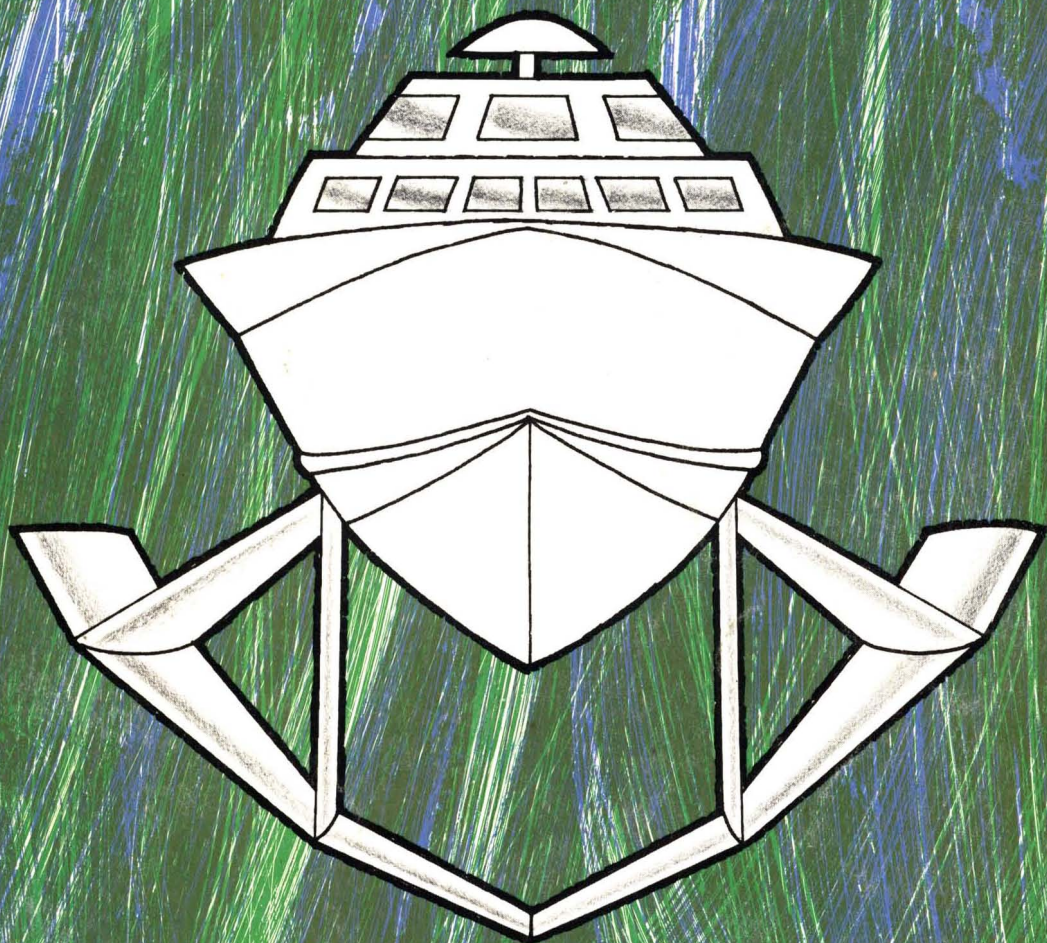


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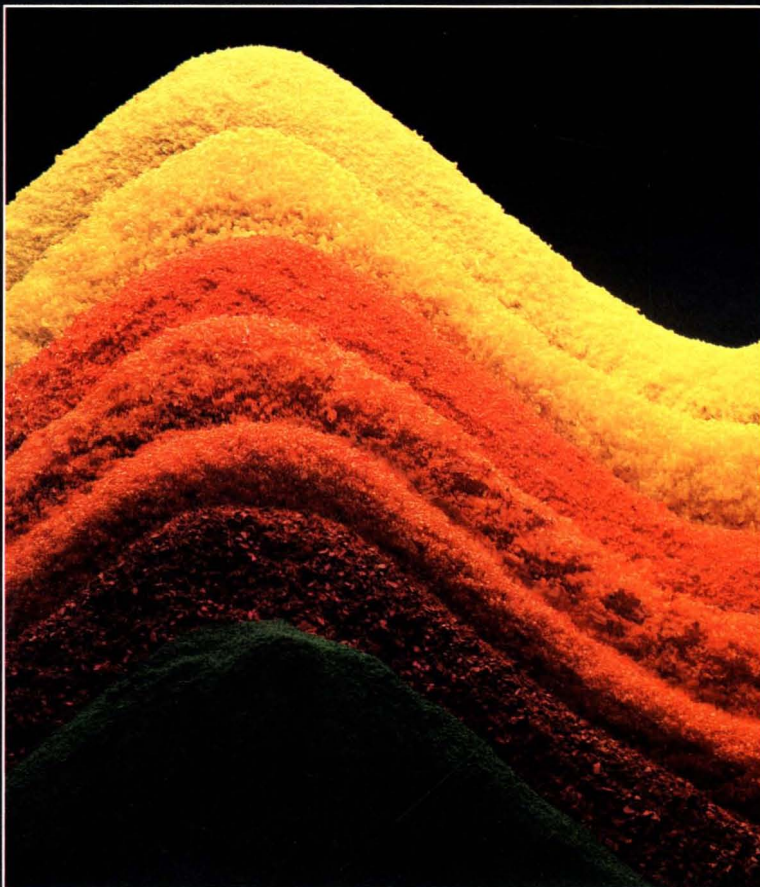
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So that the membership may again be informed of the sources of income and the areas of expense, the per cent allocations for each, as estimated for 1980, are as follows:

Income

- 44% **PUBLICATIONS**—JCT advertising and non-members subscriptions; Series on Coatings Technology; Paint/Coatings Dictionary; Infrared Spectroscopy Atlas; Year Book advertising; Pictorial Standards Manual.
- 42% **ANNUAL MEETING AND PAINT SHOW**—Sale of exhibit space; Registration fees; Program Book advertising.
- 10% **MEMBERSHIP DUES**—6,500 members in 26 Societies.
- 2% **EDUCATIONAL ACTIVITIES**—Color-matching Aptitude Test Set; Audio/visual programs.
- 2% **MISCELLANEOUS**—Miscellaneous sales; Investment Interest.

Expense

- 34% **HEADQUARTERS ADMINISTRATION**—Operation of headquarters office and staff in Philadelphia; Communications activities.
- 34% **PUBLICATIONS**—Printing, mailing, and promotion.
- 14% **ANNUAL MEETING AND PAINT SHOW**—Promotion and management of these twin annual events.
- 10% **EDUCATIONAL ACTIVITIES**—Support of: Paint Research Institute; Scholarships at five universities; Educational Committee; Grants for research to other industry organizations; Correspondence course at University of Southern Mississippi; and production of Color-matching Aptitude Test Set.
- 8% **OFFICERS, BOARD, AND COMMITTEES**—Travel expenses for officers; Transportation expense for Board of Directors, Executive Committee, and national meetings of such committees as Educational, Manufacturing, and Technical.

While it is true that inflation has affected some of these figures, it is also true that there is an encouraging amount of growth in all major areas.

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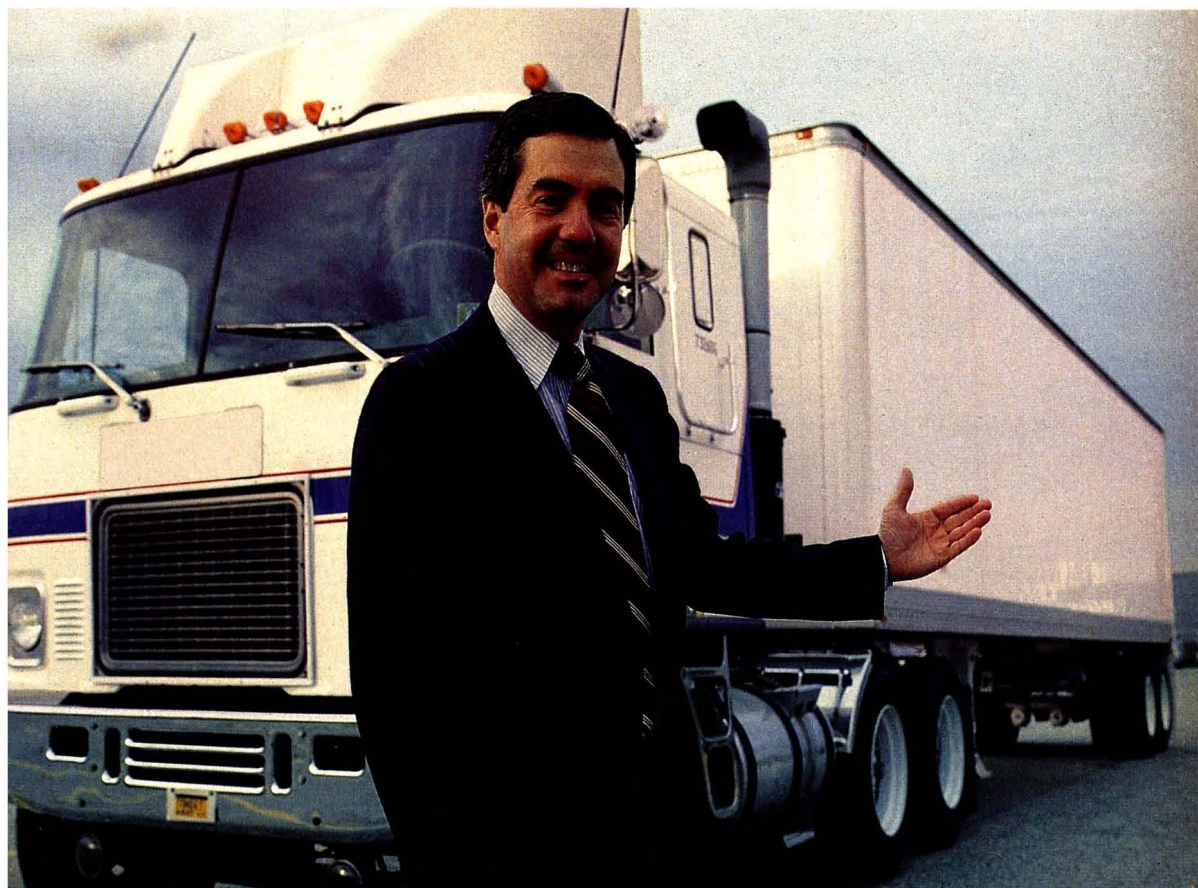
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PERFORMANCE OF SELECTED MARINE COATINGS EXPOSED TO HIGH VELOCITY SEAWATER—G.A. Gehring, Jr.

Journal of Coatings Technology, 52, No. 661, 35 (Feb. 1980)

A study was undertaken to investigate seawater velocity thresholds and failure modes for three selected marine coatings under non-cavitating, parallel flow ranging from 3 to 30 m/s. The study was accomplished in a flow channel using natural seawater. The results indicate that: (1) the velocity threshold for two of the three coatings investigated might exceed 30 m/s; (2) blister formation appears to be the critical part of the damage process leading to gross failure; (3) under the given set of experimental conditions, hydromechanical wear is negligible; (4) electrical impedance measurements enable non-destructive detection of impending coating failure; and (5) parallel flow is not as damaging as impinging or cavitating flow in the velocity range between 15 and 30 m/s.

PYROLYSIS GAS CHROMATOGRAPHIC ANALYSIS (PGC) OF METHYL METHACRYLATE (MMA)-ETHYL ACRYLATE (EA) COPOLYMERS—S. Paul

Journal of Coatings Technology, 52, No. 661, 47 (Feb. 1980)

The use of pyrolysis gas chromatography (PGC) for the quantitative estimation of methyl methacrylate (MMA)-ethyl acrylate (EA) copolymer composition has been demonstrated. In the light of the existing polymer degradation theories, optimum pyrolysis conditions have been investigated. A calibration curve for the estimation of copolymer composition in unknown polymer samples has also been obtained from the NMR and the PGC results. Based on the thermogravimetric analysis (TGA) of the copolymers containing varying mole-fractions of MMA-EA monomers, activation energies for the thermal degradation has been determined. Activation energy for the MMA-EA copolymer containing 20 mole % of EA has been found to be 14.7 K cal/mole. This observed value was lower, however, than the value reported earlier for poly-(methyl methacrylate) (PMMA), i.e., 42 K cal/mole. This has been supposed to be due to the low molecular weight of the copolymer samples investigated in our case.

WATER-BORNE SURFACE TREATMENT FOR WOOD BASED ON ZINC SALTS AND A LATEX: AN SEM/EDXA INVESTIGATION—R.L. Desai and W.A. Coté, Jr.

Journal of Coatings Technology, 52, No. 661, 57 (Feb. 1980)

The distribution and location of the components of water-borne protective zinc salt-latex systems applied to wood have been examined by scanning electron microscopy and energy dispersive x-ray analysis (SEM/EDXA) technique. Effects of soaking time, method of treatment application, and leaching of the treated wood on the distribution of the components have been investigated. The results indicate that most of the zinc was deposited on the surface when applied as the carbonate salt, but that it penetrated the substrate surface with a thiocyanate salt system. However, the thiocyanate salt system was the more leachable of the two. Increased soaking time resulted in increased latex penetration into the substrate, and it appears that the latex diffused into the substrate through ray tracheids or pits or both.

URETHANE COATINGS FROM PROPOXYLATED FATTY ACIDS AND EPOXIDIZED TALLOW—M. Zubillaga and E. Saggese

Journal of Coatings Technology, 52, No. 661, 63 (Feb. 1980)

Threo- and *erythro-*9,10-dihydroxystearic acid and azelaic acid were reacted with varying amounts of propylene oxide under basic catalysis to form polyether polyols with hydroxyl numbers ranging from 165 to 320. The polyols were evaluated as resin components in coating formulations with three commercial polymeric isocyanates. The fatty acid-based coatings were compared with coatings obtained from three commercial polyols. The properties of the films from fatty acid-derived polyols were similar to those of the controls. Additional films were prepared with the fatty polyols derived from the propoxylated products from the reaction of epoxidized tallow with sorbitol or lactitol. This latter work expands the use of polyols derived primarily from vegetable oils to include those of animal origin.

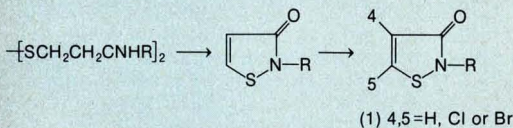
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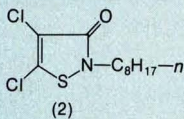
3(2H)ISOTHIAZOLONE, A NEW CLASS OF ANTIFOULING TOXICANT—G.A. Miller and T. Lovegrove

Journal of Coatings Technology, 52, No. 661, 69 (Feb. 1980)

The 3(2H)-isothiazolone (1) compounds as a general class have demonstrated high antimicrobial activity against a wide range of bacteria, fungi, and algae. The general and practical synthesis of this heterocyclic structure via amide-disulfide cyclization and subsequent halogenation have made possible rather thorough structure/activity studies in several commercially useful



areas—paint film mildewcide, industrial preservative, and water treatment microbicide. The current work describes the marine antifoulant properties of isothiazolones (1) as measured by membrane screening evaluations. Structure/activity investigations by this procedure show better activity for the ring halogenated derivatives, and specifically identify 4,5-dichloro-2-n-octyl-3(2H)-isothiazolone (2) as the optimum antifouling toxicant.



CHELATION AS A MEANS OF DEVELOPING CROSS-LINKING EMULSIONS—J.R. Grawe and B.G. Bufkin

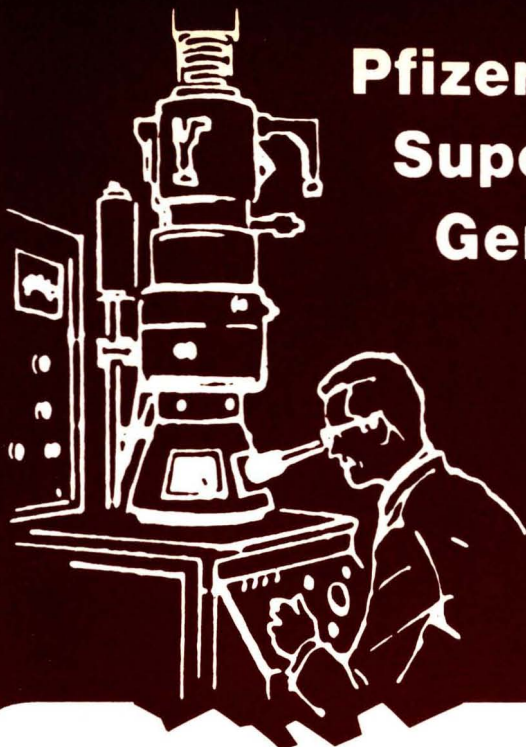
Journal of Coatings Technology, 52, No. 661, 73 (Feb. 1980)

Chelate-containing emulsions which crosslinked through the use of multivalent metal ions were investigated using 2-acetoacetoxypropyl methacrylate (2-AAPMA) as the functional monomer. The emulsions were prepared in adequate yields and with excellent tolerance to the addition of electrolytes through the use of a nonionic/anionic blend of surfactants.

The crosslinking ability of poly(2-AAPMA) was substantiated by torsional braid analysis which indicated that coatings containing ZnCl_2 cured at room temperature and after thermal treatment. Subsequently, the effectiveness of such curing agents as MgCl_2 , NiCl_2 , $\text{Al}_2(\text{SO}_4)_3$, CoCl_2 , ZnCl_2 , $\text{Zn}(\text{OAc})_2$, and SnCl_2 was evaluated using conventional physical testing of both latex and solvent-cast films of the same polymer.

Among the various crosslinking agents, $\text{Zn}(\text{OAc})_2$ at a stoichiometric level of 20% provided the most preeminent results at room temperature, yielding latex-cast coatings with excellent hardness, gloss, solvent and water resistance, and tensile strength. After thermal curing at 125°C , latex coatings containing higher levels of ZnCl_2 demonstrated very efficient crosslink densities, undergoing virtually no solvent softening after 500 dimethylformamide double rubs.

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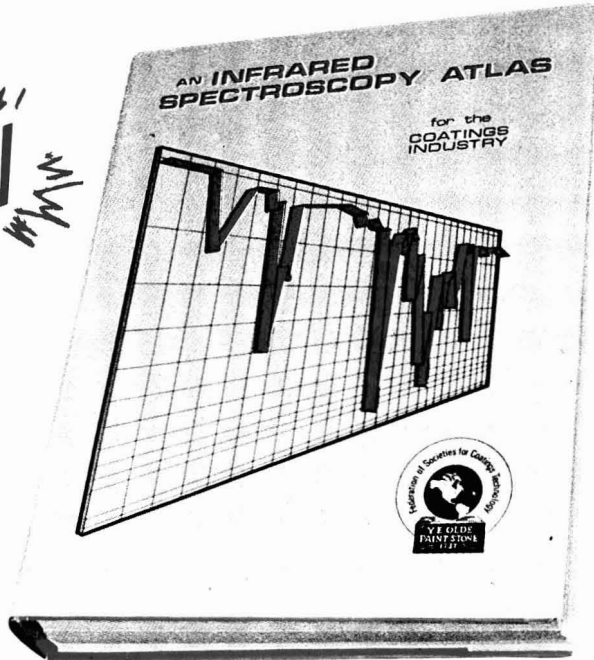
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- Hardness development: Dries to a Sward hardness of 16 – 20 in one hour.
- Hot print resistance: No print at 100°F for one hour at 4 psi.
- Cold crack resistance: Passes 4 hours at -30°F; resists cold checking for 20 cycles between 125°F and -5°F.
- Chemical resistance: Is resistant to materials such as alcohol, water, detergents, coffee, oil and soda.

Polyvinyl Chemical's NEOCRYL A-634 is a completely new, fast air-dry acrylic copolymer emulsion that performs comparably to solvent lacquers. It offers you and your customers significant economic and environmental advantages as well.

Extensive tests confirm that A-634 provides excellent results in the key performance areas of hardness, hot print, cold crack, chemical resistance and clarity.

NEOCRYL A-634 will help keep you cost competitive because high-priced organic solvents are not required. Your customers will also benefit: volatile organic emissions will be significantly reduced, facilitating environmental compliance. Lower energy costs are a further advantage.

So — when considering water for your wood finishing formulations — think of NEOCRYL A-634. For more data, write or call us.

Polyvinyl Chemical Industries

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Free paint additives kit from Dow Corning helps you paint your world.

The proof is in the painting.

Dow Corning® silicone paint additives solve an exciting variety of paint problems. In a fantasy world. And in the real world. For example, problems such as foaming, nonwetting, orange peel, and pigment separation will disappear when you add silicone paint additives to your paint formulations. And the qualities your customers look for in paints—such as gloss, slip and adhesion—will improve with the help of silicone paint additives.

When you send for your free paint additives kit you'll see, for example, how a few drops of paint additive 3 prevent pigment separation. And help keep a castle or house evenly colored from top to bottom. Paint additives 11, 14, 56, and 57 improve flow-out and

mar resistance. Which makes them great for train cars, automotive, and airplane finishes.



You'll see how paint additive 7 decreases foaming in solvent-based systems. And how paint additive 21 improves adhesion and salt-spray resistance of epoxy coatings—perfect for docks and boat hulls.

In all, there are seven silicone additives to help you solve your paint problems.

Dow Corning silicone paint additives make it easy to solve problems in your world.

The proof is in our free paint additives kit. Just write us on your letterhead and ask for a paint additives kit of your own. Send to:

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Dept. C-8580,
Midland, Mich. 48640.

DOW CORNING

DOW CORNING



FEDERATION

newsletter



122 COMPANIES SIGN UP FOR PAINT INDUSTRIES' SHOW AT ATLANTA CIVIC CENTER, OCTOBER 29, 30, 31

One hundred and twenty-two suppliers of raw materials and equipment to the coatings manufacturing industry have signed up for exhibit space in the 1980 Paint Show at the Atlanta Civic Center. They are:

Aceto Chemical Co., Inc.
Air Products & Chemicals, Inc.
Alcan Ingot & Powders
Aluminum Co. of America
C.M. Ambrose Co.
American Felt & Filter Co.
American Hoechst Corp.
American Nepheline Corp.
Applied Color Systems, Inc.
Ashland Chemical Co.
Atlas Electric Devices Co.

B.A.G. Corp.
BASF Wyandotte Corp.
Bennett's Colorant Div.
Blackmer Pump Div., Dover Corp.
Brookfield Engineering Labs
Buckman Laboratories, Inc.
Byk-Mallinckrodt Chem. Prod.

Cargill, Inc.
CDI Dispersions, Inc.
Celanese Chemical Co.
Celanese Polymers & Specialties Co.
Chicago Boiler Co.
Columbian Chemicals Co.
Color Corp. of America
Cosan Chemical Corp.

Daniel Products Co.
Degussa Corp.
Diamond Shamrock Corp., Proc. Chems.
Diano Corp.
D/L Laboratories
Dow Chemical USA
Draiswerke, Inc.

Eastman Chemical Products, Inc.
Elcometer Instruments Ltd.
Encapsulair, Inc.
Engelhard Minerals & Chems. Div.
Epworth Manufacturing Co., Inc.

Fawcett Co., Inc.
Federated Metals Corp., Sub ASARCO
Filter Specialists

Gail Industries
Gardner Laboratory, Div. Pacific Sci.
General Electric Co. Silicones
W.R. Grace & Co., Davison Chem. Div.
Graco, Inc.

Halox Pigments
Harshaw Chemical Co.
Henkel Corp.
Hercules Incorporated
Hilton-Davis Chemical Co. Div.
Hockmeyer Equipment Corp.
J.M. Huber Corp.
Hunter Associates Lab., Inc.

ICI America, Inc.
International Mins. & Chems. Corp.
Interstab Chemicals, Inc.

Jaygo, Inc.
Johns-Manville

Kenrich Petrochemicals, Inc.

Labelette Co.
Liquid Controls Corp.

MacBeth Div., Kollmorgen Corp.
Manchem Limited

1980 PAINT SHOW EXHIBITORS (cont'd)

Meadowbrook Corp.
Merck & Co., Chemical Div.
Miller Paint Equipment, Inc.
MiniFibers, Inc.
Modern Paint and Coatings
Morehouse Industries, Inc.
Myers Engineering, Inc.
MGI International, Inc.

Netzsch Brothers, Inc.
Neville Chemical Co.
NL Chemicals, NL Industries
NYCO Div., Processed Minerals, Inc.

O'Brien Industrial Equipment Co.
Omya, Inc.

