the oil absorbtion of the different grades and the surface treatment that the different grades possess.

Q. Is accelerated exposure testing on DSET EMMAQUA a good predictor for paint durability?

A. It is our opinion that the best test for exterior durability is natural weathering. We have found it very hard to correlate artificial weathering such as the QUV to natural weathering. Where we use the QUV is to cross-check various pigments or extenders in the same vehicle or resin. I am really not too familiar with the results of DSET.

RAY DIMAIO, *Secretary*

LOUISVILLE .... JAN.

"Corrosion-Resistant Zinc Phosphates"

Jim Doyle, Educational Committee Chairman, reported on the current Society-sponsored course at the University of Louisville, "Instrumental Analysis" which is being studied by 13 students. The fall course will be a laboratory hands-on "Formulation" study.

Bob Zabel, of Heubach, Inc., discussed the "NEW GENERATION OF CORROSION-RESISTANT ZINC PHOSPHATES."

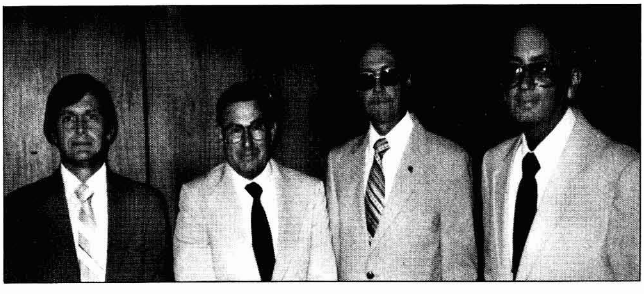
Mr. Zabel presented information resulting from a study conducted in Germany which compared the corrosion inhibiting performances of various zinc phosphates. He pointed out that the new generation of zinc phosphates come very close to performing like zinc chromates. Mr. Zabel attributed the chemical treatment of the pigment and reduced particle size as reasons for the improved performance. Mr. Zabel then described three specific new generation pigments and the protection mechanisms of each.

KEN HYDE, *Secretary*

MONTREAL .... DEC.

"Rheology Modifiers"

Six individuals addressed the subject of rheological modifiers at the Society's half-day symposium/meeting.



C-D-I-C OFFICERS for 1984-85. (Left to right): Treasurer—Richard M. Castle, of Paint America Co.; President—David C. Kinder, of Asarco, Inc.; Vice-President—Bill M. Hollifield, of Perry & Derrick; and Secretary—Joseph W. Stout, of Hanna Chemical Coatings

1984-85 OFFICERS OF THE DALLAS SOCIETY. (Left to right): Treasurer—Frederick T. Beard, of Jones-Blair Co.; President and Vice-President—Van G. Falcone, of Koppers Co.; and Secretary—Ashwin V. Parikh, of Union Carbide Corp.

Luigi Cutrone, of Tioxide Canada Inc., introduced the subject of rheological modifiers by describing their function in solvent and water-based paints. He stated that the interaction of rheology modifiers with paint ingredients is better understood in solvent systems than water-based systems.

Robert H. Dey, of NL Chemicals, described the role of organoclay and organic type additives in the coatings industry. Mr. Dey added that many of the rheological problems encountered with these additives are the results of improper selection and incorporation techniques.

Joseph W. Hagan, of Union Carbide Corp., described the role of cellulosic thickeners for latex paints. Following his presentation William H. Elfring Jr., of Rohm and Haas Co., addressed the differences and functions of latex thickeners and rheological modifiers. He stated that the associative thickeners and rheology modifiers give the formulator a choice in range of beneficial rheological and film properties.

The use of thickeners in aqueous printing inks was explained by Gerald V. Laudato, of S.C. Johnson and Son, Inc. Mr. Laudato said that thickeners employed in water-based printing inks should raise viscosity without inhibiting transfer and printability properties within the application unit.

The symposium/meeting concluded with a presentation on the use of new cellulosic polymers for rheology control of latex paints, by Kathryn G. Shaw, of Hercules Incorporated.

VIJAY SHARMA, *Secretary*

NEW ENGLAND .... JAN.

"HMIS"

In this joint meeting with the New England Coatings Association, "HMIS—HAZARDOUS MATERIALS IDENTIFICATION SYSTEM," was presented by James Elliot, Field Secretary for the National Paint & Coatings Association.

Emphasizing the importance of the HMIS system to the industry, Mr. Elliot explained why it was created, and the roles of the federal government and the coatings industry.

Mr. Elliot discussed the actions which must be taken by employees and employers in the coatings industry. Raw material producers must assess the hazards of their products. They must assign a number to each of three hazard categories: health, flammability, and reactivity. In addition, raw material safety data sheets must be prepared. Emphasizing the importance of these sheets, Mr. Elliot said that raw material distributors must check to see if proper labeling has been put on raw materials. He concluded by stating that the coatings industry should be proud of the initiative it took to protect and safeguard its employees.

KEVIN MULKERN, *Secretary*

PACIFIC NORTHWEST .. NOV.

"Hyperdispersants"

James Hampton, of I.C.I. Americas, Inc., spoke on "HYPERDISPERSANTS—A NEW TECHNOLOGY FOR THE COATINGS INDUSTRY."

Mr. Hampton described new hyperdispersants recently produced by his company. Among the advantages offered by these products is the possibility of increasing pigment volume and of attaining stable mill bases with very little or no resin content. This opens the door to producing pre-dispersions for coatings having similar solvent systems, he said.

In conclusion, Mr. Hampton claimed that the coatings manufacturer would get improved energy utilization by reducing milling time, would increase productivity by the use of pre-dispersion systems, and would attain better quality control over nagging separation and floatation problems.

Michael DePietro, *Secretary*

Constituent Society Meetings and Secretaries

BALTIMORE (Third Thursday—Eudowood Gardens, Towson, MD; Edward B. Countryman, Bruning Paint Co., 601 S. Haven St., Baltimore, MD 21214. Virginia Section—Fourth Wednesday, Ramada Inn-East, Williamsburg, VA).

BIRMINGHAM (First Thursday—meeting sites vary). D.M. Heath, Holden Surface Ctgs. Ltd., Bordesley Green Rd., Birmingham B9 4TQ England.

CHICAGO (First Monday—meetings alternate between Sharko's in Villa Park and Como's in Chicago). Ron Kleinlein, Sherwin-Williams Co., 10909 S. Cottage Grove Ave., Chicago, IL 60628.

C-D-I-C (Second Monday—Sept., Jan., Apr., June in Columbus; Oct., Dec., Mar., May in Cincinnati; Nov., Feb., in Dayton). Joseph W. Stout, Hanna Chemical Coatings, P.O. Box 147, Columbus, OH 43216.

CLEVELAND (Third Tuesday—meeting sites vary). Madelyn Harding, Sherwin-Williams Co., P.O. Box 6027, Cleveland, OH 44101.

DALLAS (Thursday following second Wednesday—Executive Inn, near Lovefield Airport). Ashwin V. Parikh, Union Carbide Corp., 2326 Lonnecker Dr., Garland, TX 75041.

DETROIT (Fourth Tuesday—meeting sites vary). Carolyn Gessner, Inmont Corp., 26701 Telegraph, P.O. Box 5009, Southfield, MI 48086.

GOLDEN GATE (Monday before third Wednesday—Alternate between Sabella's Restaurant on Fisherman's Wharf and Francesco's, Oakland, CA). Karl Sauer, Pfizer, Inc., 776 Rosemont Rd., Oakland, CA 94610.

HOUSTON (Second Wednesday—Sonny Look's, Houston, TX) David Satzger, O'Brien Corp., P.O. Box 14509, Houston, TX 77221.

KANSAS CITY (Second Thursday—Cascone's Restaurant, Kansas City, MO). Jerry P. Hefling, Loctite Auto & Consumer, 3255 Harvester Rd., Kansas City, KS 66115.

LOS ANGELES (Second Wednesday—Steven's Steak House, Commerce, CA). Ray DiMaio, Koppers Co., P.O. Box 22066, Los Angeles, CA 90022.

LOUISVILLE (Third Wednesday—Breckinridge Inn, Louisville, KY). Ken Hyde, Reliance Universal, Inc., Louisville, KY 40233.

MEXICO (Fourth Thursday—meeting sites vary). Rosa Ma. Rojas, Mobil Atlas, S.A. de C.V., Poniente 146 No. 700, 02300 Mexico, D.F., Mexico.

MONTREAL (First Wednesday—Bill Wong's Restaurant). Vijay Sharma, Swing Paints Ltd., 2100 St. Patrick St., Montreal, Que., Canada.

NEW ENGLAND (Third Thursday—Hillcrest Function Facilities, Waltham). Kevin Mulkern, Samuel Cabot, 229 Marginal St., Chelsea, MA 02150.

NEW YORK (Second Tuesday—Landmark II, East Rutherford, NJ). Kenneth DePaul, Whittaker, Clark & Daniels, 1000 Coolidge St., S. Plainfield, NJ 07080.

NORTHWESTERN (Tuesday after first Monday—Jax Cafe, Minneapolis, MN). Larry Brandenburger, Valspar Corp., 1101 Third St. S., Minneapolis, MN 55415.

PACIFIC NORTHWEST (Portland Section—Tuesday following second Wednesday; Seattle Section—the day after Portland; British Columbia Section—the day after Seattle). Dennis Hatfield, J.F. Shelton, 1067 Industry Dr., Tukwila, WA 98188.

PHILADELPHIA (Second Thursday—Dugan's Restaurant). Donald F. Denny, E.W. Kaufmann Co., P.O. Box 529, Southampton, PA 18966.

PIEDMONT (Third Wednesday—Howard Johnson's, Brentwood exit of I-85, High Point, NC.) Steve Lasine, McCullough & Benton, P.O. Box 29803, Atlanta, GA 30359.

PITTSBURGH (First Monday—A.J. Isacco, Puritan Paint & Oil Co., 1 N. Main St., Pittsburgh, PA 15215.

ROCKY MOUNTAIN (Monday following first Wednesday—Bernard's, Arvada, CO). Craig Hansen, George C. Brandt, Inc., 6500 Stapleton Dr. S., Denver, CO 80216.

ST. LOUIS (Third Tuesday—Salad Bowl Restaurant). Merle D. Held, Cyprus Industrial Minerals Co., 225 S. Meramec St., St. Louis, MO 63105.

SOUTHERN (Gulf Coast Section—Third Thursday; Central Florida Section—Third Thursday after first Monday; Atlanta Section—Third Thursday; Memphis Section—bi-monthly, Second Tuesday; Miami Section—Tuesday prior to Central Florida Section). Ronald R. Brown, Union Chemicals Div., P.O. Box 26845, Charlotte, NC 28213.

TORONTO (Second Monday—Cambridge Motor Hotel). Philip Read, Sico, Inc., 30 Bethridge Rd., Rexdale, Ont., Canada M9W 1N2.