Penn Color, Inc.
Pennsylvania Glass Sand Corp.
Pfaudler Co., Div. Sybron Corp.
Pfizer, Inc., MPM Div.
Photomarker Corp.
Polychrome Corp.
Polyvinyl Chemical Industries
PPG Industries, Inc.
Premier Mill Corp.
PVO International, Inc.

Q-Panel Co.

Reichard-Coulston, Inc.

Reichhold Chemicals, Inc.
Rohm and Haas Co.

Shamrock Chemicals Corp.
Shell Chemical Co.
Silberline Manufacturing Co., Inc.
Solem Industries, Inc.
Spencer Kellogg Textron
Sun Chemical Corp.
Sweco, Inc.

Tenneco Chemicals, Inc.
Thiele Engineering Co.
Tokheim Corp.
Troy Chemical Corp.

Union Camp Corp.
Union Carbide Corp.
Union Chemicals Div., Union Oil Co.
Union Process, Inc.
United Catalysts, Inc.
Universal Color Dispersions

R.T. Vanderbilt Co., Inc.
Viking Pump Div., Houdaille Ind.
Vorti-Siv Div., M&M Machine

Warren Rupp Co.
Wilden Pump & Engineering Co.

Zorelco Limited

As this issue of the Newsletter went to press, these additional companies signed up for the Show: Cabot Corp.....Dresser Minerals.....Ebonex Corp.....Hooker Chemical Corp.....Semi-Bulk Systems.....Thibaut-Walker Co.....Wacker Chemical Co.

Running concurrently with the Paint Show at the Civic Center will be the Program Sessions, the theme of which will be "Three R's for the Eighties: Research, Resources, and Regulations."

The theme recognizes that the coming decade looms as a time of stress and challenge. To remain an economically sound producer of increasingly valuable products, the coatings industry must utilize research and ingenuity to cope with the constraints of limited resources and restrictive regulations.

Prospective speakers are invited to present original papers on the many and varied aspects of the theme, and are requested to submit abstracts for review to Hugh W. Lowrey, FSCT Program Chairman, c/o Indurall Coatings, Inc., P.O. Box 2371, Birmingham, AL 35201. Deadline for receipt of abstracts is March 1.

ACTIVITIES OF TECHNICAL, EDUCATIONAL, AND MANUFACTURING COMMITTEES

Technical Advisory (Colin Penny, of Baltimore Society, Chairman)--Will sponsor a meeting of Society Technical Committee Chairmen on March 27-28 in Atlanta. To be discussed will be these suggestions for Society Technical Committee projects: Corrosion Control, Washability/Scrubability Standards, and a Computer Program for Paint Formulation. Regarding the latter, it is hoped that arrangements can be made for a presentation (with appropriate hardware) by a computer expert on the potential and feasibility for developing this project.

Educational (John A. Gordon, Jr., of St. Louis Society, Chairman)--Will sponsor a meeting of Society Educational Committee Chairmen on April 16 in Louisville. Current and future programs of the Societies and the Federation will be discussed. A progress report will be given on the Correspondence Course being prepared for the Federation by the University of Southern Mississippi.

Manufacturing (Donald J. Fritz, of Philadelphia Society, Chairman)--Held a meeting of its Steering Committee on January 23. Society Manufacturing Committee Chairmen will be sent the minutes of that meeting.

NEWS BRIEFS FROM THE SOCIETIES

BALTIMORE--Will hold second "Baltimore Coatings Show" on March 28.

BIRMINGHAM--Will sponsor symposium on "Effect of Current Legislation in the U.K. Paint Industry" on May 15.....Celebrating its 50th Anniversary year.

CHICAGO--Has nominated Carroll M. Scholle (Federation President in 1965-66) for Federation Honorary Membership.....SYMCO '80 will be on "Additives." Scheduled for March 25-26 in Des Plaines.

CDIC--Elected Herbert L. Fenburr and Harry Poth to Society Honorary Membership. The November meeting featured a plant tour of the General Motors Inland Div. in Dayton.

DALLAS--Presented seminar on "Formulation of Interior Flat Latex Paints," January 10.

DETROIT--Initiated the sponsorship of a seven-week lab course at the Polymer Institute, University of Detroit, with Michael J. King as Instructor.....Established an Outstanding Member Award.

LOS ANGELES--Trev Whittington is now conducting two courses at Los Angeles Trade Technical School: Paint Technical Preparation and Paint Technology Lab.

MONTREAL--Art Hagopian to chair special committee to mark 50th Anniversary in 1981... G. Caya and R. Comeau received 25-year pins.....In keeping with biennial exchange of visits, some members planning to attend February 21 meeting of New England Society.....1980 joint symposium with Toronto Society will be on "Coatings for Metal". Hy Kredentser (retired) elected Society Honorary Member.....Technical Committee has prepared first draft of "Freeze-Thaw Stability of Latex Paints".....W. Kolanitch and C. Craib have organized 15-week course in Coatings Technology. Will be given at the Sherwin-Williams Technical Centre.

NEW ENGLAND--Sponsored a seminar on "Pigment Dispersion" on January 17. Coming up is another on "Hazardous Waste," February 21.....Has announced the first annual "Coatings & Ink Expo," May 13 and 14 in Boxborough.

PACIFIC NORTHWEST--Beginning with 1980-81 year, will present \$500 scholarship (in name of Past-President George Melvin) to a student in the Dept. of Polymer Chemistry at University of British Columbia.....25-year pins presented to Doug Dicker and Larry Story.

PHILADELPHIA--Marked 60th Anniversary at December 13 meeting.

SOUTHERN--Theme of annual meeting, March 12-14, in Atlanta, will be "Personal Growth in Coatings Technology." To feature motivational talks plus panel discussions on production and purchasing topics. Meeting opens Wednesday with a seminar on Coatings Fundamentals, conducted by the University of Southern Mississippi

TORONTO--In cooperation with George Brown College, will present three courses in Coatings Technology, beginning January: Polyurethanes, with guest speakers; Acrylics, with C. Skelton as Instructor; and Emulsions, with S. Kambanis as Instructor. Also a lab course.

COMING EVENTS

- Feb. 17.....New England Society. Seminar on "Hazardous Waste." Cambridge, Mass.
Mar. 10-12...Southern Society. Water-Borne and Higher-Solids Coatings Symposium. Hyatt Regency. New Orleans.
Mar. 12-14...Southern Society. Annual meeting. Terrace Garden Inn, Atlanta.
Mar. 25-26...Cleveland Society. Conference on Advances in Coatings Technology. Baldwin-Wallace College, Berea, Ohio.
Mar. 25-26...Chicago Society and PCA. SYMCO '80. Fountain Blue, Des Plaines.
Mar. 28.....Baltimore Society. Coatings Show. Baltimore.
Apr. 14-15...Louisville Society. Symposium on "Compliance With Government Regulations" and "Hazardous Materials Waste Disposal." Marriott Inn, Clarksville, Ind.
Apr. 17-19...Southwestern Paint Convention of Dallas and Houston Societies. Hilton Inn, Dallas.
May 1.....Detroit Society. FOCUS Seminar on "Environmental Challenges to Automotive Coatings." Michigan State Conference Center, Troy.
May 1-3.....Pacific Northwest Society. Annual symposium. Hilton Hotel, Portland.
May 13-14...New England Society. "Coatings & Ink Expo." Boxborough.
May 15.....Birmingham Club. Symposium on "Effect of Current Legislation on the U.K. Paint Industry." Birmingham, England.
May 15-17...Federation Spring Meetings. Society Officers--15th; Executive Committee--16th; Board of Directors--16th and 17th. North Star Inn, Minneapolis, Minn.
June 6-7....Joint meeting of Kansas City and St. Louis Societies. Hilton Plaza Hotel, Kansas City.
Oct. 29-31...Annual Meeting and Paint Industries' Show of Federation. Atlanta Civic Center, Atlanta.

FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

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You could call Min-U-Sil a lazy crystalline silica paint filler. Because it's inert.

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burnish resistance, scrubability, and even improves sanding characteristics.

But unlike other silica fillers that only come in 30 and 15 micron grades, Min-U-Sil is available in the finer 10 and 5 micron sizes. Which gives your paint a smoother, more even film, whether applied with roller or brush.

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To show you how well Min-U-Sil works, we'll send you a free sample. Plus some impartial test results. Just write to Pennsylvania Glass Sand, Dept. A-24, Three Penn Center, Pittsburgh PA 15235. Or call (412) 234-7500.

Then test Min-U-Sil for yourself. In your paint. You'll find Min-U-Sil is the lazy way to make your paint work harder. And make profits come easier.

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The same boys and the same paint job are shown at right . . . 5 years later. The boys changed, but the paints maintain a fresh, just-painted appearance.

That's 100%-acrylic performance! . . . the kind that satisfies your customers and keeps them coming back. The kind you get from:

RHOPLEX AC-507 - For trim and house paints. Excellent gloss and gloss retention.

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RHOPLEX AC-388 - Multipurpose vehicle.

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5 years
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sheen housepaints

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“What it doesn’t do may be as important as what it does do.”

When we first developed it, we knew Nuosept 95 bactericide was an excellent preservative for water-based paints. We knew it was completely compatible in many paint formulations, and we knew it had a very low order of toxicity. Our experience told us that its liquid form made for easy handling, and its cost profile was attractive.

But, what we didn’t predict, was Nuosept 95 bactericide’s immediate acceptance by major paint producers because it *doesn’t cause yellowing* of paint while it sits on the dealer’s shelf. This is a critical factor if your paint is to match its color chip, regardless if it’s factory tinted or custom colored.

So, what it doesn’t do is the reason Nuosept 95 bactericide is so widely accepted.

If you haven’t evaluated our Nuosept 95 bactericide, you should. Our Biocontrol Laboratory will be pleased to assist in comparative evaluations against your present preservative or other preservatives you wish to test. Contact our local sales office, or write to me, Bill Stewart, at Tenneco Chemicals, Inc., Coatings & Colorants, P.O. Box 365, Piscataway, N.J. 08854.



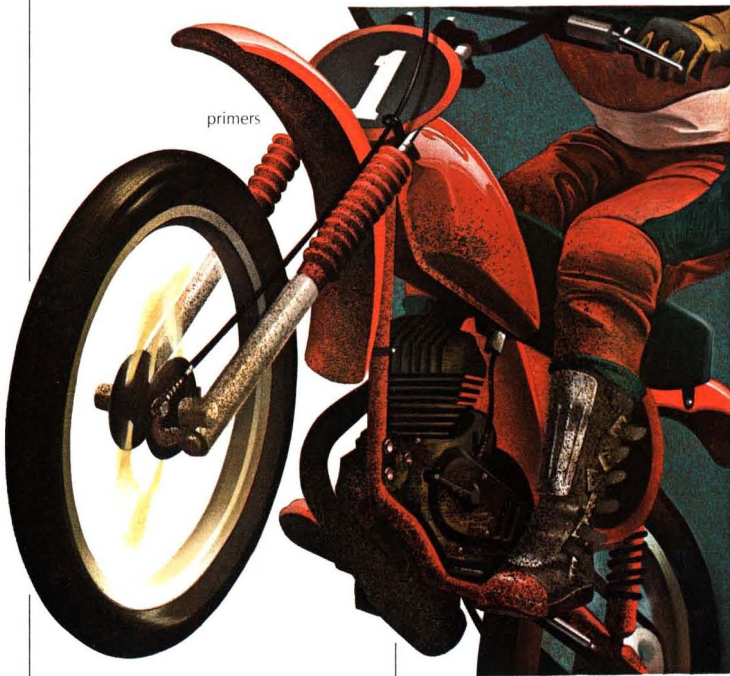
-Bill Stewart

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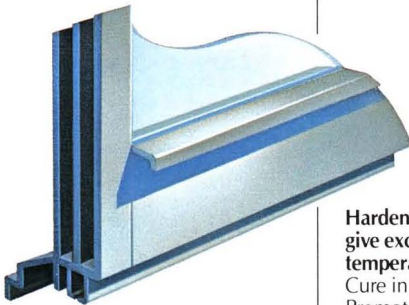
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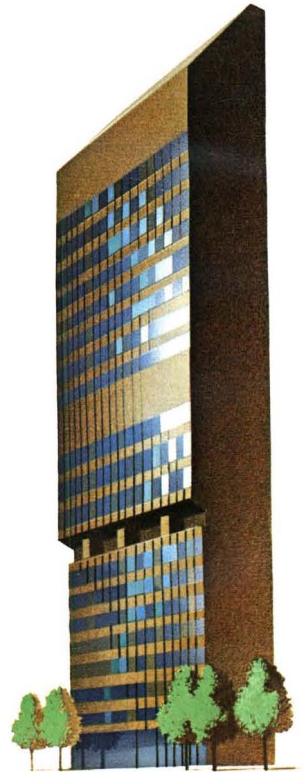


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Hardeners HT 939 and HY 940 give excellent latency at room temperature
Cure in five minutes at 100°C. Promote outstanding adhesion. Allow non-critical mixing ratios.

These special Ciba-Geigy epoxy resins have been used for years in the electronics and electrical industries. Their outstanding properties make them ideal for new high-performance coatings as well as for upgrading existing coatings systems. We have recently built new, larger manufacturing facilities, and these special epoxies are now available to the coatings industry. We believe the suggested applications illustrated are only a few of many possible uses.

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FOR A NEW GENERATION OF

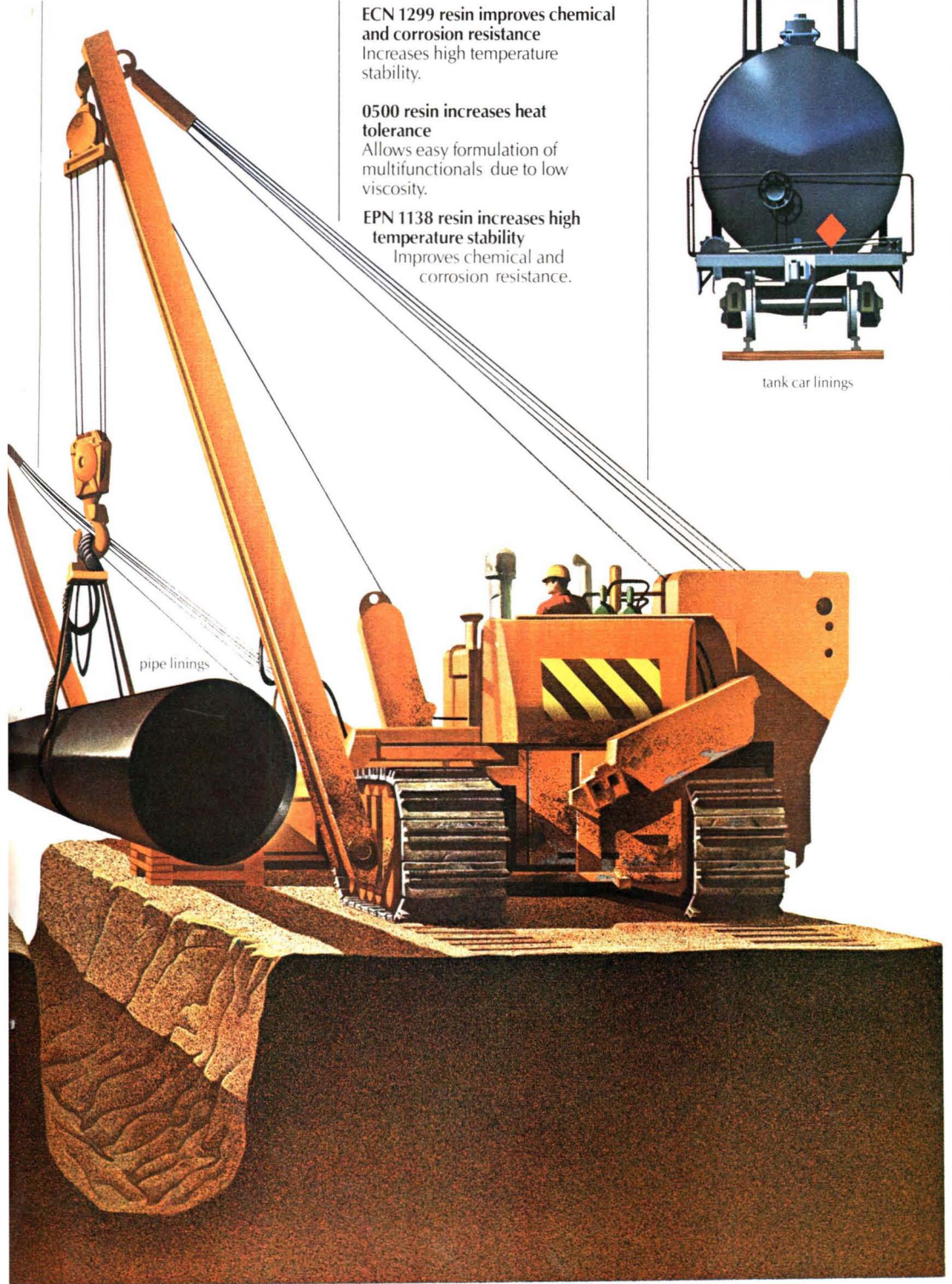
ECN 1299 resin improves chemical and corrosion resistance
Increases high temperature stability.

0500 resin increases heat tolerance
Allows easy formulation of multifunctionals due to low viscosity.

EPN 1138 resin increases high temperature stability
Improves chemical and corrosion resistance.



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pipe linings

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Typical Product Family Profiles:

	Form	Epoxy Functionality	Viscosity at 25°C/ melting point	Solution Viscosity*	Additional Features
0500	Liquid	3	3,000 cP	200 cP	high heat resistance
ECN's	Solid	2.7-5.4	35-99°C	3,000 cP	tight crosslink density, chemical resistance, hardness
EPN's	Liquid/Semi – solid	2.2-3.6	1700-3500 cP at 52°C	3,000 cP	FDA-conforms to 175.300
Aracast® hydantoins	Liquid/Solid	2	500 cP/95°C	–	non-aromatic, weatherable non-crystallizing, very low viscosity
Araldite® PT 810	Solid	3	95°C	–	non-aromatic, weatherable
HT 939	Solid	–	105°C	–	latent hardener/rapid cure
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*80% solids in Cellosolve acetate

Suggested Applications:

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ECN's	■	■	■		■	■
EPN's	■		■		■	■
Aracast® hydantoins	■	■	■	■	■	■
Araldite® PT 810		■				■
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MMA Awards for Notable Achievements Won by New York and Golden Gate Societies

The 1979 MMA Awards for notable achievements of Constituent Societies of the Federation were won by the New York and Golden Gate Societies. Presentation was made at the recent Federation Annual Meeting in St. Louis.

The awards established in 1975 by Materials Marketing Associates, a national marketing group of manufacturers' representatives, recognize notable achievements by Constituent Societies, excluding those Society papers presented at the Federation Annual Meeting.

Each winner receives \$350 in cash plus a handsome plaque.

Excellence of Educational Program

The New York Society was cited for its educational efforts to serve the various needs of its membership, through a three-fold program: to introduce college students to the industry through an active scholarship program; to increase industry exposure by making more colleges aware of the coatings industry; and to develop information sessions for industry personnel through seminars and various college-level courses.

Four scholarships have been established in memory of individuals who were active in the coatings industry and who were associated with the New York Society. Each is available at a local university and is given to qualified Chemistry or Chemical Engineering students in need of financial assistance, with preference given to students whose parents are affiliated with the chemical coatings industry and/or who have shown a desire to major in coatings technology.

Many institutions in the New York metropolitan area have been made aware of the scholarship program, and as a result these institutions are expressing interest in participating, with a concurrent increased interest in the coatings industry and its potential for career opportunities.

The Society's continuing education program includes a two-semester course in Fundamentals of Coatings Technology at New York City Community College (jointly sponsored by the New York Paint and Coatings Association), along with a lab course for paint technicians which is conducted at various industry-oriented coating laboratories.

A seminar was sponsored in conjunction with the Philadelphia Society, to which provided a significant opportunity for the membership of both groups to



R. L. Ridolfi, President of Materials Marketing Associates congratulates representatives of winning Societies in the 1979 MMA Awards competition. Shown with plaques are A. Gordon Rook (Golden Gate Society) and S. Leonard Davidson (New York Society), while W. T. Cochran, Chairman of MMA Awards Committee (second from left), and John Paul Taylor, MMA Executive Director, look on. Presentation was made at the Federation Annual Meeting in St. Louis.

conveniently attend to take part in discussions on current topics of interest in the coatings industry.

Industry Contributions Through WCCS Symposium and Show

The Golden Gate Society won its Award for sponsoring the Western Coatings Societies' Symposium and Show, February 28-March 2, 1979, in San Francisco.

The event provided an opportunity for industry personnel in the western area to learn of new technologies, raw materials, and equipment, through the technical program and the displays of supplier firms.

The three-day event featured the presentation of 23 technical papers and the exhibits of companies in 67 booths.

Funds raised by the event support the activities of the Educational, Technical, Manufacturing, and Specification Committees, as well as the Study Grant program for members.

Principles Governing Awards

The MMA Awards recognize notable achievements in the field of education, manufacturing and training procedures, technology, public service, and other achievements deemed proper and desirable by the Awards Committee.

Not eligible are Society papers offered for presentation at the Federation Annual Meeting. Although the Awards are to be presented at the Annual Meeting, it is not mandatory that they be presented to any or all categories each year.

The President of any Society wishing to enter the competition must send a letter of intent, no later than March 31, to the MMA Awards Committee Chairman, William T. Cochran, Bruning Paint Co., Inc., 601 S. Haven St., Baltimore, Md. 21224. A complete description of the Society activity to be considered in the competition must be submitted by the Society President to Chairman Cochran by August 31.

Materials Marketing Associates is composed of the following: Apco Industries Co., Ltd., Toronto, Ont.; George C. Brandt, Inc., Kansas City, Kan. and St. Paul, Minn.; The Cary Co., Chicago, Ill.; Dar-Tech, Inc., Cleveland, Ohio; E. T. Horn Co., Los Angeles, Calif.; Lukens Chemical Co., Boston, Mass.; Matteson-Ridolfi, Inc., Detroit, Mich.; McCullough & Benton, Inc., Atlanta, Ga.; Ribelin Distributors, Inc., Dallas, Texas; Wm. B. Tabler Co., Inc., Louisville, Ky.; Van Horn, Metz & Co., Inc., Philadelphia, Pa.; Walsh & Associates, Inc., St. Louis, Mo.; and C. Withington Co., Pelham Manor, N.Y. and Rio Piedras, Puerto Rico.

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Battelle: 1980 R&D Funding in U.S. to Reach \$61.8 Billion

Expenditures in calendar year 1980 for research and development in the United States are expected to reach \$61.8 billion, according to the annual forecast of Battelle's Columbus Laboratories. This represents an increase of \$10.2 billion (19.7%) over the \$51.6 billion that the National Science Foundation estimates was to be actually spent for R&D in 1979.

While most of the increase will be absorbed by continued inflation, (estimated at 12.1% from 1979 to 1980), Battelle forecasts about a 7.0% real increase in R&D expenditures, a new peak in total real funding of R&D. In the event of a severe 1980 recession, however, this estimated increase may prove slightly optimistic.

The report notes that if, indeed, a severe recession is in the offing, it will have have offsetting effects on R&D. While industrial commitments may decrease in the short term, federal support may increase in order to

maintain stability in the R&D "system" and to guard against a serious decrease in the nation's capacity.

In 1980, Battelle sees an increase of about 19.0% in federal support for R&D, with funding expected to be about \$30.6 billion. This is 49.5% of the total R&D expenditures for 1980. Industrial funding is forecast to be \$29.1 billion, up 20.9% from 1979. This sector is expected to account for 47.0% of the total R&D funding. Funding by academic institutions is expected to be \$1.3 billion (2.1% of the total), while nonprofit organizations will provide \$850 million (1.4%).

The forecast notes that national security, reflected in the Department of Defense budget, is a principal driving force in furthering R&D spending. Economic growth, founded upon both basic and applied research, is expected to remain as an objective that requires direct and indirect federal support. The continuing emphasis on energy, and the

associated problems of balance of payments and insecure resources, will result in increased efforts on the research, development, demonstration, evaluation, and diffusion of new energy technologies.

Additionally, it is anticipated that R&D moneys will continue to pour into the biological and "soft" science areas; into educational research directed toward support of expanding technical areas; into "impact" research that covers environmental, ecological, and socio-economic consequences of technological or related actions; and into research on methods of compliance with regulations.

Industrial support of research is growing in fields affected by regulations and in those fields most directly influenced by the need for more energy-efficient products and processes. Almost all industrially funded R&D will be absorbed by industry, with only 2.0% to be done by other sectors. R&D will be heavily self-funded in the manufacturing industries where, on the average, only 34.9% of the total will be supported by the federal government. The non-manufacturing industries do relatively little R&D and support for this activity will be divided almost equally between federal and industrial funding.

Since 1973, real R&D effort has increased at a six-year average rate of almost 2.7% per year, and the projections for 1980 suggest that the seven-year average rate will increase to about 3.3%. Battelle estimates that during the decade of the 1980s real R&D activity will increase at an average annual rate of approximately 3.0%.

Researchers Seek Information on Performance Of Coatings on Highway Structural Steel

Information on computer assisted record keeping and analysis of performance data for coatings on steel is sought by the Engineering Experiment Station of Georgia Institute of Technology on behalf of the Federal Highway Administration (FHWA) of the U.S. Department of Transportation.

The FHWA, a major source of information for all 50 state highway departments, is concerned with the kinds of coatings available for highway structural steel and with improved methods for evaluating their performance. One aspect of its research program on coating systems for controlling corrosion involves the use of performance data generated in tests conducted by states, various Federal agencies, private industry, and other organizations. Such information, especially that derived from controlled service testing, is

expected to be helpful in establishing correlations and developing improved performance standards and actual improved long range costs. The volume of the data expected will require the use of automated data processing.

Information is sought as to highway related and non-highway related sources of coatings performance data on steel in addition to information about computerized evaluations of coatings performance data per se. Confidentiality of any proprietary information will be strictly observed.

Anyone wishing to supply information on this subject and to obtain copies of the reports resulting from this study should contact: Dr. Charles Ray, Engineering Experiment Station, Georgia Institute of Technology, Atlanta, Ga. 30332 (telephone (404) 424-9651).

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Performance of Selected Marine Coatings Exposed to High Velocity Seawater

George A. Gehring, Jr.
Ocean City Research Corporation*

A study was undertaken to investigate seawater velocity thresholds and failure modes for three selected marine coatings under non-cavitating, parallel flow, ranging from 3 to 30 m/s. The study was accomplished in a flow channel using natural seawater. The results indicate that: (1) the velocity threshold for two of the three coatings investigated might exceed 30 m/s; (2) blister formation appears to be the critical part of the damage process leading to gross failure; (3) under the given set of experimental conditions, hydromechanical wear is negligible; (4) electrical impedance measurements enable non-destructive detection of impending coating failure; and (5) parallel flow is not as damaging as impinging or cavitating flow in the velocity range between 15 and 30 m/s.

INTRODUCTION

In recent years, there has been considerable effort directed toward the development of higher speed ships (e.g., hydrofoil, surface effect ships). High velocity seawater can cause rapid deterioration of many structural materials. For many materials, the relative stability in high velocity seawater needs to be better documented. One class of materials deserving detailed study is anti-corrosive coatings that could be used to provide corrosion protection for structural alloys exposed to high velocity seawater.

Previously, most of the pertinent high velocity coating studies¹⁻⁷ have been conducted under cavitation or impingement-type flow. Under the flow conditions established in these prior studies, conventional marine coatings (sprayable, solvent-type coatings) failed quite rapidly (often within 1 to 10 hr). Elastomeric coatings, of types not widely used in the marine industry, generally exhibited better performance. However, many of these better performing, elastomeric coatings involve rather sophisticated application techniques compared to the more conventional marine coatings. Some of the elas-

tomeric coatings might pose formidable application problems in shipyards.

While it is recognized that a high speed ship will be subjected to cavitation and impingement-type flow, it would seem that the major portion of its exposed surface will see simple, parallel, non-cavitating* flow. The conventional marine coatings might exhibit better stability at the higher velocities under parallel, non-cavitating flow conditions than under cavitating flow conditions. There appears to be a dearth of data in the literature on the performance and stability of conventional marine coatings in high velocity, non-cavitating seawater. Consequently, this study was undertaken to investigate seawater velocity thresholds and failure modes for three marine coatings under non-cavitating flow conditions ranging from 3 to 30 m/s.

EXPERIMENTAL

Background

The laboratory apparatus used primarily in previous velocity studies has included a rotating disk, water tunnel, waterwheel, and impinging jet. Each of these apparatuses was disadvantageous for high velocity seawater coating studies for one or more of the following reasons:

- (1) The size of the test specimen imposed by the apparatus was unreasonably small resulting in an artificially high edge-to-surface area ratio. Coating failures often initiate at sharp edges where complete, uniform coverage is difficult to attain. Also, fully developed uniform flow is difficult to attain with small-size specimens and the attendant boundary effects.
- (2) The nature of the high velocity flow was often complex, not easily definable or relatable to the actual service environment.
- (3) The apparatus used recirculated synthetic seawater. This prevented long term exposures because of

*It is realized that at sufficiently high velocities, small surface protrusions present either initially or those that develop during actual service due to blistering or the like can cause localized cavitation or be subject to impingement which might possibly lead to premature failure.

*Tennessee Ave. and Beach Thorofare, Ocean City, N.J. 08226.

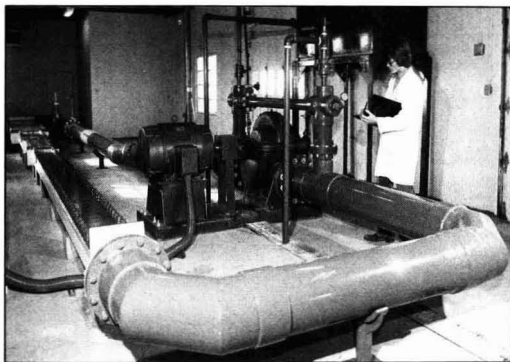


Figure 1—High velocity flow channel

problems associated with temperature control, water stagnation, and/or concentration effects.

(4) The apparatus did not permit the development separately of different types of flow (cavitating, impinging, parallel) depending on the objectives of the study.

(5) The apparatus was velocity-limited considering the present day anticipated velocities of high speed naval ships.

(6) The apparatus was limited in the number of materials that could be studied simultaneously.

(7) A comprehensive study over a broad velocity range was impractical because of high setup and teardown costs or untenable time requirements.

A flow channel available at the OCRC laboratory overcomes all of the above-listed disadvantages. The flow channel permits exposure of significantly larger test panels than heretofore possible with other high velocity test apparatus. Consequently, edge and boundary effects are minimized. The panels can be exposed to simple, parallel flow.

Flow Channel Description

Figure 1 shows the OCRC flow channel. The width of the channel cross-section varies along the length to

permit testing at six different flow velocities simultaneously (maximum velocity ≈ 18 m/s). A separate, constant-width section permits testing at higher velocities (maximum velocity ≈ 30 m/s). Figure 2 presents a simplified schematic of the channel. For the present study, the nominal test velocities were 30, 18, 15, 12, 9, 6, and 3 m/s, respectively.

Figure 3 shows the method by which test panels were mounted in the low velocity section (3–18 m/s) of the channel. Each velocity subsection accommodated five test panels ($17.5 \times 25.5 \times \approx 1.3$ cm thick). The test panels were spaced 5 cm apart using phenolic spacers to maintain a continuous center wall in each section. The interface between spacer and panel was matched as precisely as possible to avoid edge effects. Electrical leads were attached to each test panel to permit electrical impedance measurements.

In the high velocity section (30 m/s), the test panels were fitted into slots which had been precisely machined in the acrylic sidewalls. The test panels were carefully shimmed to minimize edge mismatch and eliminate cavitation. As in the low velocity section, provisions were made for external electrical connection to the panels to permit the acquisition of electrical impedance data.

Natural seawater is circulated through the channel by a double-suction centrifugal pump powered by a 100 hp motor. The flow rate can exceed 300 l/s and is measured using a factory-calibrated 316 SS orifice plate/differential pressure gauge set-up. The rate of seawater make-up into the channel is adjusted to control seawater temperature within $\pm 2.5^\circ\text{C}$ while being maintained sufficiently high to avoid stagnation or concentration effects. For the present study, the make-up rate varied between 2–5 l/s.

The channel was designed to provide high Reynolds number, Re , flow at each test velocity ($Re \approx 10^6$). This was done in order to better simulate flow conditions that might be encountered during high speed ship operation.

Coatings Selected For Study

The following three coating systems, believed to be representative of the marine coatings presently being used, were selected for study:

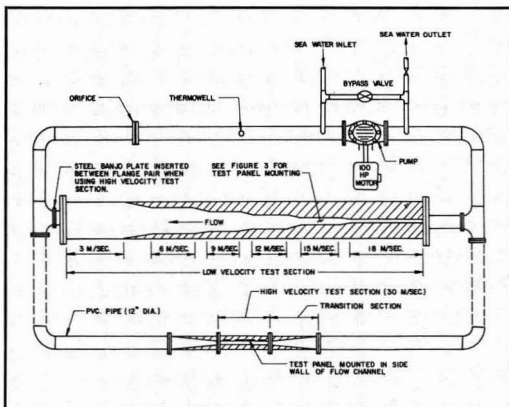


Figure 2—Schematic of flow channel

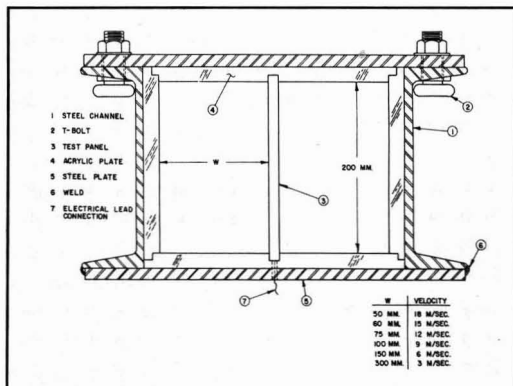


Figure 3—Cross-section view of test panel mounted in flow channel (low velocity section)

(1) Polyamide Epoxy Primer (MIL-P-24441, F150) - 75 to 100 microns nominal dry film thickness;
 Polyamide Epoxy Mid-Coat (MIL-P-24441, F151) - 50 to 75 microns nominal dry film thickness; and
 Polyamide Epoxy Topcoat (MIL-P-24441, F156) - 50 to 75 microns nominal dry film thickness.

(2) Polyamide Epoxy Primer (Devran® 201) - 50 to 75 microns nominal dry film thickness; and
 Modified Polyamide Epoxy Topcoat (Devran® 230) - 200 microns nominal dry film thickness.

(3) Coal Tar-Epoxy, Polyamide Cured (Tarsset® C-200) - two coats, each at 200 microns nominal dry film thickness.

The first coating system, MIL-P-24441 Epoxy, is used throughout the Navy as an anti-corrosive coating on both surface ships and submarines. It is a two-part epoxy coating with ≈ 56% solids by volume. It is usually applied in three coats to obtain a total dry film thickness of ≈ 200 microns (≈ 8 mils).

The second coating system consists of the Devran 201 Polyamide Epoxy primer and Devran 230 Modified Polyamide Epoxy topcoat. The 201 primer meets the MIL-P-24441 specification. The 230 topcoat is a hydrophobic hydrocarbon modified polyamide epoxy. The 230 topcoat has a volume solids of 65% and is designed to achieve a dry film thickness of ≈ 200 microns (≈ 8 mils) in a single coat.

The third coating system selected for the study was Tarsset C-200 Coal Tar-Epoxy. The two component system is polyamide cured and has a volume solids of ≈ 76%. It is designed to achieve a high build in a single coat, normally ≈ 200 microns (≈ 8 mils).

The coatings were applied by air spray to ASTM A-36 steel panels (17.5 × 25.5 × 1.3 cm) grit blasted to white

Devran is a registered trademark of Devco and Raynolds Co. Tarsset is a registered trademark of Porter Paint Co.

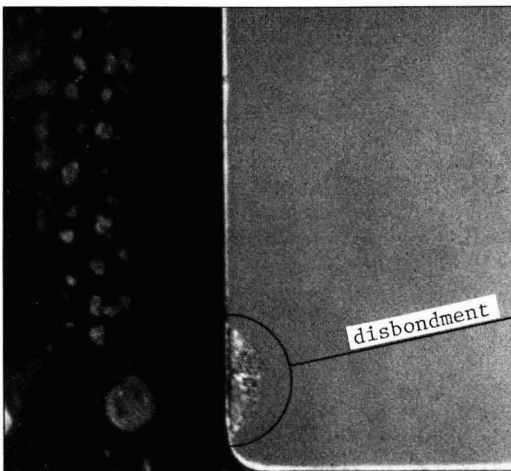


Figure 4—Coating disbondment observed at 181 hr at 30 m/s (Panel 1; MIL-P-24441 epoxy)

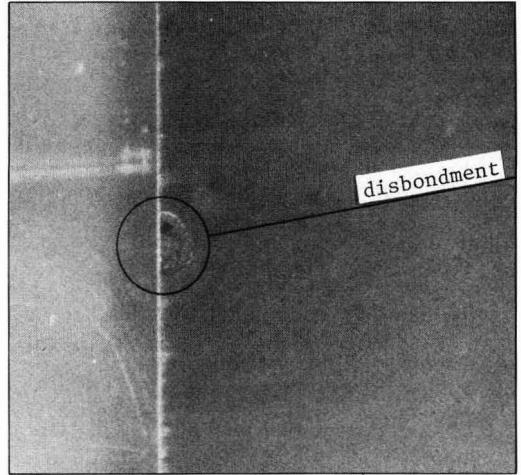


Figure 5—Coating disbondment observed at 349 hr at 30 m/s (Panel 1; MIL-P-24441 epoxy)

metal. The test panel exposure sequence as a function of velocity was as follows:

Velocity	Coating (No. of Panels)
30 m/s	MIL-P-24441 (2), Devran (1), Tarsset (1)
18 m/s	MIL-P-24441 (2), Devran (2), Tarsset (1)
15 m/s	MIL-P-24441 (2), Devran (2), Tarsset (1)
12 m/s	MIL-P-24441 (2), Devran (2), Tarsset (1)
9 m/s	MIL-P-24441 (2), Devran (2), Tarsset (1)
6 m/s	MIL-P-24441 (2), Devran (2), Tarsset (1)
3 m/s	MIL-P-24441 (2), Devran (2), Tarsset (1)

Time of Exposure

The coated panels were simultaneously exposed to uninterrupted flow for 812 hr at each velocity from 3–18 m/s (low velocity section of channel). In the high velocity section, at 30 m/s, the coated panels were exposed for 898 hr.

Data Acquisition

Data acquisition during the study included weight loss measurements (sensitivity = ± 1 g), dry film thickness measurements using a Mikrotest gauge (prior to and after exposure - sensitivity = ± 25 microns), and electrical impedance measurements.

Electrical impedance (resistance and capacitance) was measured in situ between the coated panels and platinum reference electrodes located in the sidewalls of the channel. The measurements were made at different times during each velocity exposure using a General Radio Type 1650 Impedance Bridge at 3,000 Hz. Changes in the electrical resistance and capacitance of high dielectric coatings exposed in low resistivity electrolytes generally correlate to changes in the protective nature of the coating as shown by many investigators.⁸⁻¹¹

An increase in electrical capacitance corresponds to a reduction in effective coating thickness due to water penetration as seen from the following equation:

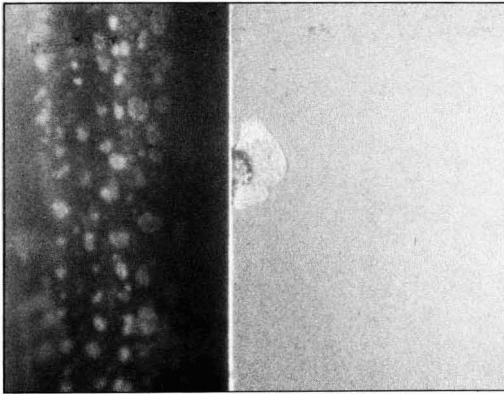


Figure 6—Coating disbondment observed at 463 hr at 30 m/s (Panel 1; MIL-P-24441 epoxy)

$$K = 11.3 C t A$$

where,

- K = dielectric constant of the coated panel
- C = capacitance
- A = exposed surface area
- t = effective coating thickness

Similarly, a decrease in electrical resistance signifies physical breakdown of the coating allowing water to short circuit the high dielectric barrier. Electrical impedance measurements are useful because they often allow early detection of significant changes in coating performance not obvious by visual inspection. Also, they are non-destructive.

RESULTS

Coated panels of each of the selected coating systems were exposed at 30 m/s for 898 hr and at 18, 15, 12, 9, 6, and 3 m/s for 812 hr.

Performance of Panels Exposed at 30 M/S for 898 Hours

(1) POLYAMIDE-EPOXY (MIL-P-24441): Two panels prepared with the MIL-P-24441 coating were tested at 30 m/s. The first sign of coating deterioration was observed on Panel 1 (upstream panel) after 181 hr in test (Figure 4). Coating disbonded over about a 2 cm² area on the leading edge. This coating holiday remained approximately the same size over the remainder of the exposure. Corrosion of the exposed steel substrate occurred quite rapidly and at the end of the exposure a pit depth of 0.300 mm was measured. The apparent stability of the coating around the initial holiday was surprising. It had been anticipated that, in the event of such a failure at this velocity, coating disbondment would proceed rapidly from this point.

The next observed failure occurred on the same panel after 349 hr. Again, the failure was in the form of disbondment initiating at the leading edge (Figure 5). The holiday remained stable in size over the remainder of the test period.

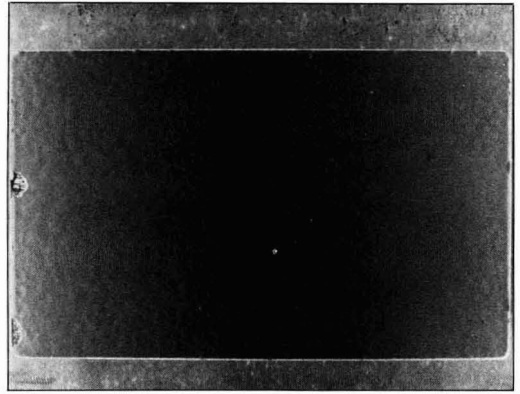


Figure 7—MIL-P-24441 epoxy after 898 hr exposure to seawater flowing at 30 m/s (Panel 1)

Similar coating failure occurred on Panel 2 (downstream panel) after 463 and 510 hr, as shown in Figure 6. These holidays also were stable over the remainder of the exposure period. Figures 7 and 8 are photographs of each test panel upon completion of the 898-hr exposure test. Upon completion of the tests, small blisters were detected at random locations along the edges of both panels.

(2) MODIFIED POLYAMIDE EPOXY (DEVTRAN 201/DEVTRAN 230): Figure 9 shows the deterioration observed on this coated panel after 898 hr exposure. Failure of this coating system first occurred at 54 hr. The failure was in the form of disbondment along the bottom edge (Figure 10). The holiday did not remain stable over the test period, but continued to increase in size (Figure 11). Significant corrosion occurred on the exposed steel substrate. The maximum pit depth was 0.517 mm after 898 hr.

At about 460 hr, disbondment of the coating was observed on the upper edge of the test panel. This holiday also continued to increase in size over the rest of the test period (Figure 12). Final inspection of the test panel showed random blistering along the edges of the test panel. The Devran coating system suffered significantly more damage than the other coatings at 30 m/s.

(3) COAL TAR-EPOXY, POLYAMIDE-CURED (TARSET C-200): The one panel coated with coal tar-epoxy did not exhibit detectable deterioration over the 898 hr velocity test (Figure 13). Final inspection revealed slight blistering at a few isolated spots along the bottom edge. The coated panel showed no significant weight loss or measurable reduction in thickness.

(4) ELECTRICAL IMPEDANCE MEASUREMENTS: The electrical capacitance and resistance measurements correlated excellently with observed coating behavior over the course of the test. Figures 14 and 15 present the data. Sharp changes in capacitance and resistance were measured at about the same time at which initial coating failures were observed for the MIL-P-24441 epoxy and Devran epoxy-coated panels.

For Panel 1, MIL-P-24441 epoxy, the measured capacitance at 174 hr was 60.3 nanofarads whereas prior

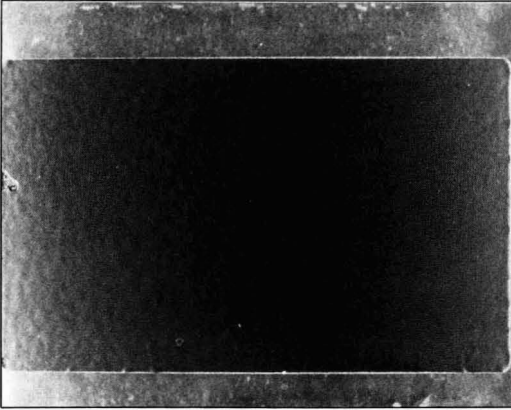


Figure 8—MIL-P-24441 epoxy after 898 hr exposure to seawater flowing at 30 m/s (Panel 2)

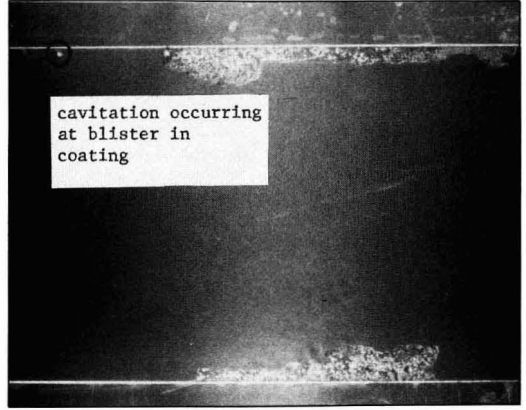


Figure 9—Devran epoxy at 898 hr exposure to seawater flowing at 30 m/s

to this time the capacitance had been fairly stable between 6–11 nanofarads. At the same time, the measured resistance decreased from 8500 to 2750 ohms. Failure on this panel was first observed visually at 181 hr.

The other MIL-P-24441 epoxy coated panel showed a significant shift in capacitance and resistance between 397 and 463 hr. Failure on the coated panel was first observed at 463 hr.

The Devran epoxy-coated panel exhibited a significant change in both capacitance and resistance between 39 and 54 hr. The first sign of failure on this panel was detected at 54 hr.

The capacitance and resistance data were fairly constant for the coal tar-epoxy coated panel over the entire test period. This corresponds favorably with the observed stable behavior of the coated panel.

Performance of Panels Exposed At 18, 15, 12, 9, 6, and 3 M/S for 812 Hours

(1) VISUAL OBSERVATIONS, WEIGHT LOSS AND DRY FILM THICKNESS MEASUREMENTS: Five coated panels (two MIL-P-24441, two Devran, one Tarsel) were exposed for 812 hr at each of the above-listed velocities. Leading edge failure in the form of disbondment was observed within the first hour after start-up on the first panel (Devran epoxy) exposed in the 15 m/s velocity section. This panel was exposed without the benefit of a spacer plate ahead of it. Thus, the leading edge was subjected to both cavitation and impingement-type flow. The first panel exposed in the 18 m/s velocity section was preceded by a spacer plate so that, essentially, it saw only parallel flow. The first panels in the other velocity sections were not preceded by a spacer plate, however, coating failure was not visually detectable over the duration of the test. Except for the above-noted panel and one other panel, none of the remaining 28 panels exhibited visually detectable coating damage over the 812 hr test period. The coating damage observed on the other panel represented a small chip in the coating at the bottom edge of the panel (Devran at 3 m/s) that most likely occurred during installation or removal of the

panel from the channel. Final inspection disclosed a slime film with some attendant algae growth on the panels exposed at 3 m/s.

Upon completion of the 812 hr exposure, the coated panels were allowed to air dry for five days, then weighed. Except for the panel which suffered leading edge failure, none of the remaining panels exhibited a measurable loss of weight (sensitivity = ± 1 g). Correspondingly, dry film thickness measurements failed to show a measurable decrease in the coating film thickness (sensitivity = ± 25 microns).