WESTERN NEW YORK (Third Tuesday—The Red Mill, Clarence, NY). Michael De Pietro, Spencer-Kellogg Div., Textron, Inc., P.O. Box 210, Buffalo, NY 14225.

PITTSBURGH .... JAN.

"Selection of Solvents"

President Clifford Schoff announced that the Society's paint technology program will be presented at St. Vincent's College, in Latrobe, PA. The presentation will be given by Morris Berger, of Gateway Paint & Chemical Co., and Anthony J. Isacco, of Puritan Paint & Oil Co.

Garland Sprinkle, Jr., of Eastman Chemical Products, Inc., spoke on "The Selection of Solvents for High Solids Coatings Systems."

Mr. Sprinkle noted that the old Rule 66 laws have been replaced in recent years by VOC regulations which limit the amount of solvent in a coating. Because these limits alter the form of the coating, the selection of new solvent is very important, Mr. Sprinkle stated.

According to Mr. Sprinkle, solvents affect the coating system in two ways—viscosity and surface tension. The viscosity will be determined by the solvent activity and solvent density. He described surface tension as the work required to form a unit area of surface between the liquid and vapor phase. Surface tension could be affected by inadequate spraying, cratering, and picture framing, as well as by contamination by petroleum oil and silicone fluid.

Increasing the solids of coatings increases the surface tension, he continued. The higher the molecular weight of the solvent, the higher is its surface tension value. He described the surface tension of solvents as fast evaporating, medium evaporating, slow evaporating, and very slow evaporating. In conclusion, Mr. Sprinkle added that the surface tension of the substrate affects the coating if the coating has a higher surface tension. The viscosity and evaporation of the solvent also affects the substrate and the performance of the coating on the substrate.

A.J. Isacco, *Secretary*

Future Society Meetings

Birmingham

(May 2)—"TWO COMPONENTS SPRAYING TECHNOLOGY"—P. Green, Binks Bullows Ltd.

(June 12)—SYMPOSIUM—"MIRACLE 85—MANAGEMENT AND INVESTMENT REQUIRED TO ACHIEVE COATINGS LABORATORY EFFICIENCY."

Golden Gate

(May 20)—"EXTERIOR LATEX PAINT STUDY"—Dan Dixon, Freeport Kaolin Company.

Kansas City

(May 9)—FEDERATION NIGHT.

Los Angeles

(May 15)—"EXTERIOR LATEX PAINT STUDY"—Dan Dixon, Freeport Kaolin Co.

(June 12)—ANNUAL MEETING, ELECTION OF OFFICERS.

New York

(May 14)—PAVAC AWARD NIGHT.

Pacific Northwest

(May 21-23)—"EXTERIOR LATEX PAINT STUDY"—Dan Dixon, Freeport Kaolin Company.

Piedmont

(May 15)—"INFLUENCE OF INACTIVE AND ACTIVE PIGMENTS ON THE CORROSION INHIBITING PROPERTIES OF PAINT FILMS"—Dr. Rolf H. Odenthal, Mobay Chemical Corp.

(June 19)—NOMINATION OF OFFICERS.

Rocky Mountain

(May 13)—"EXTERIOR LATEX PAINT STUDY"—Dan Dixon, Freeport Kaolin Company.

St. Louis

(May 21)—MANUFACTURING NIGHT.

Western New York

(May 21)—"ORGANIC PIGMENTS: PAST, PRESENT, AND FUTURE"—Dr. Hugh Smith, Sun Chemical Corp.

Elections

CHICAGO

Active

Angerer, Mark—Iowa Paint Mfg. Co., Inc., Des Moines, IA.
Augius, George J.—DeSoto, Inc., Des Plaines, IL.
Bodden, Phaedra A.—DeSoto, Inc., Des Plaines.
Bonin, Demetrius—DeSoto, Inc., Des Plaines.
Butler, James M.—The Enterprise Companies, Chicago, IL.
Cabanski, John L.—Felt Products Mfg. Co., Skokie, IL.
Chernich, James—Whittaker Corp., Batavia, IL.
Comfort, Raymond B.—Whirlpool Corp., Benton Harbor, MI.
Conti, Ronald S.—DeSoto, Inc., Des Plaines.
Dhake, Digamar G.—SCM Pigments Corp., Chicago.
Durkin, Larry—W.C. Richards Co., Blue Island, IL.
Fotis, William W.—The Enterprise Companies, Wheeling, IL.
Horgan, Kathleen M.—DeSoto, Inc., Des Plaines.
McComas, Gerald W.—DeSoto, Inc., Des Plaines.
Rector, Louis P.—DeSoto, Inc., Des Plaines.
Renkor, Edward J.—Chromatec Corp., Elk Grove Village, IL.
Superczynski, Wayne—The O'Brien Corp., South Bend, IN.
Wieder, Alicia G.—SCM Pigments Corp., Chicago.
Ziehm, Jacalyn J.—Sherwin-Williams Co., Chicago, IL.

Associate

Andres, Jim—Sacco Pigments, Chicago, IL.
Burkhard, D. Richard—Henley & Co., Inc., Elk Grove Village, IL.
Diehl, Jack W.—McWhorter, Inc., Carpentersville, IL.
Greenwald, Richard F.—S & G Resins, Chicago.
Hershfield, Neil F.—Dow Chemical Co., Rolling Meadows, IL.
Kallal, William L. Jr.—Sandoz Chemicals, Inc., Lisle, IL.
Lerch, Mariann—Kraft Chemical Co., Inc., Melrose Park, IL.

CLEVELAND

Active

Ball, Timothy A.—Sherwin-Williams Co., Cleveland, OH.
Berghoff, Wellington F.—SCM Pigments Corp., Strongsville, OH.
Brown, Roger D.—SCM Pigments Corp., Strongsville.
Cook, Steve—PPG Industries, Inc., Strongsville.
Dreka, George B.—Byk-Chemie USA, Cleveland.
Ganapathy, Ashok A.—Poly-Carb, Inc., Solon, OH.
Haagenson, Kenneth A.—Harrison Paint Corp., Canton, OH.
Haunty, William D.—Sherwin-Williams Co., Cleveland.
Hazen, James R.—SCM Pigments Corp., Strongsville.
Hoffman, Paul J.—Akron Paint & Varnish Co., Akron, OH.
Irani, Hormuz P.—Poly-Carb, Inc., Solon.
Jano, William C. Jr.—SCM Pigments Corp., Westlake, OH.
Levitan, Stephen R.—Master Builders, Cleveland.
Mallarnee, W. Roy—Plasti-Kote Co., Inc., Medina, OH.
Matko, Steven P.—Cook Paint & Varnish Co., Cleveland.
Mitchell, Carol B.—Sherwin-Williams Co., Cleveland.
Patt, Marlene R.—Sherwin-Williams Co., Cleveland.
Rebar, John T.—SCM Pigments Corp., Strongsville.
Rehberg, Irene L.—Elastic Materials Inc., Medina.
Robinson, Jolaine L.—Avery Int'l.-Refl. Div., Painesville, OH.
Sabo, Edward—Star Bronze Co., Alliance, OH.
Sathe, Sudarshan R.—Poly-Carb, Inc., Solon.
Singleton, Chloe—Sherwin-Williams Co., Cleveland.
Tuckerman, Thomas D.—Cambridge Coatings, Cleveland.
Wiita, Ron—Pluess Staufer/Omya, Wadsworth, OH.
Wise, Kuyler M.—Sherwin-Williams Co., Cleveland.

Associate

Chunat, Ronald F.—R.T. Vanderbilt Co., Bedford, OH.
Grubb, Frank—Midwest Technology, Strongsville, OH.
Huth, Douglas—Ferro Corp., Cleveland, OH.
Kish, John—Schabel Product Co., Cleveland.
Purpura, Matthew, T.—Consultant, Willowick, OH.
St. Jean, Steve—Palmar Supplies Co., Cleveland.
True, S. Garth—SCD Div. of Etna Prod., Chagrin Falls, OH.
Vidika, Mark—Palmer Supplies Co., Cleveland.

Retired

Culler, John—Rocky River, OH.
Linn, James—Parma Hts., OH.

DALLAS

Active

Garcia, Natalia C.—Kerr-McGee Chemical Corp., Oklahoma City, OK.
Plyler, Anne O.—Kerr-McGee Chemical Corp., Oklahoma City.

HOUSTON

Active

DeCelles, Patrick G.—Monarch Paint Co., Houston, TX.
Storm, Richard W.—Product Coatings, Inc., Houston.

Associate

McMullin, Bob—Byk Chemie USA, Allen, TX.
Robinson, Sara M.—Hitox Corp. of America, Corpus Christi, TX.
Terry, L. Sam—Thompson-Hayward Chemical Co., Houston, TX.

MONTREAL

Active

Jacques, Lussier C.—Bapco, Montreal, Que.

Associate

Jean, Linda—Reichhold Ltd., Ste. Therese, Que.

NEW ENGLAND

Associate

Johnson, Jimmie R.—Ciba-Geigy Corp., Lexington, MA.

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NEW YORK

Associate

SILVER, BEN—Nortman Associates, Inc., Brooklyn, NY.

NORTHWESTERN

Active

COLLINS, STUART—Sierra Corp. Inc., Minnetonka, MN.
MEHTA, UMESH—Cargill, Inc., Minneapolis.
SHAH, SUNIL—Diamond Vogel Paints, Minneapolis.

Associate

COLSTON, FORREST—Ashland Chemical Co., Shakopee, MN.
SEVENICH, JOHN—Ashland Chemical Co., Shakopee.

PACIFIC NORTHWEST

Active

AZURIN, REMEDIOS S.—Preservative Paint Co., Seattle, WA.
BOWMAN, KEN—Nero Acrylic Paint & Plasters Ltd., Lethbridge, Alberta.
FRYE, WILLIAM—Reliance Universal, Inc., Salem, OR.
MILLS, JOHN E.—Reliance Universal, Inc., Salem.
O'LEARY, KEVIN R.—McCloskey Varnish Co., Portland, OR.
PARK, DAVID W.—Gaco Western, Inc., Seattle.
PUGMIRE, REX L.—Reliance Universal, Inc., Salem.
RICHTER, RICHARD M.—Reliance Universal, Inc., Salem.
VENUS, TERRY L.—McCloskey Varnish Co., Portland.
WAMPLER, ROBERT J.—Willamette Industries, Beauerton, OR.

Associate

DUFFY, D.E.—Canada Colors and Chemicals Ltd., Richmond, B.C.
HENDERSON, PAUL T.—Celanese Chemical Co., Torrance, CA.
LARSON, MELVIN E.—McKesson Chemical Co., Auburn, WA.