(2) ELECTRICAL IMPEDANCE MEASUREMENTS: Electrical capacitance and resistance measurements were made on all coated panels at different times during the velocity exposure. The capacitance values measured on each panel immediately after start-up ranged over four orders of magnitude, from a low of 16 nanofarads to a high of 42,280 nanofarads. The initial values of resistance varied from a low of 24 ohms to a high of 10,000 ohms. There was no observed trend for one coating to be

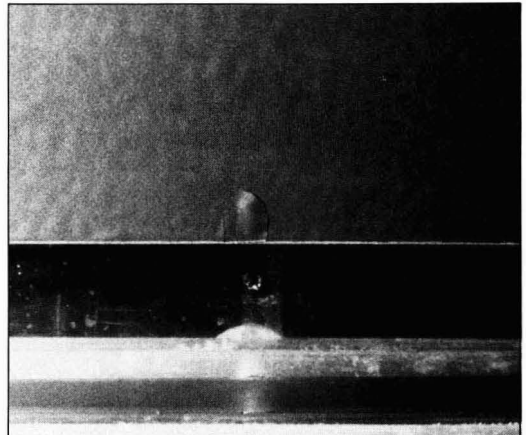
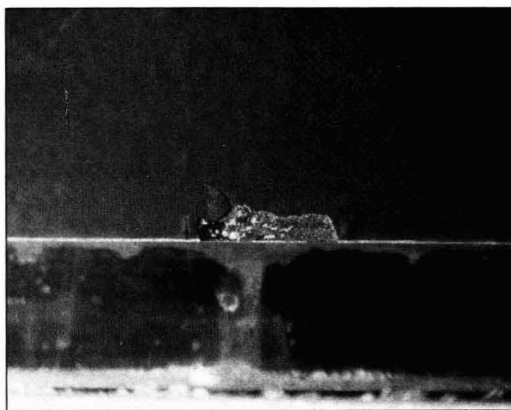


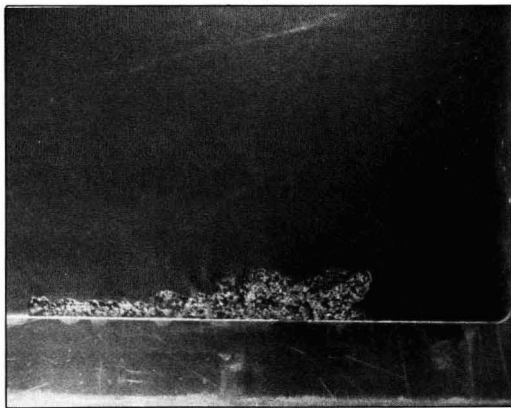
Figure 10—Coating disbondment observed at 54 hr at 30 m/s (Panel 1; Devran epoxy)

consistently higher or lower compared to the other two coatings.

The wide range of the initial impedance values was unexpected. Great care had been exercised in preparing the coated panels to insure that each panel was initially exposed in a holiday-free condition. After coating application, all panels had been inspected with a wet-sponge, low-voltage (67.5 volts) holiday detector. All holidays that were detected were repaired. All edges had been double-coated. After following these procedures and before inserting the panels in the channel, capacitance and resistance measurements were made on each coated panel when immersed in mercury. The values of capacitance and resistance measured in mercury were all within the same order of magnitude. It is the opinion of the author that, with one exception, the wider range of capacitance and resistance values measured immediately after start-up reflects differences in the degree of damage that occurred inadvertently on coated panel edges and/or the electrical lead wire insulation during insertion and positioning of the panels in the channel. The positioning of the panels in the channel was necessarily a tight fit.



(A)



(B)

Figure 11—Development of coating holiday with time (Lower edge of panel) Panel 2; Devran epoxy at 30 m/s. (A) 56 hr; (B) 463 hr

The one panel where the initial values of capacitance and resistance appear to have been significantly affected by flow was the leading panel (Devran epoxy) in the 15 m/s section. As mentioned, this panel had not been preceded by a spacer plate and suffered visually detectable leading edge failure immediately after start-up. The initial value of capacitance (42,280 nanofarads) was higher by a factor of over ten than the other initial capacitance values. At the same time, the initial resistance was lower by better than a factor of 10. This data is significant in that it provides a relative gauge as to what values of capacitance and resistance correspond to gross failure that is visually detectable. In other words, changes in capacitance and resistance toward values approaching those measured on the failed Devran epoxy panel correspond to gross failure as opposed, for example, to a few minute pinholes not visually evident. It is noteworthy that throughout the entire test run only one other panel (Devran epoxy at 3 m/s) exhibited impedance values approaching those measured initially on the failed panel at 15 m/s. As mentioned, the Devran epoxy panel at 3 m/s was the only other panel which exhibited visually detectable damage.

The most significant aspect of the impedance data obtained during the test run in the low velocity section was that only five of the 30 exposed panels showed order of magnitude changes over the entire measurement period and, except for the panel noted above, none exhibited values approaching the range that would suggest gross failure. The majority of coated panels exhibited fairly stable capacitance and resistance characteristics. This correlated with the results of the final inspection. The only indication of failure were pinholes, primarily around edges, that were detected using a low-voltage holiday detector. Neither the impedance data nor the final inspection provided clear-cut evidence that one coating system was better than another or that there was a predominant velocity effect over the range investigated. For the most part, all of the coating systems were stable over the 812-hr test period.

DISCUSSION

The subject study was somewhat narrow in scope—it investigated only three marine coatings applied to a steel substrate and exposed to parallel flow. However, the results do provide some basis for projecting velocity thresholds and considering probable failure modes under the high velocity flow conditions examined.

Velocity Thresholds

The maximum velocity investigated in the study was 30 m/s (≈ 60 knots). As reported, gross coating damage occurred on one (Devran epoxy) of the four coated panels exposed at 30 m/s while the other three panels exhibited varying degrees of lesser damage occurring primarily near the edges. At the lower velocities (3–18 m/s), there was only one incidence of gross failure—on the leading edge of a panel exposed to impinging and cavitating flow. If one chose to ignore the area close to the

edge (within $\approx 1-2$ cm), the test results would indicate that three of the four panels at 30 m/s suffered negligible coating damage over the ≈ 900 hr exposure period. It could then be inferred that two of the three coating systems were stable up to and including 30 m/s and that the velocity threshold of these coatings exceeds 30 m/s for the given set of flow conditions and exposure period.

In attempting to project velocity thresholds from the subject study, the critical question is whether failure originating at edges can legitimately be ignored or, along the same lines, how much importance should be attached to edge damage. Many investigators choose to minimize the importance of failure occurring at edges because of the difficulty in obtaining a uniform, holiday-free coating along the edge. The reasoning is that the edge to surface area ratio on a test panel is often atypically higher than most service applications and, since edges are more prone to failure, time-to-failure data might be misleading if edge failures are given equal weight. It would be unreasonable to assume that a ship hull will be free of sharp edges, burrs, or the like. However, in the opinion of the author, the relative edge to surface area ratio characteristic of the 15×25 cm test panels exposed at 30 m/s represents a far worse condition than normally would be encountered in most service applications.

The test panels were especially susceptible to edge failure because of the fashion in which they had to be mounted in the flow channel. At 30 m/s, the steel panels were precisely fitted into slots in the acrylic plastic sidewall of the channel. The wall was then masked and the coatings applied. While this procedure insured minimal edge mismatch and a smooth panel-to-wall transition, it didn't permit elimination of all edge holidays at the tight crevice created between the panel edge and the sidewall. At the lower velocities (3-18 m/s), all panel edges were double-coated, but it is believed that some edge damage still occurred during installation. Thus, for the purpose of projecting velocity thresholds, this author tended to minimize the significance of coating damage close to the edge. Mindful of this assumption, it then does not seem unreasonable to propose that the velocity threshold for the MIL-P-24441 and Tarsel epoxy coatings would exceed 30 m/s for the flow conditions and exposure period characteristic of this study.

To what extent beyond the ≈ 900 hr exposure period these coatings would exhibit stable behavior (before gross failure occurs) is uncertain. However, extrapolation of the electrical impedance data for the test panel where edge failure was not a factor (e.g., see Figure 15) suggests that the coatings might have been stable for a significantly longer period than 900 hr. It would be unwise to attempt to relate directly these laboratory results obtained under a narrow set of experimental conditions to probable performance on a high speed ship given the complex nature of the ship's duty cycle and much broader range of environmental conditions. However, the results of the study do provide some basis for arguing that some of the conventional marine coatings might, in fact, provide acceptable protection in selected areas of a high speed ship hull. Certainly, the conventional marine coat-

ings cannot, as yet, be arbitrarily disregarded for all high velocity applications.

Probable Failure Mode

Although, because of the edge effect, the number of coating holidays per unit area of test panel tended to be more numerous than might be normally encountered in service, it is assumed that the actual coating holidays were physically not unlike those that would occur in service. Thus, while less significance was attached to edge-related failures in projecting velocity thresholds, the edge failures are believed to be quite significant for analyzing probable failure modes in parallel, high velocity seawater flow.

The first visual indication of coating failure (excepting the leading edge breakdown at 15 m/s) was blistering at different points near the edges on panels exposed at 30 m/s. There is not unanimous agreement on the mechanism underlying the formation of blisters in a barrier-type coating in seawater. However, the events generally associated with blister formation are as follows: (a) uptake or absorption of seawater by the coating due to osmotic forces; (b) seawater reorientation within the coating leading to the development of micropores where seawater exists in a continuous phase; (c) eventual development and/or migration of these seawater micropores through to the substrate/coating interface; (d) corrosion

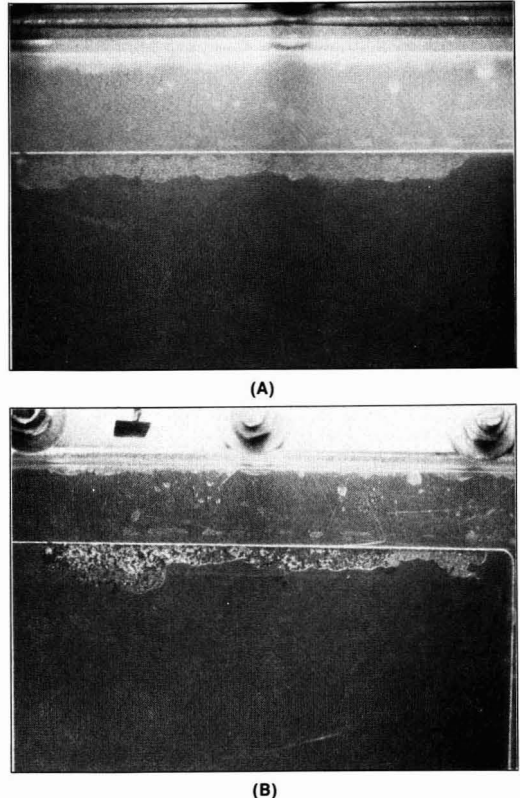


Figure 12—Development of coating holiday with time (Upper edge of panel) Panel 3; Devran epoxy. (A) 463 hr; (B) 898 hr



Figure 13—Panel 4 Taret epoxy after 898 hr exposure to seawater flowing at 30 m/s

of the substrate either at a seawater-filled micropore or at a holiday where seawater has penetrated through the substrate; (e) formation of corrosion products which cause localized expansion stresses; and (f) localized breakdown of the coating/substrate adhesive bond due to the combined action of corrosion and localized expansion stresses attributable to corrosion product formation and seawater osmosis. At 30 m/s, the experimental results suggest that the formation and development of macroscopic blisters are early stages of the overall damage process leading to gross coating disbondment. The results at the lower velocities provided little insight into probable failure modes under parallel flow since the only observed gross failure occurred on a leading edge subjected to impinging flow.

The experimental results do not provide any direct evidence as to the effect of flow velocity on the initial formation and/or the development of coating blisters. The fact that blistering was only detected at the highest velocity does not necessarily mean that the high flow velocity caused the blistering. As previously discussed, there were significant differences in the way the test panels at 30 m/s were mounted in the channel versus the panels at the lower velocities. It is believed that these differences gave rise to a greater number of edge holidays at the 30 m/s velocity and, therefore, better explain the occurrence of edge blistering at 30 m/s.

Hackworth, *et al.*¹² proposed a failure mode, dependent on blister formation, for coatings exposed to high velocity, non-cavitating flow. Hackworth hypothesized that, alone, the hydrodynamic shear stresses created in high velocity flow are much too low to cause gross coating failure. Using flow over a foil as an analog, he proposed that blisters might give rise to lifting forces of much higher magnitude that could rupture the coating. Although the subject study does not provide direct evidence to support Hackworth's proposed failure mode, in the opinion of the author, the proposed failure mode merits consideration.

In general conformance with Hackworth's proposed failure mode, Figure 16 presents a simplified picture of the forces that might arise about a blister and how the coating might ultimately respond to such forces. As shown, two mutually perpendicular forces—lift and pressure drag—are distributed over the surface of the blister.

The pertinent equations from airfoil theory describing these forces are as follows:

$$F_L = 1/2C_L A \rho V^2 \quad (1)$$

$$F_D = 1/2C_D A \rho V^2 \quad (2)$$

where,

F_L = lift force

F_D = pressure drag force

C_L = lift coefficient

C_D = drag coefficient

A = maximum projected area

ρ = density of fluid

V = velocity

A lifting force would create a differential pressure across the coating at the point of the blister. The situation would be somewhat analogous to the action of a plumber's helper (suction-type plunger). As the coating is lifted from the substrate, it would tend to create a vacuum in the area between the coating and substrate. The resulting differential pressure would accelerate seawater transport through the coating at the blister. The additional volume of seawater entering the blister would increase corrosion of the substrate leading to further breakdown of the adhesive bond and increased expansion stresses from corrosion product formation. Under the combined action of these forces, the blister would continue to grow. Ultimately, the blister reaches a critical size such that the hydrodynamic forces are resolved caus-

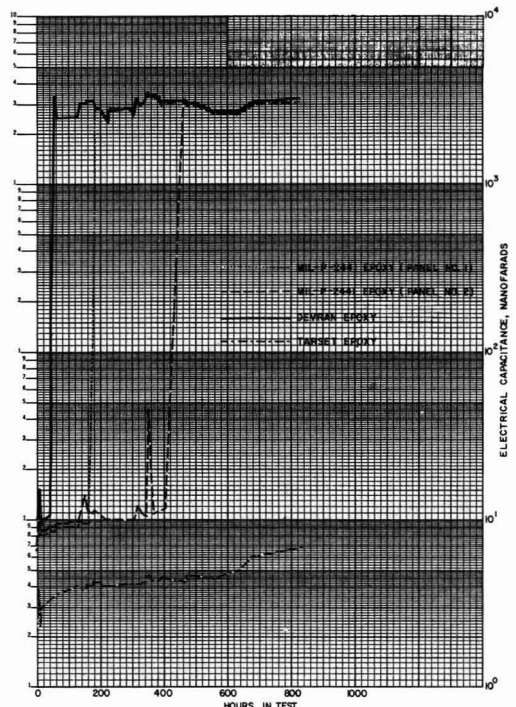


Figure 14—Electric capacitance vs time for coated panels exposed to flowing seawater at 30 m/s

ing a tensile stress in the coating that exceeds its inherent tensile strength and gross rupture occurs.

In order to get a feel for the relative magnitude of the forces that might be developed in this fashion, it is worthwhile to consider an example. Assuming a spherical-like blister, approximately 0.4 mm high and 6 mm in diameter, the resulting lift and drag forces can be estimated as follows:

Lift Force

$$F_L = 1/2 C_L A \rho V^2$$

where,

$$C_L = 20 h/d^* = (20)(0.4 \div 6) = 1.33$$

$$A = \pi (d/2)^2 = \pi (3)^2 = 28.27 \text{ mm}^2$$

$$= 2.83 \times 10^{-5} \text{ m}^2$$

$$\rho = 1000 \text{ kg/m}^3$$

$$V = 30 \text{ m/s}$$

$$F = (1/2)(1.33)(2.83 \times 10^{-5})(1000)(30)^2$$

$$F_L = 16.9 \text{ newtons}$$

Pressure Drag Force

$$F_D = 1/2 C_D A \rho V^2$$

where,

$$C_D = 0.4 \text{ (from Streeter}^{13})$$

$$A = 2.83 \times 10^{-5} \text{ m}^2$$

$$\rho = 1000 \text{ kg/m}^3$$

$$V = 30 \text{ m/s}$$

$$F_D = 5.09 \text{ newtons}$$

* $C_L = 20 h/d$, for $h/d < 0.07$ from reference (12)

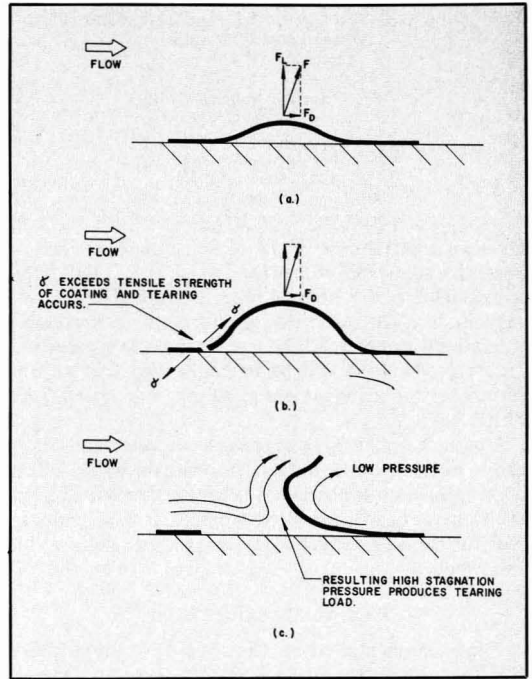


Figure 16—Possible coating failure at high velocity

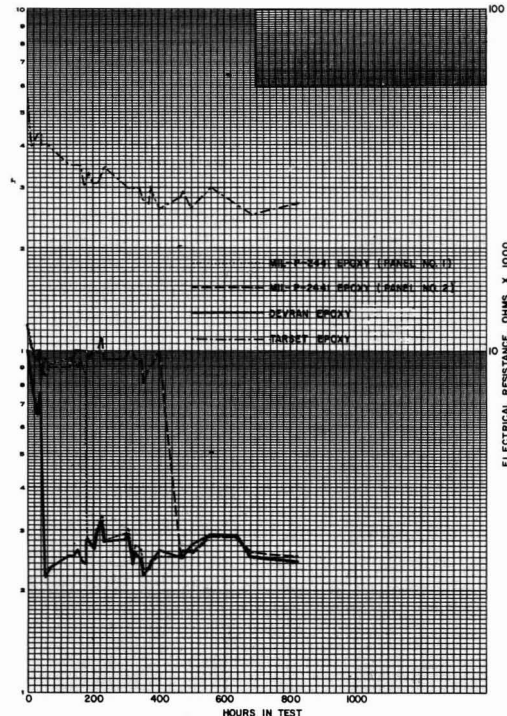


Figure 15—Electrical resistance vs time for coated panels exposed to flowing seawater at 30 m/s

The resultant of these two forces is 17.6 newtons.

If it is assumed that the resultant hydrodynamic load creates a tensile stress in the coating distributed about the perimeter of the blister somewhat like that shown in Figure 17, then a rough approximation of the coating area over which the applied load is distributed is $1/2 \times \text{circumference of blister} \times \text{coating thickness}$. Using the dimensions of the blister assumed above, the cross-sectional area is calculated as follows:

$$A = 1/2 \times \pi \times 0.006 \times 0.000254 = 2.39 \times 10^{-6} \text{ m}^2$$

Then, as a rough approximation, the resulting tensile stress in the coating is:

$$\sigma = F \div A$$

$$= 17.6 \div 2.39 \times 10^{-6}$$

$$\sigma = 7350000 \text{ newtons/m}^2 = 7350 \text{ Kpa}$$

The reported tensile strength* of the Tarjet Epoxy coating is 6900 Kpa. Adhesion measurements using an Elcometer Model 106 Adhesion Tester gave values of bond strength for all three coatings that ranged between 7,000 to 14,000 Kpa. Thus, as a final approximation, it appears that lifting forces sufficient to rupture the protective coating could be developed at blisters.

In comparison, the calculated shear stresses exerted by the fluid on the coating are significantly lower. At 30 m/s, the shear stress is calculated to be $\approx 1.64 \text{ Kpa}$ from the following equation:

$$\tau = 0.029 \rho V^2 (1/Re)^2$$

*Porter Co. Product Data Sheet A8089 (9/75)

where,

$$\begin{aligned} \tau &= \text{shear stress} \\ \rho &= \text{density} \\ V &= \text{velocity} \\ \text{Re} &= \text{Reynolds Number} = \frac{V4R}{\nu} \\ \nu &= \text{kinematic viscosity} \\ R &= \text{hydraulic radius} = A/P \text{ for} \\ &\quad \text{a rectangular channel} \\ A &= \text{cross-sectional area of channel} \\ P &= \text{wetted perimeter of channel} \end{aligned}$$

Over an area the size of the blister, this represents an applied load of ≈ 0.046 newtons which is $\approx 1/400$ of the load calculated for lift and pressure drag. The relative magnitude of the forces estimated from theoretical considerations lends credibility to Hackworth's proposed failure mode. It should be re-emphasized that all observed coating disbondment at 30 m/s was preceded by blistering.

The subject study does not provide experimental verification of Hackworth's model. However, the way in which coating failure was observed to occur certainly fits within the framework of Hackworth's model. It would appear that further work is deserving to experimentally verify this model.

Hydromechanical Wear

The experimental results showed that the applied dry film thickness of the coatings remained essentially constant during the velocity tests. In the past, there has been conjecture by many investigators, including this author, that hydromechanical wear or erosion could become a factor at the higher seawater velocities. However, the lack of significant reduction in the dry film thickness suggests that hydromechanical wear might not be a factor over the velocity range investigated under conditions of parallel flow. Again, one must guard against drawing too broad a conclusion from the limited data. Different coatings and/or a longer exposure period might have yielded different results.

Electrical Impedance Measurements

The experimental results demonstrated the utility of electrical impedance measurements as a non-destructive technique for detecting impending failure of non-conductive, barrier-type coatings. Throughout the study, mea-

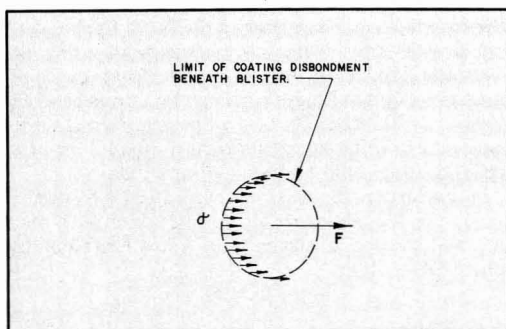


Figure 17—Possible distribution of tensile stress around blister

sured changes in coating resistance and capacitance correlated excellently with observed coating performance.

Electrical impedance data also provides a quantitative basis for ranking coating performance. When coatings are rated strictly by visual inspection, there is always a judgement factor depending on the individual making the inspection. It would seem that, with some modification of technique, the use of electrical impedance measurements could be extended to actual service situations. For example, the data might facilitate better judgement as to the condition of a protective coating on a structure and when recoat might be necessary.

Parallel Versus Impingement Or Cavitation-Type Flow

There is little direct evidence from the subject study to indicate that, at the higher velocities, parallel flow is significantly less damaging than impingement or cavitation-type flow. However, comparison of these results with the results of other work conducted under different types of flow tend to strengthen this supposition.

In the present study, the results obtained at 15 m/s provided the only indication as to the relative severity of different types of flow. Coating failure occurred almost immediately on the one panel subjected to impingement and cavitation-type flow (at the leading edge). The remaining panels not exposed to leading edge impingement and cavitation exhibited negligible coating deterioration over the 812-hr test period. At the lower velocities (3–12 m/s), impingement-type flow on the leading edge did not cause detectable coating damage. The panels exposed at 30 m/s and 18 m/s were not subjected to impingement or cavitation-type flow.

Lichtman and Kallas⁴ reported failures beginning after only one hour for vinyl-coated specimens exposed to cavitating flow at ≈ 28 m/s in a high speed nozzle apparatus. In the subject program, the first failure at 30 m/s was not observed until ≈ 54 hr of exposure.

In other work using a rotating disk apparatus, Lichtman, *et al.*¹ reported significant volume loss of teflon and polyvinyl chloride materials after 10 hr of cavitating flow at ≈ 30 m/s. In the subject study, two of the three epoxy-type coatings exhibited minimal volume loss after 898 hr exposure at 30 m/s.

The Bureau of Reclamation¹ conducted water tunnel tests on various coatings applied to conical-type steel specimens, subjected to both impingement and cavitating flow at ≈ 20 m/s. They reported gross failure of vinyl coatings in less than 10 hr test time. Thus, based on a comparison of other work, there is some supportive evidence suggesting that, at high velocities, parallel flow is not as damaging to coating materials as impingement or cavitation-type flow. This conclusion has been implied indirectly quite often in the literature but little data has been offered to substantiate it.

CONCLUSIONS

Neglecting edge-related damage, the results suggest the velocity threshold for the MIL-P-24441 Epoxy and Tarsel Epoxy coatings exceeds 30 m/s under the flow

conditions and time period characteristic of the study. It would be unrealistic to attempt to extrapolate these results to actual service situations. The results do provide an indication, however, that some marine epoxy coatings might be serviceable at velocities approaching 30 m/s, depending on the character of flow.

Under the given set of high velocity conditions, blister formation appears to be a critical part of the coating failure process. Hackworth proposed a failure mode, dependent on blister formation, that deserves consideration and that appears to justify further experimental study.

Under the given set of high velocity conditions, hydro-mechanical wear was negligible.