PITTSBURGH

Active

HARLEY, MARK A.—PPG Industries, Inc., Allison Park, PA.
KAPP, DEBORAH B.—PPG Industries, Inc., Allison Park.
KRAUSHAAR, DOUGLAS J.—Drakenfeld Colors, Washington, PA.
VERGIS, EMANUEL N.—PPG Industries, Inc., Springdale, PA.

Associate

SHIELDS, WILLIAM T. III—Neville Chemical Co., Pittsburgh, PA.

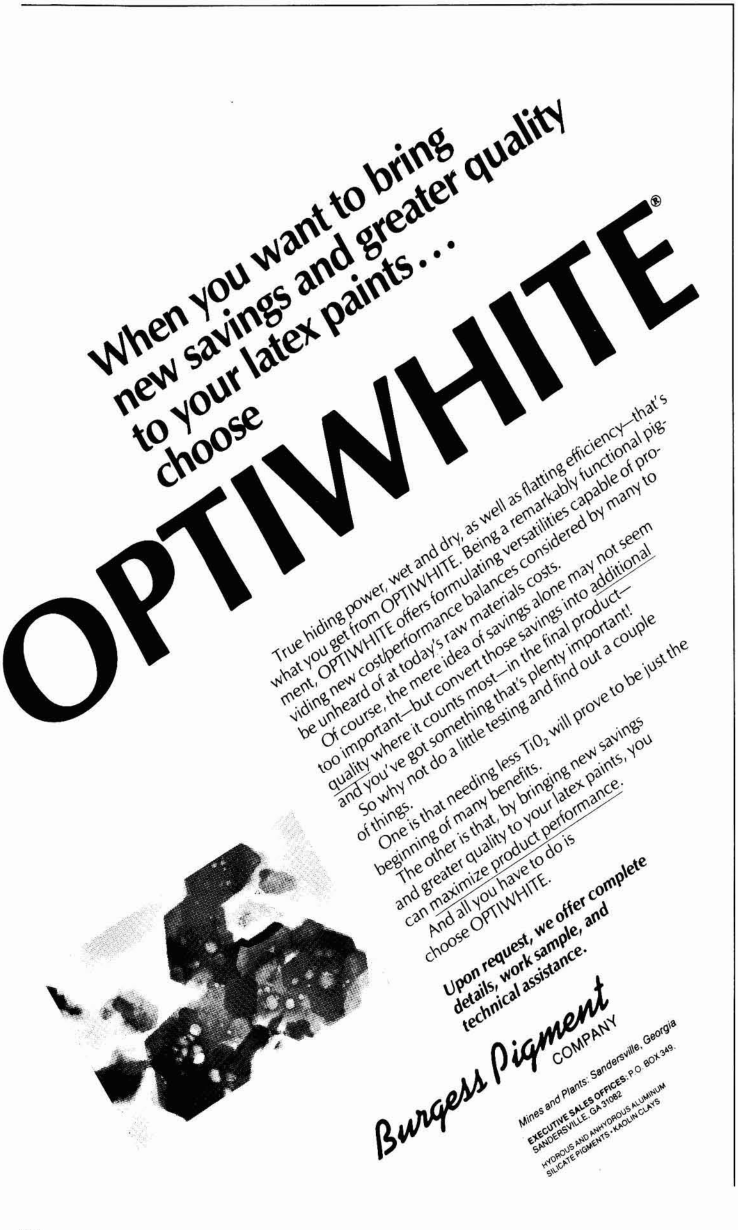
ROCKY MOUNTAIN

Active

EBERT, ED—Valley Paint Mfg. Co., Woodscross, UT.
GROVER, RON—New Mexico Highway Dept., Santa Fe, NM.
JOHNSON, JEFFREY B.—Sashco, Inc., Denver, CO.
SILVA, PAUL D.—Kwal Paints, Inc., Denver.
STULL, DEAN P.—Hauser Laboratories, Boulder, CO.
WOODHULL, LEE—Hauser Laboratories, Boulder.

Associate

HAMNER, JIM—Union Carbide Corp., Dallas, TX.
MALLETT, MICHAEL J.—Union Chemicals Div., La Mirada, CA.
MITCHELL, JOHN—Chemical Distributors, Portland, OR.
PURCELL, THOMAS E.—Rohm and Haas Co., Dallas.
STALLMO, KEVIN—Dow Chemical USA, Dallas.



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ANGH

People

The American Society for Testing and Materials (ASTM) has announced its new officers of the Board of Directors. **Lendell E. Steele,** head of the Thermostructural Materials Branch of the Naval Research Laboratory, Washington, D.C., will serve a one-year term in 1985 as Chairman of the Board. Mr. Steele previously served as Vice-Chairman of the Board, 1983–84, and as Director, 1980–82.

Also elected to the Board were: Vice-Chairman—**Robert Baboian;** Vice-Chairman—**Gladys B. Berchtold;** Treasurer—**David M. Greason;** and six new Directors: **Kenneth E. Benson, Howard I. Forman, Robert W. MacDonald, Theodore P. Pritsker, Peter E. Schilling,** and **Conrad J.J. Zellweger.**

Derrick N. Key has been named Executive Vice-President and General Manager for Roper Pump Co., Commerce, GA. All engineering, sales, marketing, accounting, purchasing, and production functions will be under Mr. Key's direction.

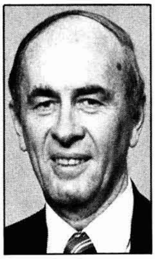
Richard E. Goodell has been named President and Chief Operating Officer of Pennsylvania Glass Sand Corp., a subsidiary of ITT. Mr. Goodell formerly served as Senior Vice-President—Operations.

Also announced by the company was the promotion of **J. Michael Zimmerman** to the position of Senior Vice-President—Marketing and Business Development. In his new capacity, he will be responsible for the marketing, sales, and product development functions of the firm.

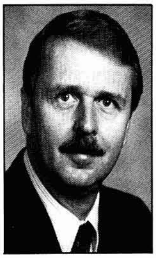
Charles W. Gilbert was awarded a Certificate of Life Membership by the Birmingham Paint, Varnish and Lacquer Club at its January meeting. Mr. Gilbert was associated with W. Canning Materials Ltd. from 1933, until his retirement in 1977. He joined the Club in 1943 and has held several committee and executive positions, including twice serving as President. He was elected to Honorary Membership in 1977.

L.E. Steele

D.N. Key



J.P. Fleming



D.L. Barker

Joseph P. Fleming has been promoted to Manager, Technical Services for Diamond Shamrock Chemical Co., Morrison, NJ. Mr. Fleming will manage technical support for all specialty chemicals products sold and will direct the technical service activities for products used in radiation curing. Mr. Fleming, who joined the firm in 1964, is a member of the American Chemical Society and the American Society for Testing and Materials.

Cook, Paint & Varnish Co., Kansas City, MO, has appointed **Melvin A. Gross** to the position of Group Vice-President of Operations. Mr. Gross's responsibilities will include manufacturing; materials management; inventory management; distribution; traffic and transportation; quality control; and safety and environmental compliance.

Raleigh Myhren has joined Morehouse Industries, Inc., Fullerton, CA, as General Sales Manager. Mr. Myhren succeeds **Dale H. Morehouse,** a director of the company, who has returned to his own medical office management company.

Ball Chemical Co., Glenshaw, PA, has promoted **Paul R. Yingst** to Senior Chemist and Manager of Research and Development in recognition of outstanding work in the development of high solid coatings, epoxies, and electrical insulating varnishes. Mr. Yingst came to Ball Chemical in 1976. He is a member of the American Chemical Society and its Organic Coatings Division.

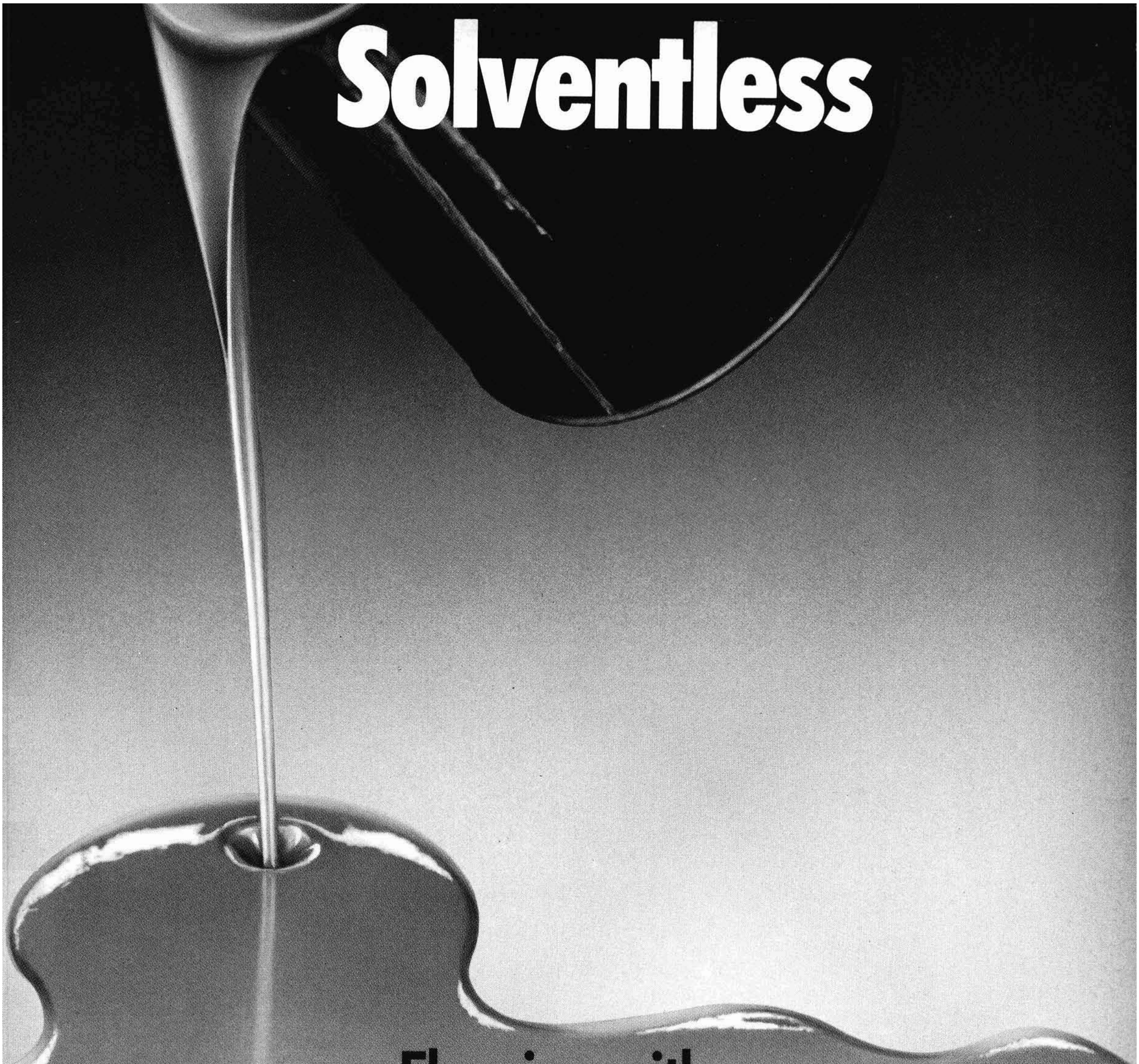
David L. Barker has joined the Coatings Division of Ferro Corp., Cleveland, OH, as Southeast Operations Manager for the company's new manufacturing facility in Wildwood, FL. Mr. Barker will be responsible for the manufacturing and sales of fiberglass reinforced plastics in the southeast portion of the U.S.

Nalco Chemical Co., Oak Brook, IL, has appointed **Shigemitsu Morita** as President of its wholly-owned subsidiary in Japan, Nalco Japan Co., Ltd. A former Director of the Board and Senior Counsel to Daicel Chemical Industries, Ltd., Mr. Morita is a veteran of over 31 years in the chemical industry.

EM Pigments, Hawthorne, NY, a division of EM Industries, Inc., has announced the addition of **Crystal Krupchak** as the Midwestern Sales Promotion Representative. Ms. Krupchak's responsibilities will include sales and technical support for the company's line of pigments as well as coordinating all regional professional societies and trade show functions.

EM Industries has also appointed **Peter Olley** to its EM Pigments Division. Mr. Olley will serve as Marketing Manager of the Plastics and Coatings Group.

Charles G. Pauli has been appointed General Manager of Koppers Coatings unit in the Construction Materials and Services Group of Koppers Co., Inc., Pittsburgh, PA. He will have overall responsibility for national sales, marketing, technical services, and production of the company's cold applied and synthetic coatings.


Solventless

FEDERATION SERIES ON COATINGS TECHNOLOGY

Unit	Title	Price (@ $3.50)
1	"Introduction to Coatings Technology"—*W. R. Fuller. (Oct. 1964) (Revised May 1973)*	
2	"Formation and Structure of Paint Films"—W. R. Fuller. (June 1965)	
3	"Oils for Organic Coatings"—F. L. Fox. (Sept. 1965)	
4	"Modern Varnish Technology"—A. E. Rheineck. (May 1966)	
5	"Alkyd Resins"—J. R. Blegen. (Mar. 1967)	
6	"Solvents"—*W. R. Fuller. (May 1967)*	
7	"White Hiding and Extender Pigments"—W. H. Madson. (Oct. 1967)	
8	"Inorganic Color Pigments"—W. R. Fuller and C. H. Love. (Mar. 1968)	
9	"Organic Color Pigments"—J. G. Mone. (July 1968)	
10	"Black and Metallic Pigments"—W. S. Stoy, E. T. Usowski, L. P. Larson, D. Passigli, W. H. Byler, R. Evdo, and W. von Fischer. (Jan. 1969)	
11	"Paint Driers and Additives"—W. J. Stewart. (June 1969)	
12	"Principles of Formulation and Paint Calculations"—W. R. Fuller. (June 1969)	
13	"Amino Resins in Coatings"—*W. L. Hensley and W. E. McGinty. (Dec. 1969)*	
14	"Silicone Resins for Organic Coatings"—R. W. Clope and M. A. Glaser. (Jan. 1970)	
15	"Urethane Coatings"—D. Lasovick. (July 1970)	
16	"Dispersion and Grinding"—M. H. Schaffer. (Sept. 1970)	
17	"Acrylic Resins"—Gerould Allyn. (Mar. 1971)	
18	*"Phenolic Resins"—R. D. McDonald. (Mar. 1971)*	
19	"Vinyl Resins"—G. M. Powell. (Apr. 1972)	
20	"Epoxy Resins"—R. A. Allen (Apr. 1972)	
21	"Nitrocellulose and Organosoluble Cellulose Ethers in Coatings"—E. C. Hamilton and L. W. Early, Jr. (Sept. 1972)	
22	"Plasticizers"—J. K. Sears (June 1974)	
23	"Interior Finishes"—Murray Abriss and Oliver Volk (Apr. 1976)	
24	"Exterior House Paint"—G. G. Schurr (May 1977)	
25	"Automotive Finishes"—*Ralph Williams (July 1977)*	
26	"Corrosion and the Preparation of Metallic Surfaces for Painting"—Clive H. Hare (Feb. 1978)	
27	"Anti-Corrosive Barrier and Inhibitive Primers"—Clive H. Hare (Feb. 1979)	

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Birmingham Paint, Varnish and Lacquer Club
c/o Mr. Ray Tennant, Carrs Paints Limited, Westminster Works, Alvechurch Rd.
Birmingham B31 3PG, England

5/83

Dr. Clifford Schoff, of PPG Industries, Inc., will present a paper, "Problems and Opportunities with Cationic ED Primers Over Zinc-Coated Steels," at the June 1985 Conference of the Oil and Colour Chemists' Association in Edinburgh, Scotland. Dr. Schoff is a Senior Research Associate with PPG's Coatings & Resins Div., in Allison Park, PA. He is the current President of the Pittsburgh Society for Coatings Technology.

During the November meeting of the Birmingham Paint, Varnish, and Lacquer Club, Distinguished Service Certificates were awarded to the following three members: **John R. Green, Roy E. Arnold,** and **David M. Heath.** Mr. Green, of B.I.P. Chemicals Ltd., has been a member for 30 years and served as Club President in 1981-82. Presently, he is Social Officer. Mr. Arnold, of Postans Ltd., joined the Club in 1962, and served as Treasurer from 1971-74 and President from 1974-75. Mr. Heath, of Holden Surface Coatings Ltd., has been a member since 1970. He was Treasurer from 1974-77, President in 1978-79, and Social Officer from 1981-84. Currently he is the Hon. Secretary.

Obituary

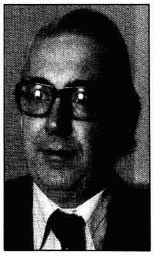
Robert J. Smith, the 18th President of the Federation (1939-40), died on January 19, in Indianapolis, IN. He was 75.

Mr. Smith was President of Indianapolis Paint and Color Co. for many years and retired from that company and the paint industry in 1968. For the next 11 years he was associated with the City of Indianapolis, of which he was a native, as Deputy Director of the Dept. of Public Works. He retired from that position in 1979. A graduate of Butler University, Mr. Smith was a Past-President of the C-D-I-C Society and the Indianapolis Paint and Coating Association.

Joseph Kiel Barry, founder of the Mid-States Paint and Chemical Co. of St. Louis, MO, died on December 16. He was 77 years old.

A former member of the St. Louis Society, Mr. Barry served as Vice-President of U.S. Paint and Lacquer Chemical Co. (now known as U.S. Paint Co.) After leaving U.S. Paint, he founded Mid-States Paint and became its President and Chairman. Mr. Barry contributed 55 years to the paint industry.

David H.W. Lovegrove, a Director of Carrs Paints Ltd., Birmingham, England, died on January 30, 1985. He was 52 years old.



Mr. Lovegrove joined Carrs in 1948, was promoted to Chief Chemist in 1962, and was made a Director in 1983. He was President of the Birmingham Paint, Varnish and Lacquer Club in 1972-73 and its Society Representative to the Federation Board since the fall of 1979. Mr. Lovegrove was awarded the Club's Distinguished Service Award in January 1984.

He is survived by his wife, Margaret, and three children: Robert, Karen, and Sarah.

Cleveland Society Conference in March Focused on "Advances in Coatings Technology"

The Education Committee of the Cleveland Society presented the 28th annual Technical Conference on "Advances in Coatings Technology," on March 12-13, at Case Western Reserve University, in Cleveland.

The program, divided into four, one half-day symposia, was as follows:

POLYMER AND PIGMENT DISPERSION STABILITY: THEORY AND CHARACTERIZATION—

"Acid-Base Interactions of Polymers" —Dr. F.M. Fowkes, Lehigh University.

"Hydrodynamic Methods for Studying the Colloidal Stability of Disperse Systems"—Dr. Mel Croucher, Xerox Research Centre of Canada.

"Electrophoretic Light Scattering: Application to Microemulsions"—Dr. Syed Qutubuddin, Case Western Reserve University.

DISPERSION STABILITY: APPLICATIONS—

"Dispersant Effects in Pigments and Coatings Applications"—Dr. Robert F. Conley, Mineral Resource Technology.

"Stabilization of Aqueous Oxide Pigment Dispersions"—Dr. William H. Morrison, Jr., E.I. DuPont de Nemours & Co., Inc.

"Synthetic Iron Oxide Pigments—Manufacturing and General Properties"—Dr. R.H. Odenthal, Mobay Chemical Corp.

ADVANCES IN COATINGS TECHNOLOGY—

"Galvanized Steel Surfaces"—Dr. Dennis J. Zalucha, Lord Corp.

"Advances in Water-Borne Epoxy Technology"—Dr. Paul R. Williams, Celanese Corp.

"Irreversible Deformation Processes in Thin Films"—Dr. Eric Baer, Case Western Reserve University.

RHEOLOGICAL MODELING AND CONTROL OF COATINGS—

"Rheological Models for Predicting Flow in High Solids Coatings"—Dr. David R. Bauer and Linda M. Briggs, Ford Motor Co.

"Surface Rheology of Coatings"—Dr. Jay Adin Mann, Case Western Reserve University.

"Unique Rheology Control Component for Solvent-Based Coatings Systems"—Dr. John S. Perz, The Lubrizol Corp.

Chairman of the Conference was Dr. Richard R. Eley, of Glidden Coatings & Resins Div. of SCM Corp., Strongsville, OH.

Chicago Society Manufacturing Committee Sponsored Coatings Seminar, March 26-27

The Manufacturing Committee of the Chicago Society sponsored a two-day seminar entitled "The Next Generation of Coatings Technology" on March 26-27 in Itasca, IL.

Those attending were provided with a variety of presentations covering manufacturing, energy conservation, and Illinois E.P.A. information. The topics and speakers for the seminar included:

"Manufacture, Formulation, and Application of High-Solids vs Conventional Coatings"—Robert N. Price, Spencer Kellogg Div., Textron.

"Water Reducible Industrial Coatings Today"—Rich Johnson, Cargill, Inc.

"Dispersion Methods for Ink in the 80's"—Richard J. Cochrane, Sun Chemical Corp., General Printing Ink Div.

"The Modern Horizontal Small Media Mill"—Gregory R. Liebert, Netzsch, Inc.

"High Speed Paint Filling"—Jill Lay, Consolidated Packaging Machinery.