Electrical impedance measurements can be used to detect impending coating failure. These measurements also provide a quantitative basis for ranking coating performance.

In the velocity range between 15 and 30 m/s, the experimental results provide some evidence, both direct and indirect, that parallel flow is not as damaging as impingement or cavitation-type flow.

ACKNOWLEDGMENT

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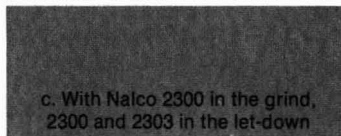
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Pyrolysis Gas Chromatographic Analysis (PGC) Of Methyl Methacrylate (MMA)-Ethyl Acrylate (EA) Copolymers

Swaraj Paul
AB Wilh. Becker*

The use of pyrolysis gas chromatography (PGC) for the quantitative estimation of methyl methacrylate-(MMA)-ethyl acrylate(EA) copolymer composition has been demonstrated. In the light of the existing polymer degradation theories, optimum pyrolysis conditions have been investigated. A calibration curve for the estimation of copolymer composition in unknown polymer samples has also been obtained from the NMR and the PGC results. Based on the thermogravimetric analysis (TGA) of the copolymers containing varying mole-fractions of MMA-EA monomers, activation energies for the thermal degradation has been determined. Activation energy for a MMA-EA copolymer containing 20 mole % of EA has been found to be 14.7 K cal/mole. This observed value was lower, however, than the value reported earlier for poly(methyl methacrylate) (PMMA), i.e., 42 K cal/mole. This has been supposed to be due to the lower molecular weight of the copolymer samples investigated in our case.

INTRODUCTION

Although there exist several physical and chemical methods for the characterization of copolymers, there has always been a need for the development of simple methods which can be used in the industrial laboratories where both time and cost play a very important role. As has often been observed, such simple methods are usually associated with complicated interpretation problems which many times lead to erroneous results. An attempt has been made here to extend the use of PGC for the quantitative estimation of copolymer composition in the light of the known polymer degradation mechanisms. These degradation mechanisms have been extensively studied and reported earlier in the literature.¹⁻¹⁷

After the first introduction of the concept of using thermal decomposition of polymers in combination with gas-liquid chromatography (GLC)¹⁸ for the identification of polymers, a large number of studies have been reported in the literature concerning the polymer characterization by PGC.¹⁹ In the beginning, PGC was used mainly for the rapid qualitative identification of polymers such as acrylate and methacrylate polymers.²⁰⁻²⁴ The first application of PGC for the quantitative estimation of the copolymer composition, directly from the chromatograms of their pyrolysis products, was made by Strassburger, et al.²⁵ PGC has also been used for both the structural characterization²⁶⁻²⁷ and the determination of polymer composition.²⁸⁻³³

Conditions essential for quantitative reproducibility in PGC have been studied by various workers.³⁴⁻⁴² Recently, American Society for Testing and Materials (ASTM) published a few recommendations to obtain reproducible results from the PGC analysis based on the interlaboratory studies for some selected polymers.⁴³ It was concluded that reproducibility could be achieved provided prescribed parameters for the gas chromatography and pyrolysis were followed carefully, i.e., the analyst uses identical clean pyrolysis elements, fast temperature rise times (~ 30 m sec), small sample sizes (< 75 μ g) and pyrolysis temperature of ~ 700°C. In some cases, even the nature and the flow rate of the carrier gas and the columns packing material have been shown to contribute to the reproducibility of the results.

The PGC offers many advantages over other techniques, such as infrared and NMR. These advantages are small sample size, little sample preparation, and rapid turn-around time. The equipment is relatively inexpensive, easily operable and it provides a possibility for characterizing even insoluble polymer samples. McCormick²⁹ and Haken, et al.³⁰ have demonstrated the use of PGC for the estimation of acrylate copolymer compositions. They showed that, based on the amounts of the monomers evolved, distinction between homo- and copolymers can

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be made. Haken, et al.³⁰ put forward certain homopolymer (HFI) and copolymer (CFI) fragmentation indices for different monomer types which can be directly used to evaluate the polymer composition by multiplying the amount of monomers evolved as determined from the chromatographic peak areas with either HFI or CFI, depending on whether it is a homo- or a copolymer.

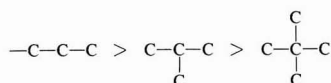
Since the degradation products as a result of the pyrolysis of polymers, as will be shown later, vary with the nature of the polymer composition due to the competition among depropagation, intramolecular and intermolecular transfer, a linear relation between the ratio of the monomers in the polymer and in the decomposition products, shown earlier,⁹⁻¹⁰ seems to be rather unreasonable, especially in the case of copolymers containing acrylate monomers. In the present paper, various mechanisms for the thermal degradation of copolymers containing acrylate monomers have been reviewed and, in the light of these mechanisms, PGC analysis of MMA-EA copolymers was undertaken. Optimum pyrolysis conditions and a calibration curve based on the PGC analysis were obtained which could later be used to characterize unknown MMA-EA copolymer samples in the latex form since the conventional methods of characterization are difficult to imply for latexes due to their insolubility in most of the solvents. Further, acrylate copolymers were chosen as a subject of this study because a large number of binders used in the coating industry are comprised mainly of acrylate polymers.

Depolymerization Mechanism

Before analyzing the results we obtained, it may be useful to understand the degradation mechanisms of acrylate polymers proposed by various workers¹⁻¹⁰ and how the nature of the degradation products varies with the polymer compositions. McCallum¹¹ has reviewed all the existing mechanisms of depolymerization of PMMA polymer. Based on these studies, two mechanisms, in general, were proposed for the thermal degradation of PMMA, one initiated by the unsaturated chain ends and the other due to the random scission of polymer chains. Each of the two mechanisms results to a different extent in a molecular weight change in the polymer samples and also gives rise to the evolution of different amount of monomers at different degradation temperatures. McNeill⁹ has shown from the thermal volatilization analysis (TVA) of PMMA and MMA-copolymers that, in the case of copolymers, both the degradation initiated by the unsaturated chain ends and the zip length decreases.

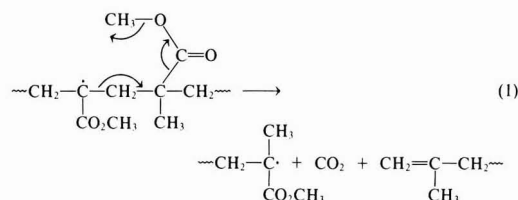
PMMA has always been shown to give only monomers on pyrolysis.⁶ This was believed mainly to be due to the random scission of the chains which are not accompanied by a hydrogen transfer due to the steric hindrance caused by the $-CH_3$ and $-COOCH_3$ groups present on every alternate carbon atom which is quaternary. The scission thus results in the formation of free radicals which, once initiated, unzip completely, resulting in monomer generation. Madorsky¹² suggested from the degradation studies of polyethylene (PE), polypropy-

lene (PP), and polyisobutylene (PIB), that the relative strengths of the $-C-C-$ bonds vary as follows:



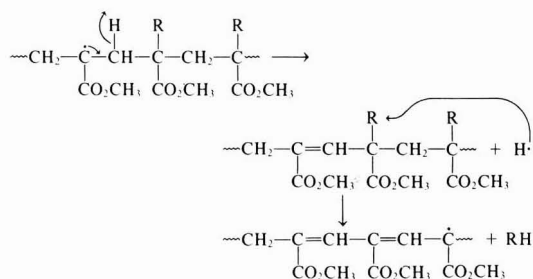
which in turn results in different monomer yields. Many interesting studies on the thermal degradation of copolymers containing acrylates have been reported in the literature.^{1-5,7-9} Based on the activation energy data for the MA and MMA homopolymers and copolymers, Grassie, et al.² suggested that the chain scission in the case of MMA-EA copolymers is an integral part of a radical chain process. Since degradation of the copolymers occurs at the same temperature as that of PMMA, it was further proposed that random initiation will be the most likely initiation process in this chain reaction and its rate will be independent of the MA content. Based on the experimental results, the following two degradation mechanisms were proposed for the copolymers containing methyl acrylate (MA).

CASE I: Propagation resulting from the abstraction of a tert hydrogen atom from a MA unit:



The radical (I) will liberate monomer and the chain fragments.

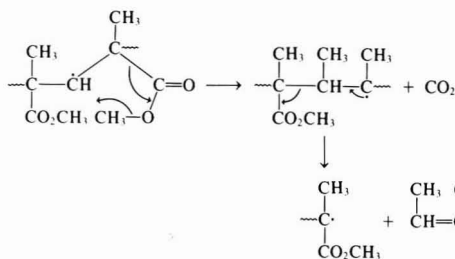
CASE II: Initiation is associated with the MA units and propagation step is random:



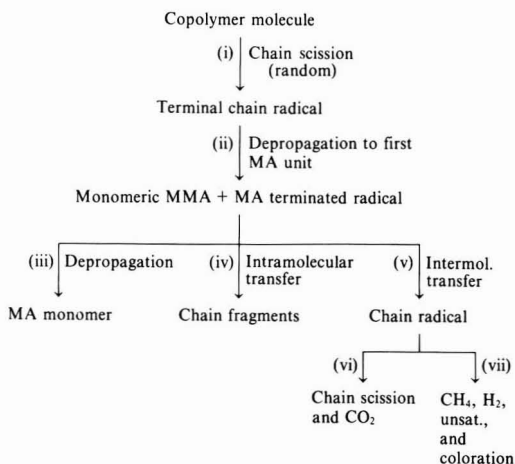
where R = H or CH_3

Based on the rate exponent of 0.5 for the degradation process, it was suggested that a significant proportion of both initiation and propagation was random. However, it was difficult to say if a proportion of initiation or propagation or both were associated with MA units. Although the tert hydrogen atom in MA will be much more liable to participate in the propagation step, a participation of the chain methylene hydrogen atoms has

also been observed and has been explained to be due to the much higher concentration of the latter. The probable mechanism for chain scission in that case can be shown as follows:



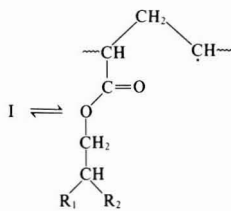
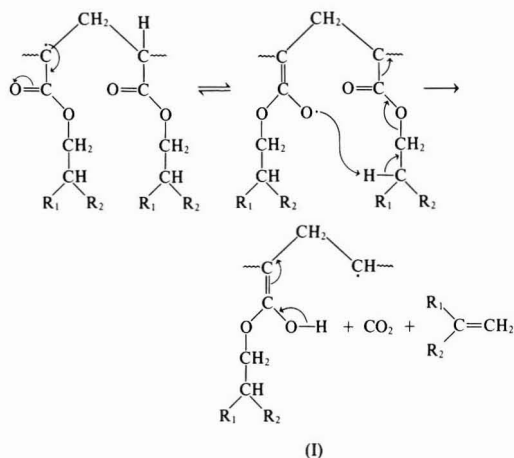
Thus the degradation mechanisms of polymers containing acrylate units in the chains can be summarized as follows:



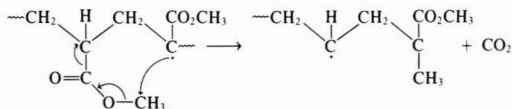
The reactions (iii), (iv), and (v) mostly determine the principal characteristics of the overall reaction and vary with the copolymer composition. If the terminal MA resulting from (ii) is single, reactions (iii) and (v) are possible, whereas (iv) becomes significant with increasing amount of MA in the copolymer. Although tertiary hydrogen atoms are more reactive than the methylene hydrogen atoms, the abundance of the latter makes (v) a random process. Further, reaction (vii) becomes relatively more important as the proportion of MA in the copolymer is increased. Grassie, et al.³ found that the amount of EA evolved from a MMA-EA copolymer containing 25 mole % EA was more than twice that evolved from the acrylate homopolymer. The obvious explanation is that the depolymerization reaction which is initiated in MMA segments of the copolymer molecule is capable of passing through and liberating isolated acrylate units, but when the depolymerization reaction encounters a sequence of adjacent acrylate units, transfer reactions, typical of polyacrylates, occur preferentially. Higher temperatures favor the production of gases, liquids, and chain fragments at the expense of residue.

Grassie, et al.,⁷ from the degradation studies of primary and secondary polyacrylates, showed that monomers are generally produced up to 30–50% conversion but at higher temperatures decomposition of the pendant ester groups results in other products. The main decomposition products resulting from the decomposition of the ester side groups in polyacrylates were shown to be CO₂, corresponding olefin, alcohols, as well as traces of monomer.¹³ Polyacrylates containing tertiary pendant ester groups such as tertiary butyl ester, result in almost quantitative ester decomposition to isobutene, acrylic, and methacrylic acid.^{14–15} The formation of the above mentioned products was explained as follows:

Generation of Olefin and CO₂



This will suggest that the ratio of CO₂ and olefin production can never exceed 1, however in practice it was greater than 1 which was explained as follows:

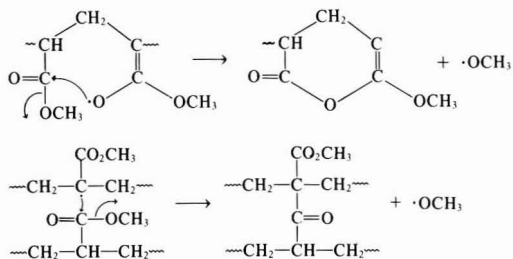


This explained the formation of corresponding methacrylates which was also found experimentally during the degradation of polyacrylates. Four different mechanisms were proposed by Cameron and coworkers^{13,16} to explain the formation of alcohols. Two of the following mechanisms have been found to be reasonable based on the

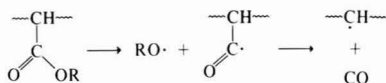
Table 1—Molar Feed Compositions for the Synthesis of Copolymers Containing Different Mole Fraction of Monomers

Sample No.	Theoretical Molar Composition		Monomer Feed Composition	
	EA (mole%)	MMA (mole%)	EA (mole%)	MMA (mole%)
RS 3	20	80	33.2	66.8
RS 4	40	60	53.9	46.1
RS 5	60	40	69.5	30.4
RS 6	80	20	83.8	16.2

presence of γ , δ unsaturated δ -lactone and the C=O groups shown by IR-analysis of the residues:



It has been shown earlier that in the gas phase, the alkoxy radicals have stabilities in the order $\text{CH}_3\text{O} \cdot > \text{CH}_3\text{CH}_2\text{O} \cdot > \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{O} \cdot > (\text{CH}_3)_2\text{CHCH}_2\text{O} \cdot$. Thus, it may be expected that the alcohol elimination reaction will be less likely to occur in the polymers with fewer β -hydrogen atoms in the ester group and thus, in turn, will account for the decreasing yield of alcohol in the primary acrylate polymers in the order ethyl $>$ n-propyl $>$ n-butyl $>$ 2-ethyl hexyl. The alkoxy radicals can, in general, react in two ways: either by hydrogen abstraction resulting in alcohol or by disproportionation resulting in aldehydes. No aldehydes were detected; this was perhaps due to the fact that disproportionation reaction is very much diffusion controlled and hence is not very favorable in the viscous polymer. Trace amounts of CO can be explained as follows:



A review of the thermal degradation mechanisms of the acrylate containing copolymers suggests that the

Table 2—Weight Average Molecular Weight (\bar{M}_w), Number Average Molecular Weight (\bar{M}_n), and Molecular Weight Distribution (\bar{d}) Results As Determined from GPC

Sample No.	$\bar{M}_w (\times 10^{-3})$	$\bar{M}_n (\times 10^{-3})$	MWD (\bar{d})
RS 3	26.5	12.9	2.04
RS 4	24.4	11.2	2.16
RS 5	23.8	9.4	2.52
RS 6	20.7	6.6	3.13
RS 7 (PEA)	19.3	5.0	3.83

nature and the amount of the degradation products formed as a result of pyrolysis depend very much on the nature of the acrylate monomer present in the polymer and on the pyrolysis temperature used. This suggests further, that in order to use the PGC for the quantitative estimation of copolymer composition, a detailed knowledge of the degradation mechanism and the influence of the pyrolysis conditions on the products formed for every particular acrylate copolymer systems is very essential. These results, in turn, can then be combined or extrapolated to elucidate the composition of polymers containing three or even more monomers. Thus, in the light of the preceding discussions, it is obvious that the main pyrolysis products of MMA-EA copolymers should be monomers, methane, ethylene, hydrogen, CO_2 , CO, ethyl methacrylate, and ethanol. Further, if the pyrolysis conditions are chosen such that the degradation of the polymers can be restricted only to the unzipping reaction, instead of decomposition of the pendant ester groups, then the amount of monomers evolved as a result of pyrolysis of the polymers may be used to obtain a calibration curve which can later be used for the estimation of copolymer compositions.

EXPERIMENTAL

Synthesis of MMA-EA Copolymers

All the samples used were random copolymers and prepared by solution polymerization in toluene in a one liter flask fitted with a stirrer, nitrogen inlet, and monomer inlet tubes. The materials used, MMA and EA, were purified according to the methods described earlier,⁴⁴ while benzoyl peroxide was used as such without any further purification. Copolymerization was carried out always using one mole of monomer, 0.00825 mole of benzoyl peroxide as radical initiator, and 2.17 mole of toluene at the reflux temperature under a nitrogen gas atmosphere. The monomer mixture together with initiator was added slowly to toluene contained in the one liter flask in a span of 3-4 hr and the polymerization was discontinued at about 70% conversion by cooling the reaction mixture. The polymer formed was isolated by precipitation twice in petroleum ether (bp 40-60°C) from chloroform solution. The amounts of MMA and EA taken in the feed for the synthesis of copolymer containing different mole-fractions of EA and MMA are summarized in Table 1. These amounts have been calculated based on the reactivity ratios of EA and MMA as 0.28 and 2.0, respectively.

Their weight and number average molecular weights were determined by gel-permeation chromatography (GPC) and the results are presented in Table 2.

Proton-NMR

The proton-NMR spectra were obtained using a Varian EM-360, 60 M Hz spectrometer. Sweep width of 10 Hz and sweep time of 5 min were employed. Deuterated chloroform and trimethylsilane were used as a solvent and internal standard, respectively. Representative spectra of two MMA-EA copolymers containing about

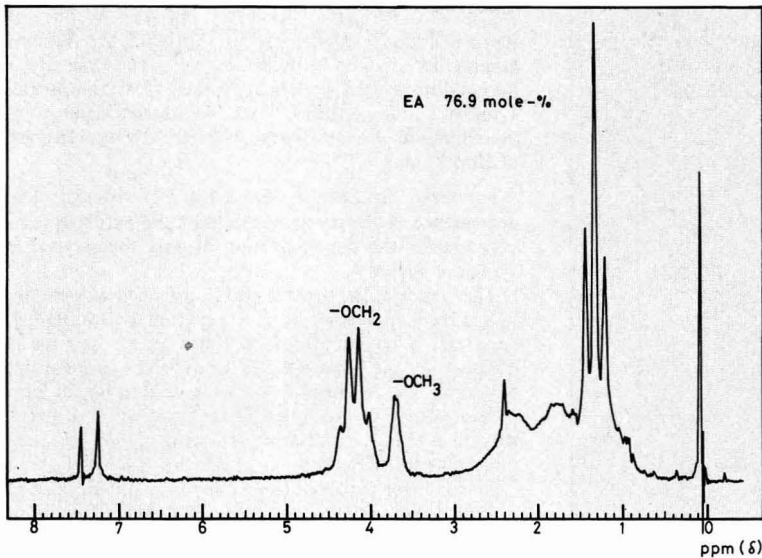


Figure 1—NMR spectra of MMA-EA copolymer containing 76.9 mole% of EA

80 and 60 mole % of EA are shown in *Figures 1 and 2*. The bands appearing at 3.7δ and between $4-4.3\delta$ were attributed to $-\text{OCH}_3$ and $-\text{OCH}_2$ protons of the ester functionality in MMA and EA and the ratio between the two has been used to estimate the copolymer composition.⁴⁵

The molar compositions obtained from the NMR results for various copolymers are summarized in *Table 3*.

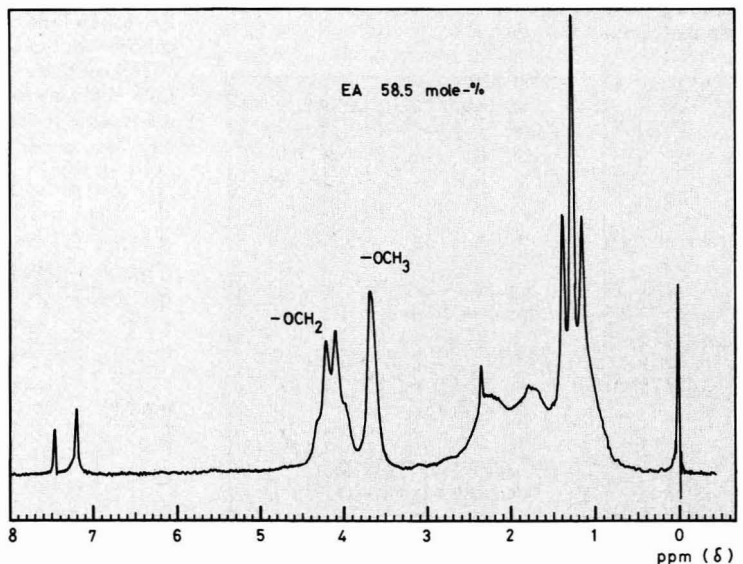
Pyrolysis Gas Chromatography

PYROLYZER: A Chemical Data System pyroprobe Model 150 was used as the pyrolyzer. $20\mu\text{g}$ of polymer samples from a 1% solution in tetrahydrofuran (THF)

was applied on the platinum ribbon and, after drying for 15 min at room temperature, the coated ribbon was mounted in the pyrolysis head and heated to 100°C for 20 sec using a ramp speed of $20^\circ/\text{ms}$. The optimum operating conditions were interface temperature, 200°C ; ramp, 20°C ; interval, 10 sec; final temperature 450°C . Interval indicates the period for which the final temperature was held.

GAS CHROMATOGRAPH: A Perkin-Elmer F 11 gas chromatograph with $2.5\text{m} \times 0.3\text{cm}$ stainless steel column packed with 100/120 mesh chromosorb W (AW) coated with 10% silicone gum rubber was used. Operating conditions used were helium flow, 30 mL/min ; injection port

Figure 2—NMR spectra of MMA-EA copolymer containing 58.5 mole% of EA



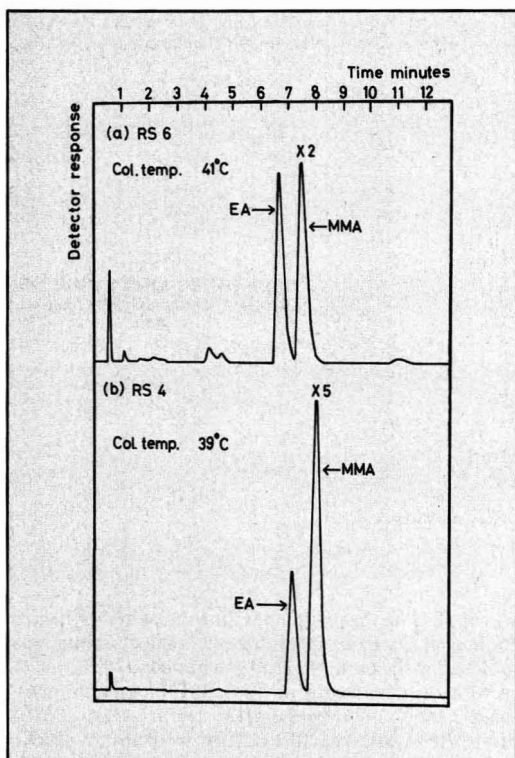


Figure 3—GPC pyrograms of MMA-EA copolymers

temperature, 150°C; flame ionization detection range 1 with suitable attenuation; oven temperature about 70°C.

Representative pyrograms at X2 and X5 attenuation are presented in Figure 3. Calculations of the percent MMA and EA in the samples were made from the ratio of the integrated areas of MMA and EA peaks from the gas chromatographs.

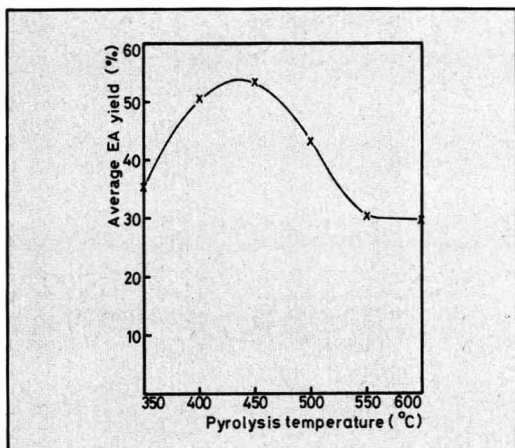


Figure 4—Dependence of %EA yield from poly(ethyl acrylate) (PEA) on the pyrolysis temperature

THERMOGRAVIMETRIC ANALYSIS (TGA): A duPont thermal analyzer model 951 was used for the thermogravimetric analysis of the copolymers. The experimental conditions used were heating rate, 10°C/min; scale, 1 mg/in.; time constant, 1 sec. The experiments were carried out in an atmosphere of N₂ gas flowing at the rate of 50 mL/min.

PYROLYSIS STUDIES OF MMA-EA COPOLYMERS: The dependence of the monomer yield on the pyrolysis temperature for the decomposition of polyethylacrylate is shown in Figure 4.

The values of the percent ethyl acrylate yield were obtained from an average of three experimentally obtained values. It is obvious from the figure that a maxima is obtained for the %EA yield at the pyrolysis temperature of 450°C. The decrease in the %EA yield at higher temperatures may be attributed to the production of products other than EA monomer as a result of inter- or intramolecular transfer reactions previously discussed.

Since longer pyrolysis times due to the similar reasons may also lead to the reduction in %EA generation, influence of the flash time on EA generation has also been studied at the pyrolysis temperature of 450°C. The results are presented in Figure 5.

It is evident from Figure 5 that %EA yield also decreases with increasing pyrolysis time; thus, a flash time of 2 sec seems to be the optimum time.

Based on these observations, it can be concluded that the optimum pyrolysis conditions for PEA are: pyrolysis temperature, 450°C; and flash time, 2 sec. Only polyethylacrylates were studied to obtain the optimum conditions because the transfer reactions are only predominant in the case of polymers containing ethyl acrylate monomer. All the pyrolysis studies of the MMA-EA copolymers were made at these optimum conditions and the ratio of ethyl acrylate and methyl methacrylate evolved was obtained from the gas chromatographic analysis. The ratio of EA vs. MMA monomers obtained as a result of pyrolysis was then correlated with EA/MMA ratio obtained from the NMR analysis of the copolymers. Such a correlation is presented in Figure 6.

This calibration curve is not linear. A quadratic function provides a reasonably good fit, showing a slope that decreases with increasing amount of EA in the copolymer. The equation of the quadratic is:

$$Y = 0.0797 + 0.4056 X - 0.0523 X^2$$

Table 3—Molar Composition of MMA-EA Copolymers Obtained from NMR Analysis

Sample No.	Monomer Composition In Feed		Monomer Composition In Copolymer	
	EA (mole %)	MMA (mole %)	EA (mole %)	MMA (mole %)
RS 3	33.2	66.8	24.7	75.3
RS 4	53.9	46.1	43.9	56.1
RS 5	69.5	30.4	58.5	41.5
RS 6	83.8	16.2	76.9	23.1

Table 4—Activation Energies of MMA-EA Copolymer Decomposition As Determined from TGA

Sample No.	Activation Energy (K cal/mole)
RS 3	14.69
RS 4	14.36
RS 5	19.42
RS 6	18.46

where $Y = \text{EA/MMA}$ ratio obtained from PGC analysis and $X = \text{EA/MMA}$ ratio obtained from the NMR analysis. A formal determination of the significance level of the above equation was made by referring the mean square ratio to the appropriate F distribution with 3 and 1 degrees of freedom. This was found to be overwhelmingly significant (0.5%). Correlation coefficient $r(X, Y)$ for the above equation was shown to be 0.98. The decrease in the slope with increasing EA content in the copolymer suggests a blocking effect as well as the transfer reactions resulting in products other than monomers due to the presence of EA units in the copolymer, as has been discussed earlier. Further evidence for the blocking effect has been provided by thermal gravimetric analysis (TGA) of the copolymers where the slope of the %wt loss vs. temperature plots has been found to decrease with increasing EA content in the copolymer. However, at higher temperatures the difference in slopes decreases due to the random behavior of thermal degradation. TGA results are summarized in Figure 7.

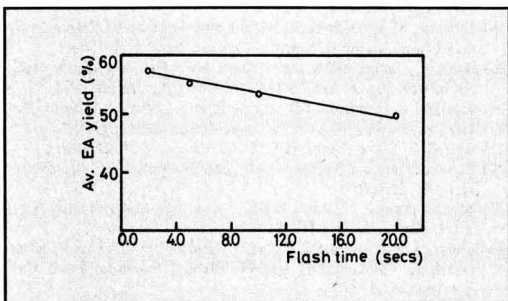
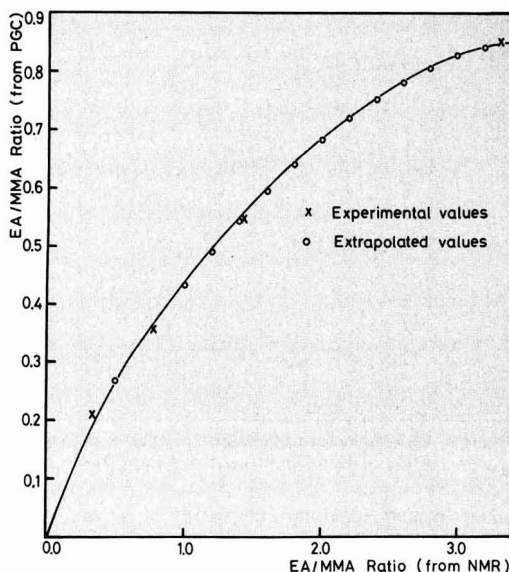
Activation energies for the thermal degradation of copolymers were determined using Coats and Redfern's⁴⁶

method. Plots of $-\text{Log} \left[\frac{-\text{Log } F}{T^2} \right]$ vs. $1/T$ are shown in

Figure 8. Here F is the fractional weight of the polymer remaining at temperature $T^\circ\text{K}$.

From the slopes of the straight lines, activation energies for the copolymer decomposition have been determined, since the slope should be equal to $E/2.3 R$, where E is the activation energy and R is the gas constant and is taken to be $1.9872 \text{ cal}^\circ\text{K}^{-1} \text{ mole}^{-1}$. The activation energies are summarized in Table 4.

However, the values of the activation energies obtained were found to be lower than the values reported

**Figure 5—Dependence of %EA yield from PEA on the flash time****Figure 6—Calibration curve for MMA-EA copolymers obtained from the PGC and NMR results**

earlier^{5,47} for PMMA, i.e., 42 Kcal/mole. The low values may be due to the low molecular weight of polymers used in our investigations. The blocking effect due to the presence of EA units can be explained as follows. It has been shown earlier⁴⁸ that the activation energy, E_o , is a composite quantity and can be represented as:

$$E_o = 1/2 E_i + E_d - 1/2 E_t$$

where E_i , E_d , and E_t are the energies required for initiation, depropagation and termination steps, respectively, of the overall reaction. In copolymers of very low acrylate content the initiation step is likely to be the same as in pure PMMA and the energy involved in the termination step, which is diffusion controlled because of the high viscosity of the degrading polymer, will not change significantly. This would imply that the increase in activation energy may be attributed to the increase in the activation energy for depropagation, E_d . But E_d can be expressed as follows:

$$E_d = E_p - \Delta H_p$$

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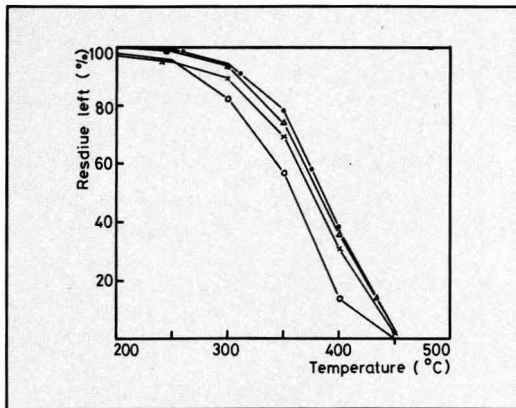


Figure 7—TGA thermograms for MMA-EA copolymers. Legend: RS3 (○); RS4 (×); RS5 (△); and RS6 (●)

where E_p and $-\Delta H_p$ are the energy of activation for propagation and heats of polymerization, respectively. Here, the value of E_p is shown to be constant for a large variety of polymers and is found to be 5 K cal/mole.⁴⁹ Thus, any increase in E_d with increasing acrylate content would be attributed to an increase in $-\Delta H_p$. In fact, it was shown experimentally⁵⁰ that heats of polymerization for ethyl acrylate are greater than MMA and the values were found to be 78 and 56 K J/mole, respectively. Thus, it is evident that the presence of increasing mole fractions of EA in the copolymers will render a blocking effect, raising the activation energy for the thermal degradation of polymers. However, at higher EA content, the activation energy was shown to decrease. This may be due to the complicated situation arising from the new initiation steps involving EA units, and was discussed earlier, resulting in the intrusion upon the simple depolymerization to monomer which is typical only for pure PMMA polymers.

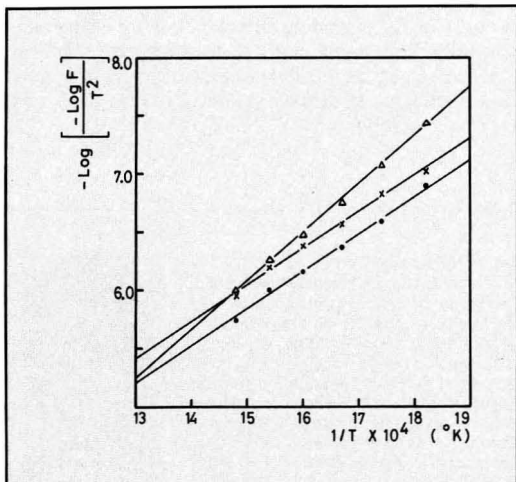


Figure 8—Activation energy estimation plots as obtained from TGA results. Legend: RS3 (●); RS4 (×); and RS5 (△)

CONCLUSIONS

It can be concluded from this investigation that a knowledge of the thermal degradation mechanisms of polymers is very essential to extend the use of PGC for the quantitative estimation of the copolymer composition. Further, if the pyrolysis temperature is kept at such a level that only unzipping reaction occurs during the thermal degradation of polymers, polymer compositions can be determined directly from the amount of monomers liberated in the decomposition products using the calibration curves for individual acrylate copolymers.

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HALOX REPORT: TR9

Inhibiting steel corrosion with calcium boro silicates

Let's review the three principal classes of anti-corrosive metal primers:

Barrier primers protect simply by raising the electrical resistance across the anodic and cathodic areas of local galvanic corrosion cells on the steel surface.

Zinc-metal containing films act as the anode and the steel itself is the cathode. The films override all local cell action on the steel surface, and initially, at least, sacrifice themselves to maintain the metal substrate intact.

Inhibitive primers, the most widely used, protect by slowly releasing inhibitive ions from pigments—or reaction products of pigments and vehicles from the film—which are carried by diffusing water to the steel surface. There, they modify the degrees of anodic and/or cathodic polarization—or increase the thickness of the air-formed layer. This increases the electrical resistance of the total coating system.

Direct and indirect

This third class can be divided into direct and indirect inhibitors. Direct inhibitors use pigments that release inhibitive ions; indirect inhibitors use pigments that, not necessarily inhibitive themselves, produce soluble inhibitive species when reacted with chemically modifying constituents from the vehicle.

Historically, the most effective inhibitive pigments have been those containing chromates (direct) or lead (indirect). But because both are under increasing criticism and regulatory constraints, the paint industry is testing alternatives.

HALOX shows promise

Halox composite pigments show considerable promise. These are silicate composites of basic phosphates or borates of calcium, barium, magnesium, or zinc.

Synergistic performance

Calcium phosphate and calcium borate are known as corrosion inhibitors, of course, but we find them to be more efficient as a composite or complex with silica or silicate minerals. We believe that the change from a crystalline to an amorphous state during manufacture improves effectiveness.

Mechanism of inhibition

Early tests in oleoresinous systems, comparing performance with that of red lead and chromated pigments, led us to the assumption that boro silicates are indirect inhibitors like lead-base products. Tests by Abou-Khalil of Cambridge University, who worked with Halox CW-221, supported this. However, our similar experiments with both CW-221 and CW-2230 (higher B₂O₃ pigments) indicated a direct as well as indirect contribution.

Mayne-Appelby tests

Results of our Mayne-Appelby tests using liquor extracts of pigments and paints on sandblasted steel appear in Table 1. The times to first corrosion for red lead pigment

and its paint show red lead to be an indirect inhibitor. Similar comparisons for CW-221 and CW-2230 show them to be both direct and indirect. The results of the same experiment with cold-rolled steel and aged liquors (Table 2) seem more definitive.

Tests of Halox pigments in both latex paints and the relatively nonreactive vinyl toluene lacquers support the assumption of a direct contribution.

Table I

Corrosion of Sandblasted Steel in Aqueous Extracts of Pigments and Paints

Extract	Time to First Corrosion Hrs.	Propagation at Six Weeks	Weight Loss in Coupon % of Original Weight
Red Lead Pigment	0.29/0.14	Complete/Complete	0.66% Loss/ 0.89% Loss
Red Lead/ Linseed Oil Primer	4.69/2.40	Moderate/ None	0.80% Loss/ 0.08% Gain
Halox CW-2230	3.36/2.70	Very Light/ None	0.56% Loss/ 0.12% Loss
Halox CW-2230/ Linseed Oil Primer	1.00/0.64	Moderate/ Moderate	0.77% Loss/ 0.64% Loss
Halox CW-221	2.32	Moderate	0.45% Loss
Halox CW-221/ Linseed Oil Primer	41.0/17.0	None/None	0.22% Gain/ 0.26% Gain

All coupons were blasted steel stored in air for 24 hours prior to testing

Table II

Corrosion of Cold Rolled Steel in Aqueous Extracts of Pigments and Paints Clean Ground Q-Panels in Aged Extract Liquors

Extract	Time to First Corrosion Hrs.	Propagation at Six Weeks
Red Lead Pigment	0.9/1.9	Complete/Complete
Red Lead/Linseed Oil Primer	1000+/1000+	None/None
Halox CW-2230	1000+/1000+	None/None
Halox CW-2230/ Linseed Oil Primer	1000+/1000+	None/None
Halox CW-221	1000+	None
Halox CW-221/ Linseed Oil Primer	24/32	Moderate/Moderate

All coupons were clean Q-Panels Ground on One Side, stored in air for several days before testing

Effective substitute

In our opinion, the calcium boro silicates, in properly formulated primer systems, are likely candidates for the nontoxic replacement of lead and hexavalent chromium-based inhibitors.

We invite your inquiry for more details on Halox pigments and specific applications where corrosion resistance is a consideration.



HALOX PIGMENTS

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A DIVISION OF HAMMOND LEAD PRODUCTS, INC.

Water-Borne Surface Treatment for Wood Based on Zinc Salts and a Latex: An SEM/EDXA Investigation

R.L. Desai

Eastern Forest Products Laboratory*

and

W.A. Coté, Jr.

State University of New York †

The distribution and location of the components of water-borne protective zinc salt-latex systems applied to wood have been examined by scanning electron microscopy and energy dispersive x-ray analysis (SEM/EDXA) technique. Effects of soaking time, method of treatment application, and leaching of the treated wood on the distribution of the components have been investigated. The results indicate that most of the zinc was deposited on the surface when applied as the carbonate salt, but that it penetrated the substrate surface with a thiocyanate salt system. However, the thiocyanate salt system was the more leachable of the two. Increased soaking time resulted in increased latex penetration into the substrate, and it appears that the latex diffused into the substrate through ray tracheids or pits or both.

INTRODUCTION

In a previous report,¹ the application of SEM/EDXA techniques to determine the distribution of some of the constituents of an ammoniacal zinc salt-latex system was discussed. In laboratory studies, this system has been found to substantially upgrade the properties of wood,²⁻⁶ improving its resistance to fungal and extractive staining and to moisture cycling. Further, the system has been found to be compatible with a variety of top coats.

The latex component used in this study differs from that used previously in that it is better suited to detection by EDXA. In addition, the effect of dipping time and the leachability of pressure-impregnated preservative chemicals were also studied.

EXPERIMENTAL

Materials

All the chemicals used were of technical grade. The wood substrate used was edge grain eastern white pine (*Pinus strobus* L.). The latex was polyvinyl chloride with 55% solids (Geon 577; particle size (average) 220 nm). No pigments were used.

Methods

PREPARATION OF THE SYSTEMS: Three systems were used: ammoniacal zinc thiocyanate salt solution; ammoniacal zinc carbonate salt solution; and the zinc salt-latex system, containing ammoniacal zinc thiocyanate and the polyvinyl chloride latex.

A concentrated solution of ammoniacal zinc thiocyanate was prepared as described in the previous report.¹ For the ammoniacal zinc carbonate concentrate, ammonium carbonate was used in place of ammonium thiocyanate and the weight ratio of zinc oxide to ammonium carbonate was 5:6. The concentrates were diluted with water, ammonia, or both to give the desired concentrations for further use. The salt-latex system was prepared by diluting the thiocyanate concentrate with water to give a 10% zinc solution (calculated as ZnO). This was mixed with an equal volume of the latex previously diluted with water to 10% solids. The resulting solution contained 5% each of latex solids and zinc (calculated as ZnO).

PREPARATION OF TREATED WOOD SAMPLES: Small white pine panels (100 × 50 × 10 mm), conditioned at 21 °C and 50% relative humidity, were treated with the salt-latex or salt solutions as follows:

- (1) Panel was dipped for 10 sec in the salt-latex solution and air-dried.
- (2) Same as (1) with 1 min dip.
- (3) Same as in (1) with 24 hr soak.

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(4) Panel was impregnated with the salt-latex solution by first evacuating to 13.3 mPa (10^{-4} torr) and the solution was applied under 750 kPa (110 psi) for 30 min. The panel was then air-dried.

(5) Panel was brush-coated with the ammoniacal zinc carbonate salt solution containing 5% zinc oxide and 3% ammonia, and air-dried.

(6) Same as in (5), using the thiocyanate salt solution.

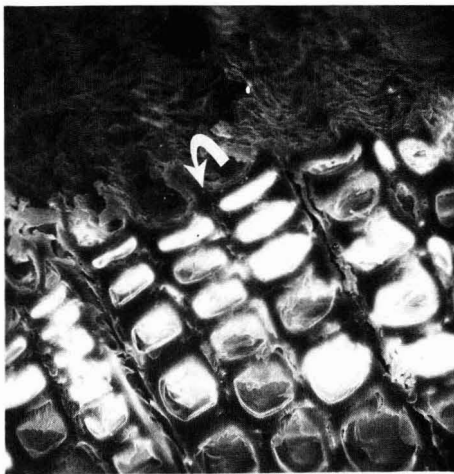
LEACHING STUDIES: For leaching experiments, panels with treatments (4), (5), and (6) were soaked in a large quantity of water for 24 hr and then air-dried.

SEM/EDXA ANALYSIS: Specimen preparation and SEM/EDXA measurements were the same as in the previous report.¹ The instrumentation and the proce-

dures for obtaining various scanning micrographs and the elemental maps were identical to those used previously. For the leaching studies, microtome sections were cut from the ends (outer) as well as from newly created surfaces (center) by cutting the soaked panel in half. The direction of microtoming was perpendicular to the coated surface and section thickness was approximately 30 μm . It should be noted that *Figures 1a* and *5a* are microtomed surfaces, but are not sections.

RESULTS AND DISCUSSION

The scanning electron micrograph and corresponding Zn X-ray map for the panel brush-coated with the carbonate salt system (treatment 5) are given in *Figure 1*. A

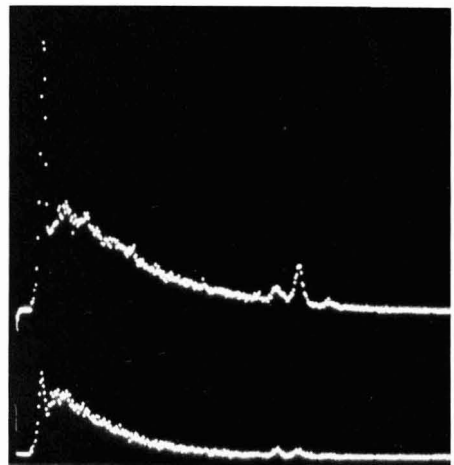


(A)

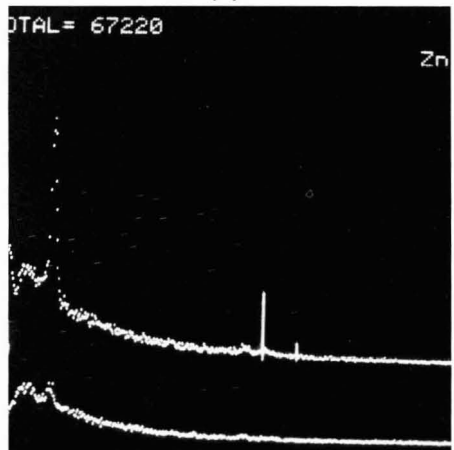


(B)

Figure 1—Scanning electron micrograph and corresponding Zn X-ray map for the panel brush-coated with the carbonate salt system (treatment 5). Arrows indicate interfacial zone between coating and substrate. Both *Figures 1a* and *1b*, 400 X

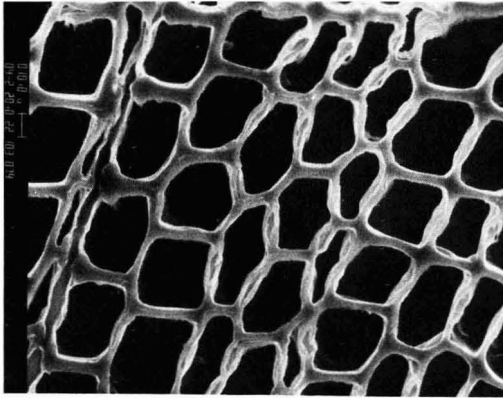


(A)

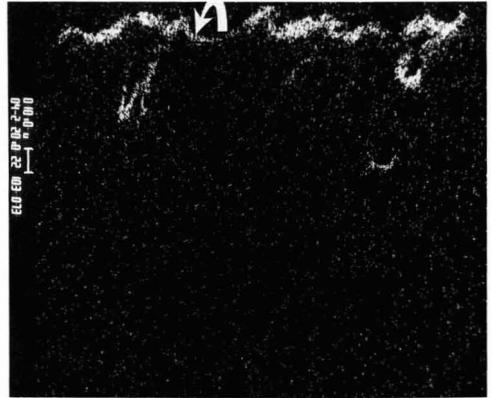


(B)

Figure 2—Zn X-ray spectra for the panels brush-coated with the carbonate and the thiocyanate salt systems before and after soaking in water for 24 hr. Vertical bars indicate location of Zn peaks. 2a—carbonate salt system; 2b—thiocyanate salt system

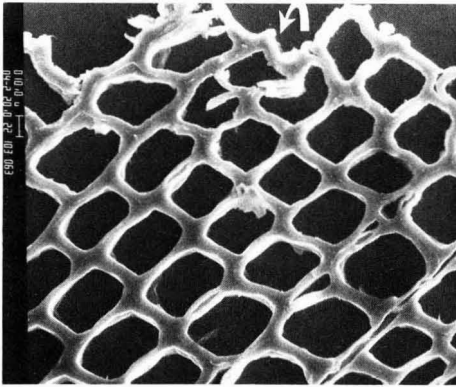


(A)

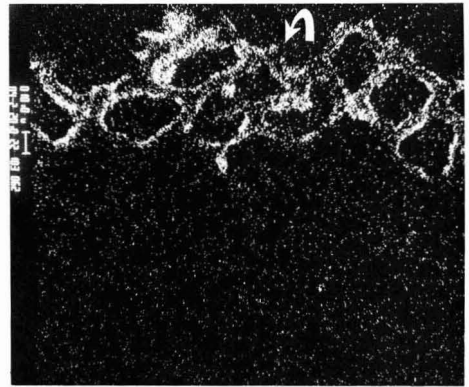


(B)

Figure 3—Scanning electron micrographs and corresponding Cl X-ray maps for panels treated with the salt-latex system. 10 sec dip time. Arrows indicate coated surface. Dots in Figure 3b indicate extent of latex penetration. Figures 3a and 3b, 500 X

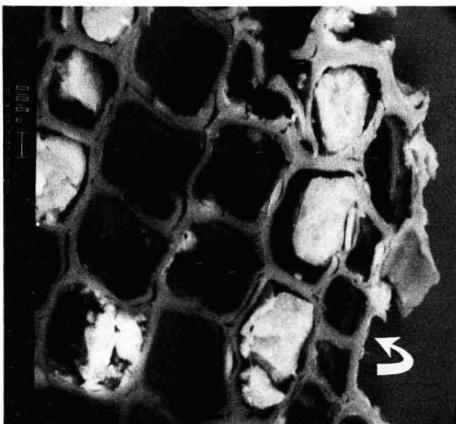


(A)