"Filling By Weight"—William Cegles, Serac, Inc.

"State-of-the-Art Computerized Color Control"—Wesley A. Coppock, Jr., Applied Color Systems, Inc.

"Using Computer History to Plan for the Future"—Jacque Picoh.

"Automation of a Paint Plant"—Michael Kenes, Straubing, Rubin & Kenes.

"Aerosol Automatic Filling Equipment"—Dennis Whitehorn, The Kartridg Pak Co.

"Air Pallet® Container"—Charles S. Alack, Semi-Bulk Systems.

"Air Operated Double Diaphragm Pumps"—Don Vise, Wilden Pump and Engineering.

"Resins and Coatings Manufacturing Safety for the 80's and Beyond"—Charles A. Hawk, S.C. Johnson & Son, Inc.

"Energy Conservation"—John S. Streep and Richard T. Furlano, Furst Energy, Inc.

"Illinois E.P.A. Update"—Bill Child, Illinois E.P.A.

The Chicago Manufacturing Committee is headed by Marvin J. Tomberg—United States Gypsum Co. Committee members include: Secretary—Philip H. Goldblatt, PRA Laboratories, Inc.; M.S. Fujimoto—Retired, The Sherwin-Williams Co.; Sabi A. Samara, Rust-Oleum Corp.; Robert Ericsson, Rust-Oleum Corp.; and Audrey LeNoble, Carl Lechner, Inc.

Colorimetry Short Course Offered by Munsell

"Colorimetry: An Intensive Short Course for Industry," will be presented by the Munsell Color Science Laboratory at the Rochester Institute of Technology campus, May 13-16, 20-23, and June 3-6.

Directed by the R.S. Hunter Professor Franc Grum and Dr. Roy S. Berns, the class will provide technologists and business people with information of the fundamentals of color science and engineering required to understand and make effective use of colorimetric instrumentation, theory, and practice.

Early enrollment is suggested; attendance is limited to 16 participants.

Also, an advanced course in colorimetry featuring leading scientists in the field of color science has been scheduled on campus for August 26-28. Arrangements can be made to have an abridged version of this course presented at company locations.

For enrollment information or details on the company presentations, contact Ms. Martha Pschirrer, Munsell Color Science Laboratory, Rochester Institute of Technology, P.O. Box 9887, Rochester, NY 14623.

"Future Scientific Developments" Selected as Theme Of September 1986 FATIPEC Congress in Venice, Italy

The Scientific Committee for the XVIIIth Congress of FATIPEC has announced that the theme of the September 21-26, 1986 event in Venice, Italy, will be: "The Scientific Developments in the Coatings and Printing Ink Industries on the Doorway of the 21st Century."

FATIPEC is the Federation of Associations of Technicians in the Paint, Varnish, Lacquer, and Printing Ink Industries of Continental Europe. It is composed of seven member associations from France, Belgium, Holland, Germany, Italy, Switzerland, and Hungary. The official languages of FATIPEC are French, German, and English.

The hosts for the meeting in Venice will be the Italian member of FATIPEC: AITIVA (Associazione Italiana Tecnici Industrie Vernici e Affini) and the Italian Chemical Industry Association: FEDERCHIMICA (Federazione Nazionale dell'Industria Chimica Raggruppamento Industrie Vernici ed Inchiostri da Stampa).

The President of FATIPEC is Dr. Gianni Varasi, Managing Director of Maxfin S.p.A. Holding. The Vice-President, who will be in charge of arrangements for the event in Venice, is Dr. Amleto Poluzzi, of Ashland Chemical Co.


Dr. Varasi


Dr. Poluzzi

Anyone wishing to present a paper at the Congress must contact: Segreteria Organizzativa, del XVIII Congresso FATIPEC, c/o MGR s.r.l., Piazza S. Ambrogio 16, 20123 Milano, Italy.

Registration for the Congress is now open and forms are available from the Federation office in Philadelphia, or from the above address in Milano.

Running concurrently with the program sessions will be FATIPEXPO, an exhibition of raw materials, finished products, machinery, and other equipment.

NPCA Conducts 25th Marine Coatings Conference

The National Paint and Coatings Association held its 25th anniversary Marine and Offshore Coatings Conference on March 13-15, at the Omni International, Norfolk, VA.

Divided into five sessions, the conference included panel discussions and question and answer periods. The topics presented and the moderators are as follows:

"Coatings Update"—Robert Doyle, Ameron Protective Coatings Group.

"Corrosion Protection"—Theodore Dowd, Naval Ship Systems Command.

"Surface Preparation Update"—James A. Einspanier, Carboline Marine Corp.

"Specialty Coatings"—Vickie V. Verbyla, Seaguard Corp.

"Environmental, Health and Safety Regulations—How They Affect the Marine Coatings Industry and the Applicator—David Bloodgood, Devoe Marine Coatings Co.

Binks Manufacturing Offers Spray Painting Seminars

Binks Manufacturing Co., Franklin Park, IL, has announced that it will offer a series of week-long Spray Painting Seminars in 1985. Designed to help participants properly select, operate, and maintain current spray finishing equipment, the seminars will be of significant value to both the experienced operator and the novice.

For full information, including cost, accommodations, and application forms, contact the Training Div., Binks Manufacturing Co., 9201 W. Belmont Ave., Franklin Park, IL 60131.

Spray Finishing Workshop Scheduled for May

A Spray Finishing Technology Workshop will be held May 13-17 by Bowling Green State University and the DeVilbiss Co. Workshop sessions will be held at the University campus and in the Technical Center of the DeVilbiss Co., World Headquarters, Toledo, OH.

Featured will be sessions on equipment and material selection, spray finishing techniques associated with automotive refinishing, furniture, and industrial spraying. Additionally, topics associated with the latest technology—powder coating, automatic spraying and plural components—will be included.

Each participant will have an opportunity for "hands-on" practice. A package plan includes inexpensive lodging and airport transportation. Outstanding recreational and leisure opportunities also exist including golf, ice skating, and use of a new $8-million Recreation Center.

The workshop is under the direction of Dr. Richard A. Kruppa, Professor of Manufacturing Technology, School of Technology, Bowling Green State University, Bowling Green, OH 43403. For additional information, contact Ms. Judy Jennings at the above address.

"Colloids and Interfaces" Course Offered At Carnegie-Mellon, May 20-24

For the tenth year, Carnegie-Mellon University will present a short course on "Colloids and Interfaces" at its campus. Scheduled for May 20-24, the five-day seminar is designed for those who work with adhesives, sealants, wetting agents, pigments, paints, and printing inks.

The course provides a foundation in the fundamentals, as well as an introduction to the latest research techniques. "Colloids and Interfaces" covers not only equilibrium phenomena, but also key aspects of flow and transport near interfaces and in colloidal systems. Course format provides problem-solving sessions for individual interaction with the faculty, coupled with basic theory lectures and daily laboratory demonstrations.

Course Chairman is Dr. Geoffrey D. Parfitt, Professor of Chemical Engineering. The tuition of $850 includes a text, course notes, and laboratory fees.

For further information, contact Frank E. Nowak, Director of Post College Professional Education, Carnegie-Mellon University, Pittsburgh, PA 15213.

Literature

Polyamide Resin

A resin designed for the formulation of high-solids flexographic inks has been described in literature. The resin also complies with government air quality regulations, prints on substrates such as high-slip polyethylene, and offers adhesion as well as gel and water resistance. Further details on Versamid 759 polyamide resin may be obtained from Henkel Corp., 7900 W. 78th St., Minneapolis, MN 55435.

High Solids Alkyd

A new high solids short oil alkyd designed for industrial baking enamels with amino resins has been discussed in literature. The new resin formulates to a VOC of 2.75 or lower and develops hard chemical-resistant films. Details on Beckosol Synthetic Resin Solution 12-510 can be obtained from Reichhold Chemicals, Inc., Resins & Binders Div., P.O. Box 1433, Pensacola, FL 32596.

Tank Cleaning Units

A new line of tank cleaning units is featured in literature. The units incorporate the mechanical action of scrubbing brushes together with a liquid spray system and promote a cleaner and healthier working atmosphere. For additional information, contact Len Verhagen, Hockmeyer Equipment Corp., P.O. Box 113, Harrison, NJ 07029.

Cavity Pump

A newly published data sheet outlines the technical specifications of a series of progressive cavity pumps. Designed for pumping media of heavy solid concentration, high viscosity, or materials with virtually no flow characteristics, the pumps have open-throat suction and augur-feed to the rotor/stator elements. For details, contact Netzsch Inc., 119 Pickering Way, Exton, PA 19341.

Wet-Ground Mica

An extremely fine-particle, wet-ground mica that retains its platy character is described in literature. The mica is recommended for use in lubricants, inks, paper, rubber, coatings, as well as plastic resins. Samples and detailed product information can be obtained by contacting The Mearl Corporation, 41 East 42nd St., New York, NY 10017.

Spectral Comparison Chart

A four-color wall chart which will aid in the evaluation of light stabilizers may now be obtained. The chart illustrates the spectral emission characteristics of several commonly used weathering devices along with the energy output of the sun. To receive a copy, write to the Additives Dept., Ciba-Geigy Corp., Three Skyline Dr., Hawthorne, NY 10532.

Silica

A new silica designed to give efficient, economical performance as a thickener and thixotrope in a wide range of products is described in literature. The product is suitable for any composition that uses synthetic silica, and can be used to thicken putties, undercoatings, roof coatings, cosmetics, and pharmaceuticals. The informative leaflet on the Hi-Sil T-690 silica may be obtained from PPG Industries, Inc., 8 North, One PPG Place, Pittsburgh, PA 15272.

UV/Radiation Curing

Technology Marketing Corporation has published its most comprehensive book on UV/radiation curing, *UV Curing: Science & Technology, Volume II,* Edited by Dr. S. Peter Pappas, the 353-page book contains seven chapters written by the foremost experts in the field. Among the topics covered are photoinitiators, factors affecting viscosity, reactive oligomers, and weathering of UV coatings.

This volume, which contains recent advances in the field, complements two earlier books on UV curing, *A Practical Guide to UV Curing in Screen Printing Circuits and the Graphic Arts,* and *UV Curing: Science & Technology, Volume I.*

For more information, write to Technology Marketing Corporation, 17 Park St., Norwalk, CT 06851.

Respirator

The benefits of a new half-mask pressure demand air-line respirator which allows users to work for extended periods in hazardous environments has been outlined in literature. The respirator protects against gases, vapors, toxic dust, and other atmospheres not immediately dangerous to life or health. For complete information, contact MSA Co., at 600 Penn Center Blvd., Pittsburgh, PA 15235.

Epoxy System

Information is available on a newly developed paint that can cover rusty metal and aged coating systems in a single application to give an anti-corrosive finish claimed to last more than five years in most industrial environments. Inquiries on the Interplus 770 system should be directed to Barry Hendrix, Marketing Manager, International Paint, P.O. Box 920762, 6001 Antoine, Houston, TX 77292.