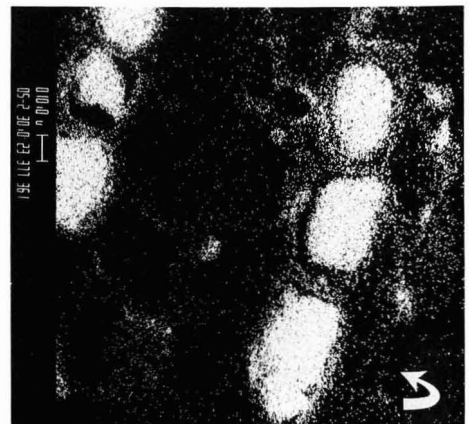


(B)

Figure 4—Scanning electron micrographs and corresponding Cl X-ray maps for panels treated with the salt-latex system. One min dip time. Arrows indicate coated surface. X-ray dots indicate extent of latex penetration. Figures 4a and 4b, 500 X



(A)



(B)

Figure 5—Scanning electron micrographs and corresponding Cl X-ray maps for panels treated with the salt-latex system. 24 hr soak time. Arrows indicate coated surface. Location of Cl X-ray dots also indicates extent of latex penetration. Figures 5a and 5b, 500 X

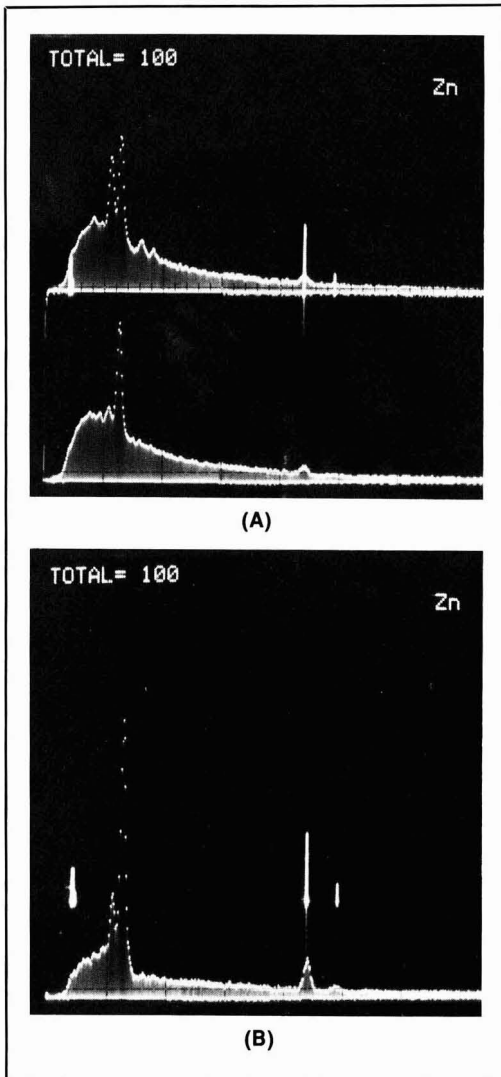


Figure 6—Zn X-ray spectra for panels treated with the salt-latex system. Vertical bars indicate Zn peaks. 6a top—10 sec dip time; bottom—1 min dip time; 6b—24 hr dip time

comparison of these figures with the corresponding micrograph and maps obtained with the brush-coated thiocyanate salt system reported in the previous study¹ reveals a difference in Zn distribution between the two salt systems. While most of the Zn is deposited at the surface with the carbonate salt system, a more even distribution with penetration of Zn into the substrate surface results with the thiocyanate salt solution. Four identically treated samples (WP-1, WP-2, WP-7, and WP-11) were analyzed and the analysis was repeated three times, at approximately three-week intervals. The wood was all of one species, eastern white pine, which was selected to avoid use of reaction wood or other obvious abnormalities.

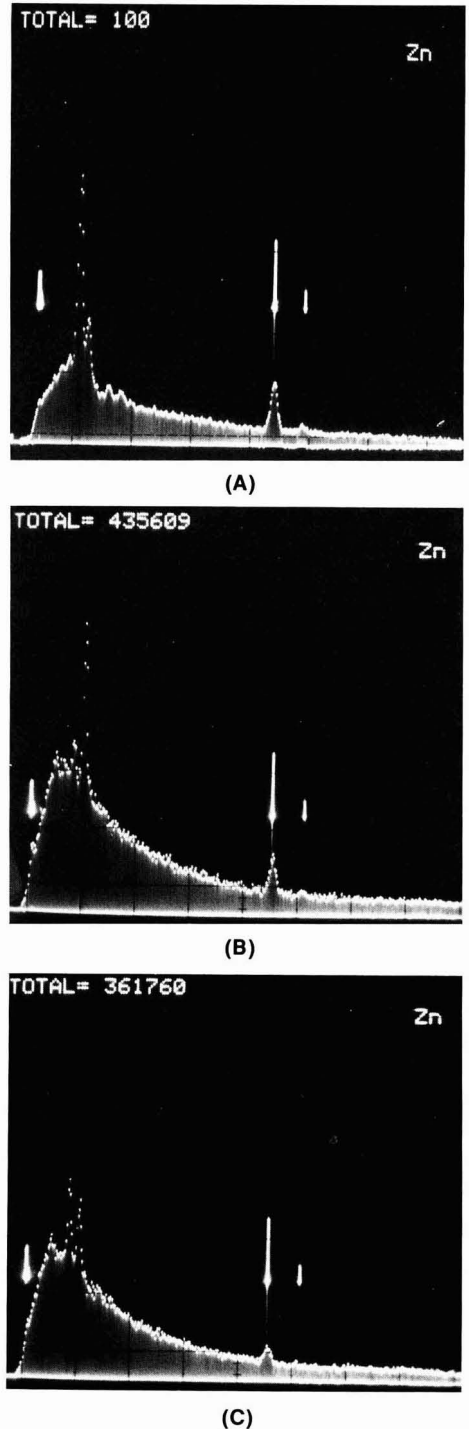


Figure 7—Zn X-ray spectra for sections from panel impregnated with the salt-latex system (treatment 4) and without 24 hr soaking. Vertical bars indicate location of Zn peaks. 7a—unleached panels; 7b—outer section leached panels; 7c—center section

The Zn X-ray spectra for the panels brush-coated with the carbonate and the thiocyanate salt systems (treatments 5 and 6), before and after soaking in water for 24 hr are displayed in *Figure 2*. Even after the 24 hr soak treatment, some Zn was still retained by the specimen from the panel treated with the carbonate salt system, while it was absent from that treated with thiocyanate salt.

The effect of time of dipping is shown in *Figures 3-6*. The scanning electron micrographs and corresponding Cl. (chlorine) X-ray maps for panels treated with the salt-latex system (treatments 1, 2, 3) indicate that, as the time of dipping was increased from 10 sec to 24 hr, the latex penetrated to an increasing extent into the substrate surface. With 24 hr soak time, considerable latex was deposited into the cell lumens. A similar effect is observed with the Zn X-ray spectra for these three panels (*Figure 6*) from the peak height and total counts over identical times. Zn X-ray spectra for sections from a panel impregnated with the salt-latex system (treatment 4) with and without 24 hr soaking are given in *Figure 7*. The area under the Zn peak decreases with the soak treatment. Further, the Zn peak is smaller for the center section of the specimen than for the outer section.

The results indicate that the thiocyanate salt system is more leachable than the carbonate salt system. This is not unexpected since both ammonium thiocyanate and zinc thiocyanate are more soluble than the corresponding carbonate salts. Zinc oxide was practically insoluble in ammonium carbonate solution alone and some ammonia was always added to dissolve it. With the ammonium thiocyanate solution, zinc oxide could be dissolved, though slowly, without ammonia. Thus, once the respective salts are deposited into the substrate and excess ammonia is lost from the system, the carbonate salt is more difficult to leach out by prolonged soaking than the thiocyanate salt.

There was a considerable time lag, ranging between five weeks to two months, between the treatment of the samples and their examination by SEM/EDXA. Hence, some aging had already taken place. However, in order to determine the effects of artificial and natural weathering, additional work will be required.

The difference between the Zn peaks from the center and the outside sections of the leached panel suggests that, as the salts are leaching out, the center of the panel is depleted first, while the outside section still contains considerable amounts of the salts. It appears that the zinc in the outer sections is more leach-resistant than the center sections. The reason for such a preferential leaching of zinc from the center section is difficult to understand at this time. Perhaps this may arise from the location of zinc in the substrate. It has been observed¹ that, although

most of the zinc is present in the cell lumen, some limited penetration of zinc in cell walls also occurs. It is quite possible that most of the zinc in the center section is present in the cell lumens and is easily leached. More detailed study will be required before a definite conclusion can be drawn from the observed results.

The results with salt-latex system are interesting. The latex, as evidenced by the Cl X-ray maps, is mainly confined to the cell wall areas for the shorter dipping times (*Figures 3-5*). However, with prolonged dipping, Cl is heavily deposited in the cell lumen. With an average particle size of latex of 220 nm, it is unlikely that it can penetrate or diffuse into cell walls. The latex particles are probably strongly adsorbed on the wall surface. With shorter dipping times, the latex pickup is just sufficient to be adsorbed. However, when excess latex is available with longer dipping times, lumen deposition is observed.

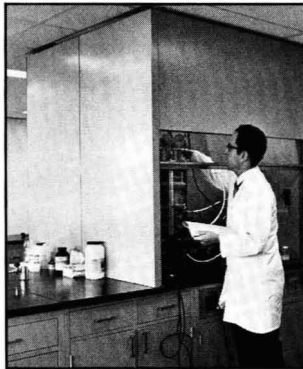
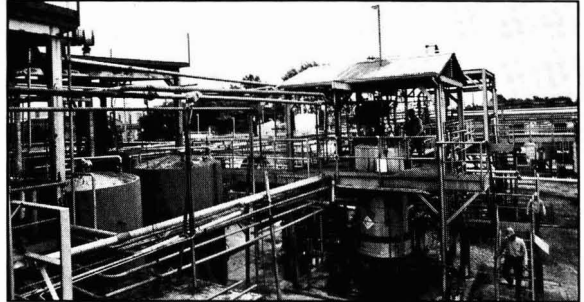
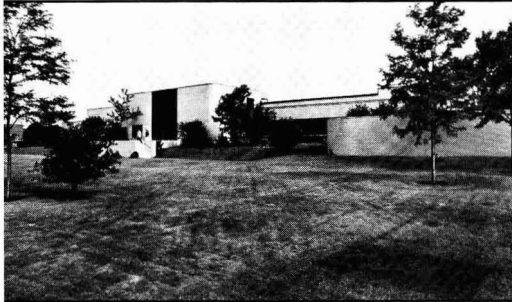
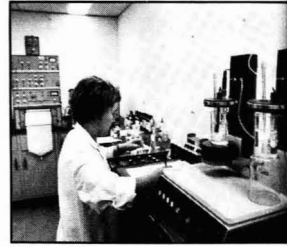
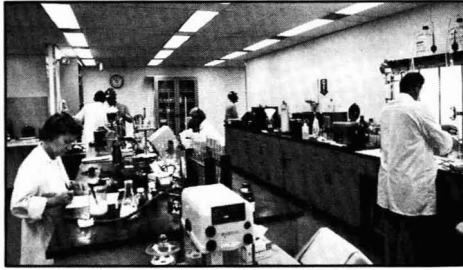
Figure 5 shows that not all cell lumens are filled; latex may diffuse through ray tracheids, or pits, or both. If latex particles could diffuse through the cell wall, then these particles would have been deposited more uniformly in the different rows of cell lumens, decreasing progressively further away from the surface. More detailed studies will be required to understand the exact pathways taken by the latex for the observed distribution in the substrate structure.

CONCLUSIONS

This study on the distribution of protective systems based on ammoniacal zinc salts reveals that the ammoniacal zinc thiocyanate system is more leachable than the corresponding carbonate system. While most of the zinc was deposited on the surface with the carbonate salt system, it penetrated the substrate surface with the thiocyanate salt system. With the zinc salt-latex system, the amount of latex penetration into the substrate increased with the time of dipping or soaking and it appeared that the latex diffused into the substrate through ray tracheids, or pits, or both.

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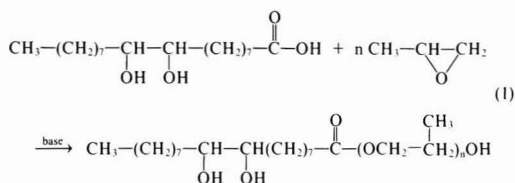
Urethane Coatings From Propoxylated Fatty Acids And Epoxidized Tallow

M. Zubillaga and E. Saggese
United States Department of Agriculture*

Threo- and *erythro*-9,10-dihydroxystearic acid and azelaic acid were reacted with varying amounts of propylene oxide under basic catalysis to form polyether polyols with hydroxyl numbers ranging from 165 to 320. The polyols were evaluated as resin components in coating formulations with three commercial polymeric isocyanates. The fatty acid-based coatings were compared with coatings obtained from three commercial polyols. The properties of the films from fatty acid-derived polyols were similar to those of the controls. Additional films were prepared with the fatty polyols derived from the propoxylated products from the reaction of epoxidized tallow with sorbitol or lactitol. This latter work expands the use of polyols derived primarily from vegetable oils to include those of animal origin.

INTRODUCTION

Previous work in this laboratory demonstrated that polyols derived from epoxidized tallow or hydroxylated fatty acids can be utilized as resin components in the preparation of low, medium, and high density polyurethane foams.¹⁻³ In other studies, hydroxylated fatty acids have been used as components in the formulation of polyester films.^{4,5} The results of the urethane foam studies suggested utilizing propoxylated fatty polyols as resin components in polyurethane coating formulations. To test this feasibility, several series of fat-based polyols were prepared for use as resin components in such formulations. Two series were prepared by reacting propylene oxide with *threo*- and *erythro*-9,10-dihydroxystearic acid to give polyols having a functionality (average number of reactive sites) of three. The reaction is summarized by equation (1), in which *n* has the average value of 3, 5, and 11.



As with simple carboxylic acids,⁶ it is considered that the acid group is consumed in the first stages of reaction with propylene oxide to form propylene glycol monoesters. With additional propylene oxide, reaction presumably continues to a larger extent at the β -oxypropyl, and to a small extent at the vicinal secondary glycol function, still disfavored by comparatively low acidities. Owing to self-alcoholysis during propoxylation, the products are more complex than diagrammed and comprise a mixture of monoesters, diesters, and free polypropylene glycols.^{7,8}

The series corresponding to the reaction of approximately 3, 5, and 11 moles of propylene oxide per mole of *threo*- and *erythro*-9,10-dihydroxystearic acids were designated as the T series and the E series, respectively. An additional series was prepared from azelaic acid to give polyols having a functionality of two. The azelaic acid series, in which about 2, 4, and 8 moles of propylene oxide were fixed, was designated as the Az series. The reaction sequence illustrated by equation (1) was also employed in preparing the azelaic acid series. In this series, the product mixture has hydroxyl units at both ends of the chain.

The polyols described above were reacted with two aromatic and one aliphatic commercial polyisocyanates. A two-package, Type V (ASTM) nonaqueous system containing either 60% or 100% nonvolatiles (NV) was used in this study. The ratio of NCO/OH equivalents used was 1.1 to 1. The solvent was a 1:1 mixture of Aromatic 100 and xylene. Another series of propoxylated polyols of functionality greater than three was obtained with esters resulting from the reaction of epoxidized tallow with sorbitol or lactitol, which were

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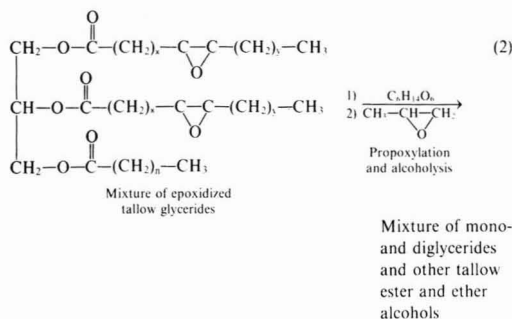
Table 1—Polyols

Polyols ^a	Hydroxyl Number	Viscosity cps 25°C
Az ₁ ^b	321.4	990
E ₁	324.4	1100
T ₁	327.1	1240
Az ₂ ^b	248.2	340
E ₂	286.1	180
T ₂	280	420
Az ₃ ^b	176.2	320
E ₃	181.1	220
T ₃	187.8	440
St ₁	517.4	23000
St ₂	356.2	3340
Lt ₁	334.2	—
Lt ₂	300.7	—
Lt ₃	270.4	3600
F 13	341.9	880
F 15	257.8	450
CO DB	175.1	770

(a) Propoxylated azelaic acid: Az₁, Az₂, Az₃; propoxylated *erythro*-9,10-dihydroxystearic acid: E₁, E₂, E₃; propoxylated *threo*-9,10-dihydroxystearic acid: T₁, T₂, T₃; propoxylated sorbitol esters of tallow fatty acids: St₁, St₂; propoxylated lactitol esters of tallow fatty acids: Lt₁, Lt₂, Lt₃; glycerol monoricinoleate: F 13; ethylene glycol monoricinoleate: F 15; Castor oil DB: CO DB.

(b) Ascending order of subscripts indicates increasing degree of propoxylation.

designated as St and Lt series, respectively. The reaction sequence is schematically shown with sorbitol by equation (2).



Under appropriate conditions of acid followed by basic catalysis, each epoxy group gave rise, by reaction with propoxylated sorbitol, to six hydroxyl groups. Under basic catalysis, the alcoholysis of glyceride linkage occurred. In addition, this alcoholysis of glyceride linkage conferred hydroxyl functionality even on the wholly saturated triglyceride. The reaction product consisted of a mixture including diglycerides, monoglycerides, fatty esters of propylated sorbitol, and sorbitol hydroxy ethers of the mixed glyceride.⁹ These polyols were evaluated as resin components in coatings to determine the effect of using fatty polyols of high functionality on resultant properties. Subscripts (1, 2, and 3) are used to show progressive increase in propoxylation in each series (Table 1).

It was anticipated that the fat-based polyols, because

of their inherent plasticizing capacity, hydrophobic character, and good thermal stability, might impart improved properties to protective coatings. This work represents a preliminary study for determining the feasibility of utilizing fatty acid-derived materials from animal fats in the urethane coating field.

EXPERIMENTAL

Materials

Threo- and *erythro*-9,10-dihydroxystearic acids were prepared from oleic acid as previously described.^{10,11} The propoxylated sorbitol and lactitol esters of tallow fatty acids were prepared in this laboratory by Bilyk and Monroe according to standard methods.⁶ The following materials were used as received from commercial suppliers: azelaic acid, propylene oxide, and the solvents xylene and Aromatic[®] 100 (Exxon Co.,* U.S.A.). The isocyanate resins[†] were Mondur[®] CB-75, Desmodur[®] N-75, and Mondur MRS (Mobay Chemical Corp.). The commercial polyols employed were Castor Oil DB (low acidity and moisture content), Flexricin[®] 13, and Flexricin 15 (NL Industries, Inc.). The flow agent and catalyst were Modaflow[®] (Monsanto Co.) and Desmorapid[®] PP (Mobay Chemical Corp.), respectively.

Propoxylated *Threo*-9,10-dihydroxystearic Acid

The propoxylated reaction used in this study was based on literature procedure.⁶ Into a 500 ml 3-neck round bottom flask, equipped with a condenser, mechanical stirrer, addition funnel, and thermometer, was placed 79.5 g (0.25 mole) *threo*-9,10-dihydroxystearic acid and 0.46 g of potassium hydroxide, 0.5% based on weight of acid. The mixture was heated to 130°C under a N₂ atmosphere, and propylene oxide (43.5 g; 0.75 mole) was added dropwise over a period of 2 hr. The temperature was then increased to 155°C for an additional hour. After cooling to ambient temperature, the mixture was dissolved in chloroform (200 ml), and the catalyst was neutralized with concentrated hydrochloric acid. The inorganic salts were filtered off and the solvent was removed on a rotary evaporator. The polyol weighed 122 g (hydroxyl number, 327.1, Table 1).

In a similar manner, a series of propoxylated *erythro*-9,10-dihydroxystearic derivatives and another of azelaic acid derivatives were prepared (Table 1).

Coating Preparation

The following technique was used to prepare all of the coatings in this study except for specific modifications as noted. The ratio of NCO/OH eq. wt. used was 1.1 to 1.

Into a 2 oz polyethylene cup was placed 5.15 g of polyol T₁ (OH eq. wt. 171.5) and 5.17 g each of dry xylene and Aromatic 100. To this was added 0.31 g of the flow agent, Modaflow (5% ethyl acetate solution, 0.1% based on total weight of resin). The polyol was mixed and

*Reference to brand or firm name does not constitute endorsement by the U.S. Dept. of Agriculture over others of a similar nature not mentioned.

†Proper precautionary measures should be exercised when handling isocyanate resins in view of their suspected toxicological properties.

thoroughly blended with 10.34 g of isocyanate resin, Mondur CB-75 (NCO eq. wt. 313.4). The mixture was allowed to stand for 1 hr before being used. Glass test tubes were dip-coated up to one-half of their length and placed vertically in a tray in an inverted position. The substrate to be coated (glass, tin, steel, and aluminum) was placed on a Howard suction plate, the polymer mixture was poured onto the upper section of the substrate and the film was applied by a Bird Applicator with a pre-fixed clearance of 3 mil. The final dry film thickness was about 1.5 mil. The test tubes and the other coated items were baked in an oven at 148°C for 20 min. After the films were cured by baking, their physical properties and chemical resistance were evaluated. The samples used for drying time evaluation were air dried at RT.

The formulations with Mondur CB-75 were also made without a flow agent. The compositions with Desmodur N-75 required the use of a catalyst (Desmorapid PP, 0.5% based on weight of the resins) and a flow agent; those with Mondur MRS were 100% N.V. and no flow additives were used.

Test Methods

The standard procedures described below were used to evaluate the physical properties and the chemical resistance of the coatings prepared.

DRYING TIME: Tack Free (set-to-touch) and Dried-hard (hard-to-touch) (Federal Test Method Standard 141, Method 4061.1).

HARDNESS: Sward Rocker Tester (American Society of Testing Method D2134-66), glass equals 100.

IMPACT FLEXIBILITY: General Electric Tester (Federal Test Method Standard 141, Method 6226).

CHEMICAL RESISTANCE: Test tubes (12 cm × 1 cm) were dip-coated and immersed one-fourth of their length in the following substances for three weeks except where noted: deionized water at room temperature, boiling water (15

Table 3—Formulations: Application and Baking Times (148°C)

Polyols	Isocyanate ^a	Formulation Type ^b	Reaction Time (Before Application)	Baking Time (After Application)
Controls ^c and experimental polyols ^d	Mondur CB-75	A	1 hr	20 min
		B	1 hr	20 min
	Desmodur N-75	B	1.5 hr	30 min
	Mondur MRS	A	15–20 min	20 min
Propoxylated sorbitol esters of tallow fatty acids	Mondur CB-75	A	10 min	20 min
		B	10 min	20 min
	Desmodur N-75	B	30 min	30 min
Propoxylated lactitol esters of tallow fatty acids	Mondur CB-75	A	10 min	20 min
		B	10 min	20 min
	Desmodur N-75	B	30 min	30 min

(a) Commercial resins (Mobay Chemical Corp.).
 (b) A—no additives; B—additives.
 (c) Castor Oil DB, glycerol monoricinoleate (F 13) and ethylene glycol monoricinoleate (F 15).
 (d) Propoxylated azelaic acid and propoxylated *three-* and *erythro-*9,10-dihydroxystearic acid.

min), 10% hydrochloric acid solution, 5% sodium hydroxide solution, and toluene. The samples were examined periodically to determine any changes. Samples which passed the chemical test were designated by a plus sign (+), whereas those that failed were designated by a minus sign (–).

RESULTS AND DISCUSSION

Coatings were prepared from five series of fat-based polyols (Table 1) and from three commercial polyols. Three of the series (Az, E, T) had hydroxyl numbers ranging between 175 and 327; the other two series (St, Lt) had hydroxyl numbers ranging between 270 and 518. For comparison, coatings were also prepared from three commercial polyols (F 13, F 15, CO DB). The control polyols were selected on the basis of their functionality (diol or triol structure) and similarity of hydroxyl numbers with the experimental polyols described herein. Shown in Table 2 are the drying times observed for coatings formulated with and without additives. In general, the drying times increased as polyols with lower hydroxyl numbers were used and were longer for the diol series than for the triol series. This same trend was noted for pot life.