Filter Vessel

A bulletin outlining the performance specifications and optional equipment of a 14-bag, high capacity filter vessel is now available. The vessels are made to exact ASME code and OSHA compliance. To obtain a copy, write to FSI, Filter Specialists, Inc., 100 Anchor Rd., Michigan City, IN 46360.

Coating Thickness Gauges

A new family of hand-held microprocessor-based gauges, designed for economical on-the-job measurement and analysis of the thickness of non-magnetic coatings on ferrous and non-ferrous surfaces, is described in a product bulletin. These battery-operated instruments offer memory, statistics, and a hard copy printout. Further details can be obtained from Elcometer, Inc., P.O. Box 1203, Birmingham, MI 48012.

Water-Soluble Tin Compounds

Data sheets featuring two new water-soluble tributyltin tin compounds—tributyltin methane-sulphonate and tributyltin ethane-sulphonate—may now be obtained. These compounds could lead to expanded uses in the manufacture of improved biocides. A free copy is available on request from Tin Research Institute, Inc., 1353 Perry St., Columbus, OH 43201.

Analyzer

Featured in a four page, full-color pamphlet is a new sub-micron particle size analyzer. The instrument is ideal for the analysis of a wide variety of particles including pigments, iron oxides for magnetic recording tapes, silver halide emulsions, and small polymers. Contact Vickers Instruments, Inc., P.O. Box 99, 300 Commercial St., Malden, MA 02148.

Horizontal Mills

A new horizontal mill system featuring efficient processing, superior wear performance, and quick media removal has been developed. For more details on this cost-saving, state-of-the-art mill, write to Morehouse Industries, Inc., P.O. Box 3620, Fullerton, CA 92632.

Color Statistical Software

A new software package suited to the color control needs of automotive, finishes, textile, and plastics industries is described in a brochure. Write to Macbeth, P.O. Box 230, Newburgh, NY 12550, for further details on the Series 1500/PLUS system.

Balance Scale Catalog

A full-color, 20-page catalog listing specifications and features of a complete line of balances and scales has been published. For a free copy, write to Carole Rudosky, Mettler Instrument Corp., P.O. Box 71, Hightstown, NJ 08520.

Liquid Storage Tanks

Information describing the composition and properties of flat bottom storage tanks has been published. To learn more about the tanks, which hold from 6,000 to 30,000 gallons, contact Certified Equipment & Mfg. Co., P.O. Box 298, Springfield, IL 62705.

Aqueous Urethane Dispersions

A guide to aqueous urethane dispersions that categorizes typical properties and suggested uses for eight products in a company's line is now available. Uses for each product are listed by industries which include plastics, metal, wood, concrete, leather and textiles. For copies, contact the Organics Div., Witco Chemical Corp., 1000 Convery Blvd., Perth Amboy, NJ 08861.

Sodium Carboxymethyl Cellulose

Performance specifications of a new series of sodium carboxymethyl cellulose (CMC) products is the focus of a publication. These products are reported to have a lower molecular weight and are designed to improve performance in rheology control, film forming, and emulsion stabilization/polymerization. They have uses in detergents, adhesives, printing, textiles, and paper coatings. For copies, contact Product Inquiry, Hercules Incorporated, Hercules Plaza, Wilmington, DE 19894.

Synthetic Resin Emulsion

Performance specifications of a new synthetic resin emulsion are highlighted in a recently published bulletin. The new resin features blister resistance and wet adhesion, along with excellent gloss retention and touch-up capabilities. To obtain more details on the Wallpol 97-725, write to Reichhold Chemicals, Inc., Emulsion Polymers Div., P.O. Drawer K, Dover, DE 19903.

Polymer Study

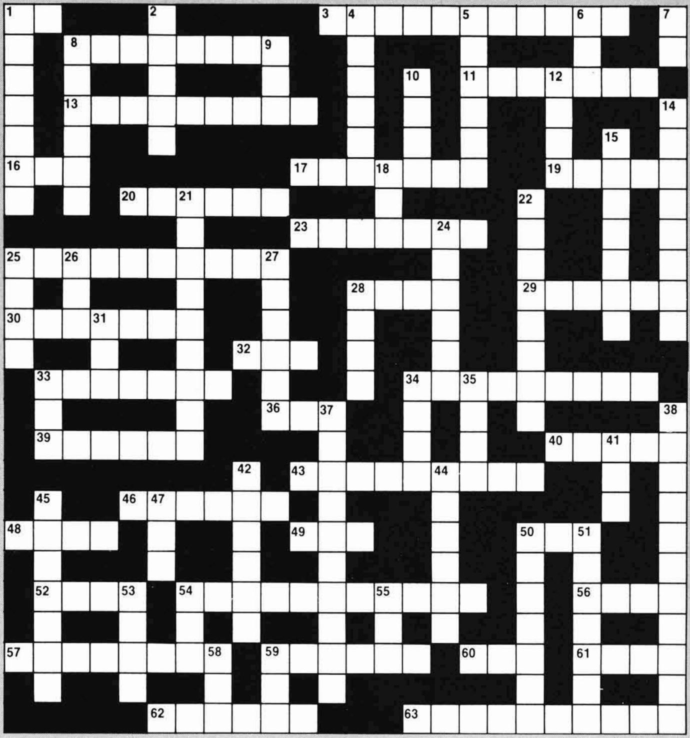
A multi-client, techno-economic marketing study entitled "Automotive Applications for Polymers" has recently been completed. The 472-page report provides an in depth analysis of some 43 different polymeric compositions utilized by the auto industry. For a free brochure containing a copy of the table of contents, write to Skeist Laboratories, Inc., 112 Naylon Ave., Livingston, NJ 07039.

High Solids Hardener

A new hardener for the formulation of high solids coatings has been developed and is described in literature. Combined with liquid epoxy resins, low VOC coatings can be formulated. This product should be considered in primers for steel and concrete, and in coatings for containers, pipes, bridges, railcars, marine, and automotive applications. For samples and a data sheet of XU 283, write: Robert Crespi, Resins Dept., CIBA-GEIGY Corp., Three Skyline Dr., Hawthorne, NY 10532.

CrossLinks

by Earl Hill



No. 5

Solution to be published in May Issue

DOWN

1. Substrate for 13 across
2. To make one lot at a time
4. Goes on last (see 59 across)
5. Elastic binder
6. Another yardstick (see 60 across) (Fin.)
7. Color Index (Abr.)
8. Repetitious sequence (Lab/Factory) (Pl)
9. Useful metal in 11 across
10. _____ balance (Lab)
12. Coating conversion process
14. Needle shaped
15. _____ acid C_6H_5COOH
18. Substituted amine (Abr.)
21. Unique liquid mixture (Chem.)
22. Siding substrate
24. "Red Oil" derivative
25. Opp. of alkaline (Chem.)
26. Lively executive attribute
27. Rare earth
28. Referred to in connection with adhesion
31. Pricing term (Abr.)
33. Container for early morn lift
34. _____ flow (Finan.)
35. Slang for 34 down
37. Diverse element form (Chem.)
38. Resinous mixture B_____
41. Resinous secretion
42. Semidrying vegetable oil A_____
44. To stick
45. Random polymer structure
47. Surfactant classification (Abr.)
50. Hydrocarbon substance or residue
51. Heat measurement unit
53. English association (Abr.)
54. Featured animal—Argus adv.
55. Color relative of 40 across
58. _____ group (Polymer Chem.)
59. Vinyl plastic (Abr.)

ACROSS

1. H-ion concentration (Abr.)
3. Measured in the Lab & elsewhere
8. Makes a reaction go
11. Bug exterminator
13. A wash coat
16. Used to see color
17. Hansa Yellow G relative
19. Philippines natural oleoresin
20. Acacia gum
23. Oxanilide
25. Either a___ or b___ (see 1 across)
28. Tote that _____
29. Close relative of 19 across
30. To repaint damaged surfaces
32. Storage location for pigments
33. Fragrant plasticizer
34. Tough flammable plastic (uses 33 across)
36. Amine (Abr.)
39. Terpene hydrocarbon
40. Luminosity
43. Organic compound (open chain)
46. Saturation (color)
48. New analytical method (Abr.)
49. Sub-technique of 48 across (Abr.)
50. Path of a spray gun
52. Place to keep tools
54. Color contributing group
56. Said of a thin resin mixture
57. Form of Al_2O_3
59. Goes on first
60. Financial yardstick (Abr.)
61. List of names
62. Chemical addition product
63. Yellow-green

Book Review

POLYMERS FOR FIBERS AND ELASTOMERS

Edited by
Jett C. Arthur, Jr.

Published by
American Chemical Society
Washington, D.C.
433 pages, $69.95

Reviewed by
Dr. Robert F. Brady, Jr.
U.S. Naval Research Laboratory
Washington, D.C. 20375

This stimulating book contains 26 papers presented at a Symposium of the Macromolecular Secretariat held at an American Chemical Society meeting in August, 1983.

The book is divided into six sections. The topics covered include: characterization of cellulosic or polypropylene fibers by instrumental methods (five chapters); synthesis and characterization of novel polymers (two chapters); elastomers and processability (four chapters); the molecular orientation of fractions of pitch and its relationship to the production of carbon fibers (five chapters); the formation, chemical modification, and surface treatment of textile and other fibers (six chapters); and the state of the art in the design and process technology of fibers with specific physical properties (four chapters).

Coatings chemists will be especially interested in the section on elastomers and processability, which contains three superb chapters on important materials with promise for future use in coatings. The first of these describes silicone and fluorosilicone elastomers and relates the characteristics of the bulk material to the molecular chemistry and physics of the elastomer. The second chapter covers phosphazines, the halogen-free polymers having a backbone of alternating phosphorus and nitrogen atoms, and describes their potential as organic polymers having low evolution of smoke and toxic gases and diminished flame spread. Another chapter describes the synthesis and characterization of vinylidene fluoride elastomers and describes their remarkable resistance to flames, solvents, heat, chemicals, and oxidation. Individuals looking for the coatings resins of the future would do well to study these three chapters.

Each chapter contains many references to the literature. The text is complemented by many clear figures and photographs, and contains useful author and subject indexes.

SOLVENT PROBLEMS IN INDUSTRY

Edited by
George Kakabadse
University of Manchester Institute of Science and Technology
Manchester, UK

Published by
Elsevier Applied Science Publishers
New York, NY
268 pages, $45.00

Reviewed by
Granville D. Edwards
Shell Chemical Co.
Houston, TX

This book is an interesting collection of papers from the 3rd and 4th European Solvents Symposia for Industry, held at the University of Manchester Institute of Science and Technology, Manchester, UK in 1983.

These "first of their kind" symposia and the book deal with the science and industrial applications of solvents highlighting new developments and problem areas. The value of the book is enhanced by the first-hand perspectives offered by each author from their individual fields of experience and expertise. Topics are discussed, beginning with basics in a clear and concise fashion.

The book is divided into four sections including: "General," dealing with fundamentals of solvent chemistry and an introduction to solvents uses; "Applied," discussing uses of solvents in specific industries; "Solvent Recovery and Disposal," including a section on heat recovery from solvent incineration; and "Health, Legislation and Safety," presenting a guide for determining the hazards of chemical compounds and the latest EEC directives for labeling solvents.

Some topics easily could have been treated in greater depth and supported by additional references. However, the extensive number of subjects discussed makes the book a valuable reference for anyone interested in solvents.

Letters to the Editor

"Iacocca"—Required Reading

To the Editor:

Occasionally, a best seller comes along which merits the recommendation of must reading. Such a book is the current best seller, *Iacocca,* the autobiography of Chrysler's Chairman, Lee Iacocca.

Iacocca's story clearly demonstrates that the American Dream is still possible when coupled with hard work, strong moral character, and driving determination in the face of overwhelming odds. Interwoven between two career opportunities, Mr. Iacocca describes his love for the automotive business and his rise to the top of Ford—only to be fired.

Rising from the ashes of this experience he takes over Chrysler as the ship is sinking. He successfully challenges and overcomes these obstacles: a negative press, a failing economy, hostile bankers, labor, foreign competition, Congress, and a poor public attitude toward Chrysler. All in all this is the miracle of the decade.

This book must be read by Congress and all those who have a stake in America's future. We in the coatings industry have a big stake in the automotive industry but, more importantly, in America's future and need to learn from someone who has been over the brink and returned.

Read the book!!

Thomas J. Miranda
Whirlpool Corp.
Benton Harbor, MI

Coming Events

FEDERATION MEETINGS

For information on Federation meetings, contact FSCT, 1315 Walnut St., Philadelphia, PA 19107 (215-545-1506).

(Apr. 17-18)—Symposium on Color and Appearance Instrumentation. Jointly sponsored by FSCT, Manufacturers Council on Color and Appearance, and Inter-Society Color Council. Sheraton Station Square, Pittsburgh, PA.

(May 14-17)—Federation "Spring Week." Seminar on 14th and 15th; Society Officers on 16th; and Board of Directors on 17th. Omni International/Baltimore, MD.

(Oct. 7-9)—63rd Annual Meeting and 50th Paint Industries' Show. Convention Center, St. Louis, MO.

1986

(May 13-16)—Federation "Spring Week." Seminar on 13th and 14th; Society Officers on 15th; and Board of Directors on 16th.

(Nov. 5-7)—64th Annual Meeting and 51st Paint Industries' Show. World Congress Center, Atlanta, GA.

SPECIAL SOCIETY MEETINGS

(Apr. 17)—Baltimore Society Mini-Show. Baltimore Hilton, Pikesville, MD. (Joseph Giusto, Lenmar, Inc., P.O. Box 4434, Baltimore, MD 21223).

(Apr. 25-27)—Pacific Northwest Society. Annual Symposium. Empress Hotel, Victoria, B.C. (Ottwin Schmidt, Helzer Canada Ltd., 8531 Cullen Crescent, Richmond, B.C., V6Y 2W9 Canada).

(May 6)—Philadelphia Society Seminar on "Additives for New Technology Coatings." Airport Hilton, Philadelphia, PA. (Wayne A. Krause, Hercules Incorporated, Research Center, Wilmington, DE 19899).

(June 14-15)—Joint Meeting of St. Louis and Kansas City Societies for Coatings Technology. Lake of Ozarks, MO.

(Nov. 5-6)—Symco '85. 15th Annual Chicago Society Coatings Symposium, "The Right Stuff." Knickers Restaurant, Des Plaines, IL. (Lonnie U. Haynes, Chairman, Hercules, Incorporated, 300 E. Schuman Blvd., One Energy Center, Suite 260, Naperville, IL 60540).

1986

(Mar. 25-27)—Southern Society. Annual Meeting. Hilton Hotel, Savannah, GA. (Ronald R. Brown, Union Chemicals Div., P.O. Box 26845, Charlotte, NC 28213).

(Apr. 9-11)—Southwestern Paint Convention of Dallas and Houston Societies. Houston, TX. (Mike Winters, Ribelin Distributors, Inc., 7766 Blankenship, Houston, TX 77055).

OTHER ORGANIZATIONS

(Apr. 15-16)—25th Annual Technical Symposium, Washington Paint Technical Group. Twin Bridges Marriott Hotel, Washington, D.C. (Ken Zacharias, NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(Apr. 15-17)—Short Course on "Estimating for Painting Contractors and Maintenance Engineers." University of Missouri—Rolla, Rolla, MO. (Paint & Coatings Program, Chemistry Dept., Univ. of Missouri—Rolla, Rolla, MO 65401).

(Apr. 16-18)—PaintCon '85. O'Hare Expo Center, Chicago, IL. (Professional Exposition Management Co., Inc., Ste. 205, 2400 E. Devon Ave., Des Plaines, IL 60018).

(Apr. 22-26)—Short Course on "High Temperature Materials and Coatings." Windsor Castle Hotel, Great Britain. (Continuing Education Institute—Europe, Rörtorpsvägen 5, S-612 00 Finspang, Sweden).

(Apr. 24-25)—"Professional Development—Pigments, Whites, and Extenders." Sponsored by the Protective Coatings Div., Chemical Institute of Canada. Old Mill Restaurant, Toronto, Ont. (April 24) and Le Pavillion Hotel, Montreal, Quebec (Apr. 25). (J. Fiocco, Symposium Chairman, NL Chemicals, Canada, 2140 Sun Life Bldg., Montreal, Que., Canada H3B 2X8).

(Apr. 28-May 3)—189th National Meeting. American Chemical Society. Miami Beach, FL. (ACS, A.T. Winstead, 1155 16th St., N.W., Washington, D.C. 20036).

(May 6-8)—RadCure '85—Association for Finishing Processes of the Society of Manufacturing Engineers Conference and Exposition. Basel, Switzerland. (AFP/SME Public Relations, Society of Manufacturing Engineers, One SME Dr., Dearborn, MI 48121).

(May 6-10)—"Dispersion of Pigments and Resins in Fluid Media" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(May 13-16)—"Colorimetry: An Intensive Short Course for Industry." Munsell Color Science Laboratory, Rochester Institute of Technology campus, Rochester, NY. (Martha Pschirrer, Munsell Color Science Laboratory, RIT, P.O. Box 9887, Rochester, NY 14623).

(May 13-17)—Spray Finishing Technology Workshop. Sponsored by Bowling Green State University, Bowling Green, OH and DeVilbiss Co., Toledo, OH. (Judy Jennings, School of Technology, Bowling Green State University, Bowling Green, OH 43403).

(May 13-17)—Short Course on "Physical Testing of Paints and Coatings." University of Missouri—Rolla, Rolla, MO. (Paint & Coatings Program, Chemistry Dept., Univ. of Missouri—Rolla, Rolla, MO 65401).

(May 20-23)—Steel Structures Painting Council Annual Meeting and Symposium. Netherlands Plaza Hotel, Cincinnati, OH. (Harold Hower, SSPC, 4400 Fifth Ave., Pittsburgh, PA 15213).

(May 20-23)—"Colorimetry—An Intensive Short Course for Industry." Munsell Color Science Laboratory, Rochester Institute of Technology campus, Rochester, NY. (Martha Pschirrer, Munsell Color Science Laboratory, RIT, P.O. Box 9887, Rochester, NY 14623).

(May 20-24)—"Adhesion Principles and Practice for Coatings and Polymer Scientists" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(May 20-24)—"Colloids and Interfaces" Short Course, Carnegie-Mellon University, Pittsburgh, PA. (Frank E. Nowak, Director of Post College Professional Education, Carnegie-Mellon University, Pittsburgh, PA 15213).

(May 28-June 2)—International Surface Finishing and Printed Circuit Board Making Exhibition. Beijing, People's Republic of China. (Sino Trade Promotions, Tak Cheung Building, 22-24 Wing Lok St., Central, Hong Kong).

(June 3-6)—"Colorimetry: An Intensive Short Course for Industry." Munsell Color Science Laboratory, Rochester Institute of Technology campus, Rochester, NY (Martha Pschirrer, Munsell Color Science Laboratory, RIT, P.O. Box 9887, Rochester, NY 14623).

(June 3-7)—"Advances in Emulsion Polymerization and Latex Technology" Short Course. Lehigh University, Bethlehem, PA. (Dr. Mohamed S. El-Aasser, Dept. of Chemical Engineering, Sinclair Lab #7, Lehigh University, Bethlehem, PA 18015).

(June 3-7)—"Applied Rheology for Industrial Chemists" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(June 10-12)—International Conference on Biologically Influenced Corrosion, sponsored by the National Association of Corrosion Engineers, Washington, DC. (Meetings Manager, NACE Headquarters, P.O. Box 218340, Houston, TX 77218).

(June 11-13)—Eastern Plant Engineering & Maintenance Show and Conference. Georgia World Congress Center, Atlanta, GA. (Show Manager, Plant Engineering & Maintenance Shows, 999 Summer St., Stamford, CT 06905).

(June 16-19)—Dry Color Manufacturers' Association. Annual Meeting. The Greenbriar, White Sulphur Springs, WV. (DCMA, 206 N. Washington St., Ste. 202, P.O. Box 931, Alexandria, VA 22313).

(June 26-29)—Oil & Colour Chemists' Association's Biennial Conference. Edinburgh, Scotland. (R.H. Hamblin, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, HAO 2SF England).

(July 4-7)—Oil and Colour Chemists Association Australia. 27th Convention. Hunter Valley, NSW, Australia. (Ted Saultry, 46 Tamarisk Ave., Glen Waverly VK.3150, Australia).

(July 8-12)—11th International Conference on Organic Coatings Science and Technology. Athens, Greece. (Dr. A.V. Patsis, Institute in Materials Science, S.U.N.Y., New Paltz, NY 12561).

(Aug. 26-28)—Advanced Colorimetry Course. Munsell Color Science Laboratory, Rochester Institute of Technology campus, Rochester, NY. (Martha Pschirrer, Munsell Color Science Laboratory, RIT, P.O. Box 9887, Rochester, NY 14623).

(Sept. 2-4)—Federation of Scandinavian Paint and Varnish Technologists. 11th Congress. SAS Hotel Scandinavia, Oslo, Norway. (Paal Ivan, Nodest Industries A/S, Boks 500, N-3001 Drammen, Norway).

(Sept. 2-6)—Short Course on "Films and Coatings for Technology." Davos Congress Center, Switzerland. (Continuing Education Institute—Europe, Rörtorpsvägen 5, S-612 00 Finspang, Sweden).

(Sept. 9-13)—Short Course on "Nitride and Carbide Coatings." LSRH Research Center, Neuchatel, Switzerland. (Continuing Education Institute—Europe, Rörtorpsvägen 5, S-612 00 Finspang, Sweden).

(Sept. 17-19)—FINISHING '85. Cobo Hall, Detroit, MI. (AFP/SME, P.O. Box 930, Dearborn, MI 48128).

(Sept. 21-24)—Canadian Paint and Coatings Assn. 73rd Annual Convention. Clevelands House, Minnett, Ont. (CPCA, 515 St. Catherine St. W., Montreal, Que., Canada H3B 1B4).

(Oct. 8-11)—A.F.T.P.V. (French Association of FATIPEC). Biennial meeting. Strasbourg, France. (J. Roire, AFTPV, 5 Rue Etex, 75018 Paris, France).

(Oct. 14-18)—European Conference on Applications of Surface and Interface Analysis. Veldhoven, The Netherlands. (ECASIA Conference Bureau: QLT Convention Services, Keizersgracht 792, 1017 EC Amsterdam, The Netherlands).

(Nov. 4-6)—National Paint and Coatings Association. 98th Annual Meeting. Hilton Hotel, New Orleans, LA. (Karen Bradley, NPCA, 1500 Rhode Island Ave. N.W., Washington, DC 20005).

(Nov. 6-8)—National Coil Coaters Association Fall Technical Meeting. Hyatt Regency Dearborn, Dearborn, MI. (Tony Carroll, NCCA, 1900 Arch St., Philadelphia, PA 19103).

(Nov. 15-17)—38th National Decorating Products Association Show. McCormick Place, Chicago, IL. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

1986

(Apr. 14-15)—ASTM Symposium on "Testing of Metallic and Inorganic Coatings," Chicago, IL. (Teri Carroll, ASTM Standards Development Div., 1916 Race St., Philadelphia, PA 19103).

(Sept. 8-13)—190th National Meeting. American Chemical Society. Chicago, IL. (ACS, A.T. Winstead, 1155 16th St., N.W., Washington, D.C. 20036).

(Sept. 9-11)—RadCure '86—Association for Finishing Processes of the Society of Manufacturing Engineers Conference and Exposition. Baltimore Convention Center, Baltimore, MD. (AFP/SME Public Relations, Society of Manufacturing Engineers, One SME Dr., Dearborn, MI 48121).

(Sept. 21-26)—XVIIIth Congress of FATIPEC. (Federation of Associations of Technicians in the Paint, Varnish, and Printing Ink Industries of Continental Europe). Venice, Italy. (C. Bourgery, Secretary General of FATIPEC, 76 Blvd. Pereire, 75017 Paris, France - or - Amleto Poluzzi, AITIVA, Piazzale R. Morandi 2, 20121 Milano, Italy).

Advertisers Index

'Humbug' from Hillman

Aging Marty Miller has been seen jogging ("staggering" is probably the better word) around New York City wearing a T-shirt with words that announce his philosophy—

> *"Age and Treachery Will Always Win*
> *Over Youth and Skill"*

• • •

From the crew at Polyvinyl Chemical Industries, some overheard comments in the paint lab on clean-up day:

- What the hell is this!
- It's not labeled.
- Chuck it.
- It's as hard as a rock.
- That was discontinued in 1978.
- How old is this?
- Was it this color when we got it?
- Who ordered this?
- How do we legally dispose of this?
- Is that supposed to be growing in there?
- Who was using that color?
- You'll be blowing that stuff out of your nose all weekend.
- Leave it there and let someone else use it.
- What's that—floating on it?
- Arrange them alphabetically.
- I could give you lessons on cleaning.
- How long do you think it will stay this way? . . . Until research gets to it.

And in conclusion there's always one wise guy who will ask—

- Where's the defoamers??!!

• • •

Our transplanted friend, Howard Jerome, acknowledges that he talks differently from his St. Louis neighbors. To help them comprehend his well-ingrained pronunciations, he quotes from Dana Lynn Wilson's 1976 book, "Moa Boston English."

For example, he's had to explain with the help of Ms. Wilson that Bawstonians buy "cans of tuner" at the "mocket." Other helpful examples are—

- *Lahr:* a system of legal rules. "If you break the lahr, you may get arrested."
- *Khakies:* Metal instruments for opening automobile doors. Howard says he has given up driving his "cah" for an automobile.
- *Airier:* A particular extent of land. "Cousin Buffy hahd to leave the Bawston airier. Poa thing."
- *Torrist:* a person who travels.
- *Mock:* To give heed to. "Mock my words. Some day the rest of Ameriker will lun to speak like Bawstonians."

In the January issue of *Materials Performance,* Howard found some very important supporting information. In the article, "How to survive in Boston-a visitors guide to the language," the author, who wisely identifies himself only as JCD, gives some valuable hints. Among them:

- One goes "up" to Boston and "down" to anywhere else, particularly Maine.
- Worcester is "Wooster."
- Peabody is "Pee Buddee."
- People seldom say Massachusetts (Can you blame them?) They say, "Mass Pike" for Massachusetts Turnpike and "Mass av."
- "Scrod" is young codfish.
- "Dropped" egg is poached.
- You season food with a salt "cellar."

Howard concludes by noting, "Frankly, I understand "Boston English" with no difficulty. It's the rest of the country I worry about."

• • •

I usually hesitate in quoting from the *Chemical and Engineering News* because so many Humbug fans are subscribers but Milt Glaser found the following, which I quote, in excerpt, since it is apropos.

As noted in Newscripts, Washington (D.C.) is creating revolting new words, often by manufacturing verbs from other parts of speech.

"We're going to incentivize these people by multiyearing their programs." Newscripts observes, "This sort of thing does not reach the heights of imbecility typified by "human resources" (i.e., people), but it will do for the nonce."

Milt Glaser quotes the following comment by Disraeli about his opponent, Gladstone. To how many bosses would this apply?—

"I wish I was as sure of anything as he is sure of everything."

Milt also says that, "Sometimes the process is as important as the project itself."

—Herb Hillman
Humbug's Nest
P.O. Box 135
Whitingham, VT 05361

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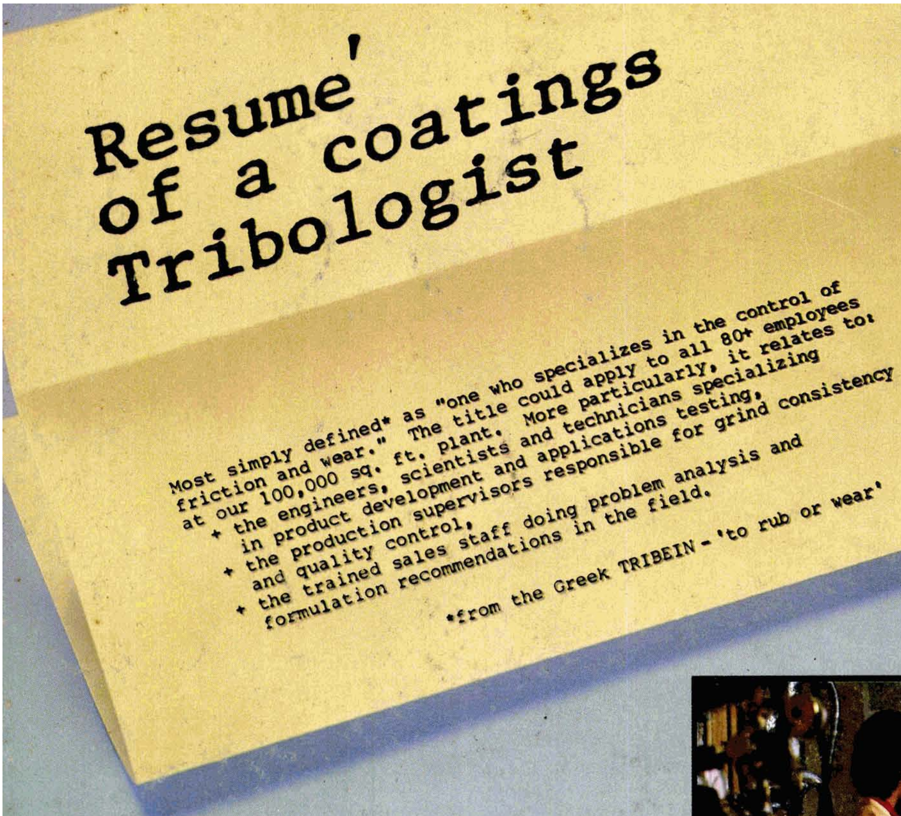

Resume' of a coatings Tribologist

Most simply defined* as "one who specializes in the control of friction and wear." The title could apply to all 80+ employees at our 100,000 sq. ft. plant. More particularly, it relates to:

- the engineers, scientists and technicians specializing in product development and applications testing,
- the production supervisors responsible for grind consistency and quality control,
- the trained sales staff doing problem analysis and formulation recommendations in the field.

*from the Greek TRIBEIN - 'to rub or wear'

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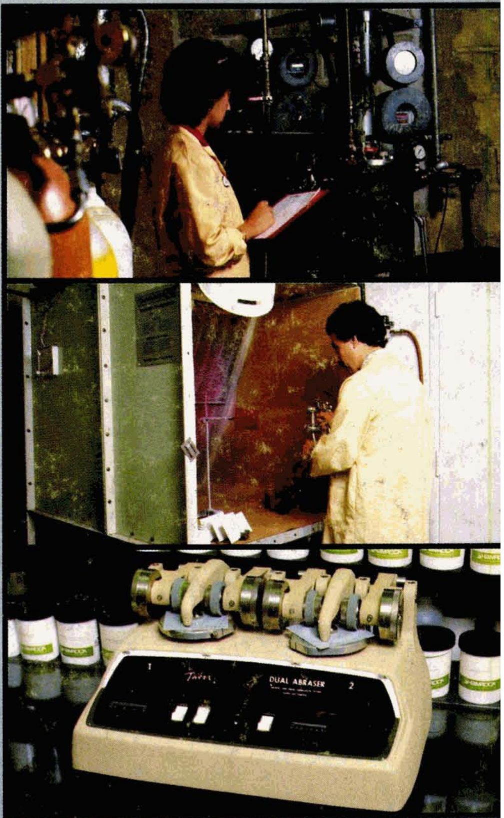

TOP: *Reactor produces developmental polyethylene waxes.* MIDDLE: *Samples are prepared by roller, brush, draw-down and spray application techniques.* BOTTOM: *Testing is conducted for slip and friction, rub and mar, and wear and abrasion.**

*Taber Abraser shown distributed by Testing Machines, Inc.

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