The formulations with flow additive gave films with longer tack free times than those films cast without the additive, whereas hard-to-touch and pot life values were generally similar.

Table 3 summarizes the time allowed between mixing and application to substrate for polyols studied. This time was shorter for the more highly functional polyols, propoxylated sorbitol, and lactitol esters of tallow fatty acids than for the lower functional experimental polyols and controls. The extent of the reaction of the polymer

Table 2—Drying Times^a of Coatings Formulated with Mondur CB-75

	Polyol Hydroxyl Numbers					
	From 321 to 341		From 248 to 280		From 175 to 188	
	Diol ^b	Triol ^{c,d}	Diol	Triol	Diol	Triol
A (no additives)						
Tack free	0.5	0.5(3)	1.5	0.5(3)	2.5	2(3)
Hard-to-touch . . .	2.5	2(3.5)	4	2(3.5)	4	4
Pot life	24	24	48	24	1 week	24
B (additives)						
Tack free	2	2	2	2	3.5	3
Hard-to-touch . . .	3.5	2	4	2	6	4
Pot life	48	24	48	24	1 week	48

(a) Time units = hours (RT, 25°C).

(b) Az series polyols and F 15 control polyol.

(c) E and T series polyols and F 13 control polyol.

(d) Values in parentheses are differences noted for E series polyols.

Table 4—Physical Properties and Chemical Resistance of Coatings Formulated with Mondur CB-75^a

Polyol	Appearance ^b		Hardness (Glass Plates) Sward Rocker Test		Chemical Resistance ^c to 5% NAOH	
	No Additives	Modaflow ^d	No Additives	Modaflow	No Additives	Modaflow
	F 13	3	3 sn	60	60	+
Az ₁	3	3	60	40	+	+
E ₁	3	3	46	46	+	2 weeks
T ₁	3	3	50	50	+	2 weeks
F 15	4	3	60	50	+	1 week
Az ₂	4	3	58	40	+	1 week
E ₂	4	4	60	54	+	1 week
T ₂	4	4	60	54	+	1 week
CO DB	3	3 sn	40	16	+	3 weeks
Az ₃	3	3 sn	36	30	+	+
E ₃	4	3 sn	20	20	+	+
T ₃	4	3 sn	40	40	+	+

(a) Commercial resin (Moby Chemical Corp.).

(b) Numerical rating code based on ASTM D714-56: 4—very good (no signs of imperfections, cratering, blistering, etc), 3—good (some negligible imperfections), 2—fair (moderate visible imperfections), 1—poor (more pronounced imperfections), 0—bad (no film evaluation).

(c) Samples coded + showed no signs of deterioration during three weeks of observation. The chemical resistance to the following media were all (+): deionized water at room temperature, boiling water, 10% hydrochloric acid, and toluene.

(d) sn: (scratched by fingernail).

solution before application is judged by the increase in viscosity which depended on the nature of the polyol and the type of the isocyanate used, aromatic or aliphatic.

Also shown in *Table 3* is the baking time. The incorporation of additive did not make any difference in the times noted, but the isocyanate used did. When Desmodur N-75 was used instead of Mondur CB-75 the reaction time increased because the viscosity increased more slowly.

The physical and chemical properties of the films prepared from the fatty acid-derived polyols and controls reacted with Mondur CB-75 are given in *Table 4*. In general, all of the coatings cast with this polyisocyanate gave films whose appearance, hardness, impact flexi-

bility, and chemical resistance were similar to those of the controls, except that about half the films prepared with the use of a flow agent exhibited some loss in hardness and reduced chemical resistance to alkali. The experimental polyols with higher hydroxyl numbers (subseries 1) gave films with lower hardness values than the control F 13, whereas the intermediate (subseries 2) and low hydroxyl number polyols (subseries 3) had hardness values similar to or higher than those of the controls (F 15, CO DB). Films obtained with the E series of polyols had lower hardness values than those obtained from the T series polyols. It is assumed that this difference is due to the diastereomeric characteristic of the hydroxyl function between the two series.

Table 5 shows values for the same series of polyols formulated with the aliphatic polyisocyanate Desmodur N-75. Since Desmodur N-75 contains a biuret structure and is a less reactive isocyanate polymer than Mondur CB-75 (urethane structure), a catalyst was used for these formulations. Coatings prepared from the fatty acid-based polyols with high and intermediate hydroxyl numbers (subseries 1 and 2, respectively) exhibited good appearance and impact flexibility of >60% elongation. The films were soft except for the control F 13 and the polyol T₁. Drying times for the films prepared from Desmodur N-75 ranged from 2.5 to 3.0 hr, and pot life for the resin polymers ranged between 24 and 48 hr except for the E₁ polyol formulations, which had drying times as long as 72 hr.

Table 6 gives the data obtained on the coatings prepared from the polyol series reacted with the aromatic polyisocyanate Mondur MRS in a solventless system. The polyols with hydroxyl numbers in the 330 range (subseries 1) had hardness and flexibility values similar to those of the control, while their appearance could be

Table 5—Physical Properties and Chemical Resistance Of Coatings Formulated with Desmodur N-75^a

Polyol	Appearance ^b	Hardness (Glass Plates) Sward Rocker Test	Chemical Resistance ^c to 5% NAOH
F 13	2	26	1 week
Az ₁	3	6	2 weeks ^f
E ₁	3	6	2 weeks
T ₁	3	34	(+)
F 15	2	4	24 hr
Az ₂	3 ^d	4	1 week
E ₂	3	6	1 week
T ₂	3	6	1 week
Az ₃	2	2	48 hr

(a)(b)(c) See footnotes for *Table 4*.

(d) Abbreviation (t) for tacky.

(e) Polyols within this hydroxyl number range (T₁ and E₁ including the control) gave unacceptable films.(f) Az₁ in H₂O deteriorated after one week.

Table 6—Physical Properties and Chemical Resistance of Coatings Formulated with Mondur MRS^a with No Additives or Solvents

Polyols	Appearance ^b	Hardness (Glass Plates) Sward Rocker Test	Chemical Resistance ^c				
			Deionized Water Room Temp.	Boiling Water	10% HCl	5% NaOH	Toluene
F 13	2	50	+	+	+	72 hr	+
Az ₁	2	62	3 week D ^e	5 min	+	+	+
T ₁	2	50	+	+	+	+	+
F 15	2 sn ^d	16	+	+	+	+	+
Az ₂	3	14	+	+	+	+	+
E ₂	3	26	+	+	+	72 hr	+
T ₂	3	46	+	+	+	+	+
CO DB	1 t	0	+	+	+	72 hr	+
Az ₃	3 t	2	+	+	+	+	+
T ₃	1 t ^f	4	+	+	+	24 hr	+

(a)(b)(c)(d) See footnotes for Table 4.

(e) D (dew).

(f) † (precipitation).

Table 7—Physical Properties and Chemical Resistance of Coating from Propoxylated Sorbitol Esters of Tallow Fatty Acid Formulated with Mondur CB-75 and Desmodur N-75^a

Polyol	Appearance ^b	Hardness (Glass Plates) Sward Rocker Tester	% Elongation (Tin Plates) G.E. Impact Flexibility Tester	Chemical ^c Resistance To Deionized Water (Room Temp.)
St ₁ (No additives)	4	64	10	3 weeks H ^d
St ₁ (Additives)	4	64	10	+
St ₂ (No additives)	3	54	5	3 weeks H
St ₂ (Additives)	3	54	5	+
Desmodur N-75				
St ₁ (Additives)	4	52	60	+
St ₂ (Additives)	2	30	60	+

(a)(b)(c) See footnotes for Table 4.

(d) H: hazy.

Table 8—Physical Properties and Chemical Resistance of Coatings from Propoxylated Lactitol Esters Of Tallow Fatty Acids (Lt₃) Formulated with Mondur CB-75 and Desmodur N-75^a

Isocyanate	Appearance ^b	Hardness (Glass Plates) Sward Rocker Test	% Elongation (Tin Plates) G.E. Impact Flexibility Test	Chemical Resistance ^c				
				Deionized Water (Room Temp.)	Boiling Water	10% HCl	5% NaOH	Toluene
Mondur CB-75 (No additives)	3	50	20	+	+	+	+	+
Mondur CB-75 (Additives)	3	50	40	+	H ^d	+	+	+
Desmodur N-75 (Additives)	2	22	40	+	+	H	3 weeks	+

(a)(b)(c) See footnotes for Table 4.

(d) H: Hazy.

evaluated as only fair. The coating prepared from Az₁ series showed some signs of cloudiness in the boiling water test after 5 min and some vapor condensation in the water immersion test after three weeks. The polyols with hydroxyl number in the 250 range (subseries 2) gave coatings with appearances as good as and hardness values generally higher than those of the control. The polyols with hydroxyl numbers in the 180 range (subseries 3) gave poor films with Mondur MRS. The films showed some degree of tackiness after being cured and exhibited a greater wetting ability on metal than on glass. Tack free times for the azelaic acid series varied from 3 to 12 hr, hard-to-touch times from 6 to 48 hr, and pot life times from 12 to 24 hr. The latter increased with decreasing hydroxyl number of the polyol. By comparison, the E and T polyols gave films with drying times of 3 to 4 hr and pot life values of 0.5 to 1 hr.

A separate series of propoxylated sorbitol esters of epoxidized tallow (St series) was also utilized as polyol resin components. These polyols were selected from a larger series previously used in urethane foam formulations.¹² This series was formulated with the polyisocyanate Mondur CB-75 with and without the aid of a flow agent and was allowed to react for 10 min prior to application to the substrates. Pot life for the coating formulations with a flow agent was 1.5 hr, whereas that for those without a flow agent was only 20 min. All of the films cast from this series of polyols had hard-to-touch times of 20 min. The results noted for the coatings formulated from this series are given in Table 7. Films from a polyol with high hydroxyl number (St₁) had very good appearance and high hardness values but relatively low elasticity (10% elongation). The polyol with lower hydroxyl number (St₂) gave films with hardness and appearance similar to those with St₁ but only 5% elongation. The lower elasticity values for this series of films are probably due to the higher polyol functionality which results in a more highly crosslinked polymer network in the final urethane film. The chemical resistance tests were acceptable except for the films formulated with Mondur CB-75 and additives which turned hazy after immersion in water for three weeks.

The polyols in the St series, also shown in Table 7, were formulated with Desmodur N-75, with a flow agent and Desmorapid PP used as catalyst. The mixtures were allowed to stand for 30 min before application and had a pot life of 1.5 hr. Although the hardness values were somewhat reduced from those films formulated with Mondur CB-75, the elasticity was improved to >60% elongation. The films from this series had very good appearance and acceptable chemical resistance. The hardness values for the St₂ films were approximately one-half of those given by the St₁ films.

Coatings were also cast from formulations containing propoxylated lactitol esters of tallow fatty acid polyols

(Lt) and the polyisocyanates Mondur CB-75 and Desmodur N-75. In this series, however, polyols with hydroxyl numbers greater than 270 failed to produce acceptable coatings in all the formulations studied. Those polyols with a low degree of propoxylation gelled when mixed with these isocyanates. The Lt₃ polyol with hydroxyl number 270, however, did produce acceptable films when formulated with the above isocyanates (Table 8). Reaction time, drying time, and pot life for these coating formulations closely paralleled those noted for the St series of polyols. The hardness values and appearance were generally comparable to those observed for the St series of films. However, their flexibility values, when formulated with Mondur CB-75, were superior to the tallow sorbitol polyols.

CONCLUSION

It was found that the preparation of protective urethane coatings from new fat-based intermediate polyols is feasible. This study indicates a new area for the use of tallow in commercial application. The final cured coatings from tallow contained between 8.4 and 13.5% fatty material.

The criterion used in this study was such as to permit an objective determination of the practicability of making protective urethane coatings from animal fat derivatives. The physical properties and chemical resistance exhibited by the films cast from high and intermediate hydroxyl number polyol resins were, in general, acceptable.

This study indicated that the fat-based derivatives have a potential use as resin components in coating formulations. The formulations described were arbitrarily selected and not designed for any specific application.

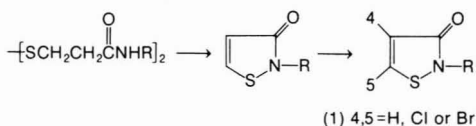
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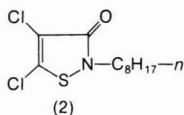
3(2H)Isothiazolone, A New Class of Antifouling Toxicant

G.A. Miller
Rohm and Haas Company*
and
T. Lovegrove
International Paint Marine Coatings†

The 3(2H)-isothiazolone (1) compounds as a general class have demonstrated high antimicrobial activity against a wide range of bacteria, fungi, and algae. The general and practical synthesis of this heterocyclic structure via amide-disulfide cyclization and subsequent halogenation have made possible rather thorough structure/activity studies in several commercially useful areas—paint film

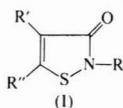


mildewcide, industrial preservative, and water treatment microbicide. The current work describes the marine antifouling properties of isothiazolones (1) as measured by membrane screening evaluations. Structure/activity investigations by this procedure show better activity for the ring halogenated derivatives, and specifically identify 4,5-dichloro-2-n-octyl-3(2H)-isothiazolone (2) as the optimum antifouling toxicant.

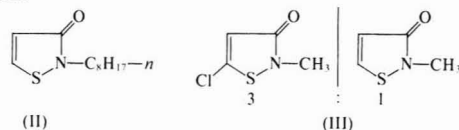


INTRODUCTION

The 3(2H)-isothiazolone heterocyclic series (I) has recently been identified as an important class of antimicrobial toxiphore. The range of structural modifi-



cation (R, R', R'') possible with (I) allows the selection of specific analogs particularly suited to individual application uses because of appropriate spectra of biological activity and pertinent physical properties. Guided by this structure-activity principle, two commercial products have developed from the 3(2H)-isothiazolone series. Compound (II), 2-n-octyl-3(2H)-isothiazolone, possessing excellent fungicidal activity and high persistence to water leaching, is a commercially important paint film mildewcide.** Similarly, the 2-methyl-3(2H)-isothiazolone (III), being a broad spectrum microbicide against bacteria, fungi, and algae, and highly soluble in water, is commercially successful as a water treatment microbicide and as an industrial preservative.** The following report summarizes recent structure-activity investigations with 3(2H)-isothiazolones as marine antifouling toxicants.



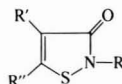
SYNTHESIS

Preliminary to a discussion of the antifouling data and structure-activity conclusions, we shall briefly consider the chemical synthesis of 3(2H)-isothiazolone (I).

Synthesis in the 3(2H)-isothiazolone series has a relatively short history in the chemical literature. In 1963, Goerdeler and Mittler¹ reported the syntheses of 5-methyl- and 5-phenyl-3-hydroxyisothiazoles (IV) as well as 2-methyl-5-phenyl-3(2H)-isothiazolone (V) by bromination of the appropriately substituted thioacylaceta-mides. Goerdeler and Keuser² reported, in 1964, the preparation of several 5-anilino-3-hydroxyisothiazoles (VI) from the respective thiomalonamide derivatives. Hatchard,³ also in 1963, described the synthesis of the disodium salt of 3-hydroxy-4-cyano-5-mercaptoisothiazole (VII) from di(sodiomer-capto)methylenemalononitrile. The first synthesis of the parent 3-hydroxyisothiazole and the 2-methyl- and 2-ethyl-3(2H)-isothiazolones

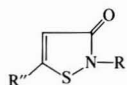
*Spring House Research Laboratories, Spring House, Pa. 19477.
†Biological Laboratory, Newton Ferrers, Plymouth, Devon, England.

**Skane® (II) and Kathon® 886 (III) are registered trademarks of the Rohm and Haas Co. for these products.

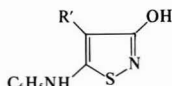
Table 1—Structure-activity Relationship; Effect of R/R' Substitution

Substituent	Number Compounds Tested	Number of Test Compounds Rated Effective	
		Algae	Barnacle
H	H	12	0
CH ₃	H	6	0
Cl	H	4	1
Br	H	8	3
H	Cl	30	13
CH ₃	Cl	36	12
Cl	Cl	31	16
Br	Br	6	3

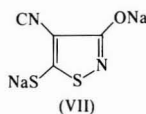
(VIII) was reported in 1964 by Crow and Leonard⁴ from the appropriate *N*-substituted *cis*-3-thiocyano- and thio-sulfatoacylamides.



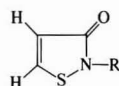
(IV), R=H, R''=CH₃, C₆H₅
 (V), R=CH₃, R''=C₆H₅



(VI)
 R'=CN, CONH₂, CO₂C₂H₅,
 COCH₃, COC₆H₅



(VII)

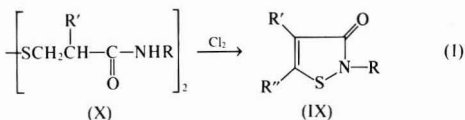


(VIII)

R=H, CH₃, C₂H₅

A very simple, convenient, and general synthesis of the 3(2H)-isothiazolone system (I) is the chlorine-induced cyclization of 3,3'-dithiodipropionamides [equation (1)]. This procedure was discovered in the Rohm and Haas Co. Research Laboratories and was reported in 1971.⁵ The major advantages of this preparative route are the generality of 3(2H)-isothiazolone structures (IX) easily accessible from it and the ready availability of 3,3'-dithiodipropionamide intermediates (X), conveniently prepared from the corresponding diacids.

By halogenation of the 3(2H)-isothiazolones (IX), a comprehensive series of halogen substituted derivatives are also available (R'=H, CH₃, Cl, Br; R''=H, Cl, Br).



R=H, C₁₋₁₈ alkyl, aryl, aralkyl
 R'=H, CH₃
 R''=H, Cl

ANTIFOULANT SCREEN

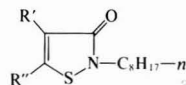
The membrane screening⁶ of 3(2H)-isothiazolones (I) was conducted by T. Lovegrove and his associates at the International Paint Co. biological testing site at Newton Ferrers, Devon, England. This screening technique is very useful in predicting commercially practical antifouling activity. The principal characteristics investigated are:

(1) Control of fouling organism, both plants and animals; and

(2) Toxicant water solubility under simulated use conditions, thus allowing preliminary estimation of the probable persistence of the chemical in paint coatings.

The membrane screen was conducted on so-called "Turtle" rafts,⁶ exposed to the marine environment of the Yealm Estuary, Devon. The screening panels on the rafts were so arranged that the trials were conducted with immersions of a test material under several different conditions. An immersion closest to the surface received maximum light to encourage weed settlement; the deepest immersion was more suitable for animal growth; and an intermediate immersion had average conditions for both types of settlement.

Each immersion panel was duplicated and usually contained 35 test materials and five standard controls. A known quantity, usually 0.3 g, of a test material was placed into a small circular plastic container whose open end was then covered with a porous membrane. From past experience, a membrane over a standard antifouling biocide, or a promising experimental one, would be essentially free of fouling over the normal 13 week immersion period. At the test completion, a visual check was taken of the container for test material loss, and this evaluation indicated the water solubility and consequent persistence of the chemical. From experience, an empty or near-empty container indicates an unacceptably high water solubility.

Table 2—2-n-Octyl-3(2H)-isothiazolone; Structure-activity Effect of R/R' Substitution

R'	R''	Algae ^a	Barnacle ^a	Aqueous Solubility ^b (ppm)
H	H	0	0	480
Br	H	0	0	50
Cl	H	++	0	60
H	Cl	++	++	70
CH ₃	Cl	++	++	40
Cl	Cl	++	++	2
Br	Cl	++	++	3
Br	Br	++	0	2
Control ^c		++	++	4-6 ^d

(a) Activity: (++) Effective; (+) Some; (0) None.

(b) Solubility measured at 25°C by ultraviolet absorption analysis.

(c) Tributyltin fluoride and phenoxarsine 10-oxide were employed.

(d) Approximate solubility based on the toxicity of a saturated aqueous solution against *Enteromorpha* spores.

From this screening evaluation, the basic potential of a test compound as an antifoulant can be assessed. Three degrees of activity are recognized:

- (1) Effective—prevents major fouling organisms from settling and/or persisting;
- (2) Some—allows organisms to settle but in considerably reduced quantity compared to the non-toxic control; algae may be stunted; and
- (3) None—settlements are very similar to those of the non-toxic control.

As follow-up to the membrane screen, laboratory evaluations were conducted with the 3(2H)-isothiazolones (I) which had shown promise as weed antifoulants. These tests determined the LC_{50} against *Enteromorpha* spores, and allowed a more precise assessment of algacidal activity and consequent weed antifouling potential, as well as solubility in seawater.

STRUCTURE-ACTIVITY EFFECT

By use of the membrane screen and laboratory follow-up, chemicals can be classified according to degree of antifoulant potential as well as weed and barnacle selectivity. This ability to demonstrate the bioactivity in relation to chemical structure was well demonstrated with the 3(2H)-isothiazolone (I) and provided some interesting structure-activity conclusions.

Substituent R'/R''

The effect of substitution on the isothiazolone ring at R' and R'' can be generalized, as shown in Table 1. The activating influence of halogenation is strong and one of the dominating structural modifications.

The activating effect of halogen substitution at R'/R'' in the particular case of the 2-n-octyl-3(2H)-isothiazolone series is apparent, as summarized in Table 2. The aqueous solubilities also shown give a preliminary estimate of the likely effective persistence of the chemicals; the commercial controls employed in these tests have

Table 3—2-Benzyl and 2-phenyl-3(2H)-isothiazolones; Structure-activity Effect of R'/R'' Substitution

R	Substituent		Algae ^a	Barnacle ^a	Aqueous Solubility ^b (ppm)
	R'	R''			
-CH ₂ C ₆ H ₅	H	H	0	0	—
	H	Cl	+	++	390
	CH ₃	Cl	++	+	100
	Cl	Cl	++	++	50
-C ₆ H ₅	H	H	0	0	—
	H	Cl	++	+	180
	CH ₃	Cl	++	+	200
	Cl	Cl	++	++	10

(a) Activity: (++) Effective; (+) Some; (0) None.
(b) Solubility measured at 25°C by ultraviolet absorption analysis.

Table 4—2-n-Alkyl-4,5-dichloro-3(2H)-isothiazolone; Structure-activity Effect of n-alkyl Chain Length

Alkyl(n), C _x	Substituent		Aqueous Solubility ^b (ppm)
	Algae ^a	Barnacle ^a	
X = 4	++	0	1000
6	++	++	120
8	++	++	2
10	++	0	1
12	0	+	1
14	0	0	1

(a) Activity: (++) Effective; (+) Some; (0) None.
(b) Solubility measured at 25°C by ultraviolet absorption analysis.

aqueous solubility <10 ppm. This information, along with the biological activity, indicate the Cl/Cl and Br/Cl analogs as preferred antifoulant candidates in this series.

The activating influence of halogen substitution applies similarly to other series of 3(2H)-isothiazolone derivatives. The benzyl and phenyl analogs are shown in Table 3. In both series, the Cl/Cl analogs are preferred on the basis of bioactivity and solubility.

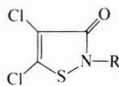
Substituent R

The substituent R in the 3(2H)-isothiazolone series (I) was extensively varied. In view of the biological activation and the more suitable solubility of 4,5-dichloro analogs (R'/R''=Cl), this substitution is preferentially treated here.

Table 5—4,5-Dichloro-2-octyl-3(2H)-isothiazolone; Structure-activity Effect of Octyl Group Branching

Octyl	Substituent		Aqueous Solubility ^b (ppm)
	Algae ^a	Barnacle ^a	
-C ₈ H ₁₇ (n)	++	++	2
-CH(CH ₂) ₂ CH ₃ CH ₃	+	++	50
-CH ₂ CHC ₄ H ₉ (n) C ₂ H ₅	++	++	30
-CHCH ₂ CHC ₂ H ₅ C ₂ H ₅ CH ₃	0	0	90
-CCH ₂ C(CH ₃) ₂ (CH ₃) ₂	++	+	40

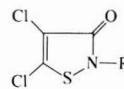
(a) Activity: (++) Effective; (+) Some; (0) None.
(b) Solubility measured at 25°C by ultraviolet absorption analysis.

**Table 6—4,5-Dichloro-3(2H)-isothiazolone;
Structure-activity Effect of Nitrogen Substitution**

R	Algae ^a	Barnacle ^a	Aqueous Solubility (ppm)
C ₈ H ₁₇ (n)	++	++	2
C ₆ H ₅	++	++	10
CH ₂ C ₆ H ₅	++	++	50
CH(CH ₃)C ₆ H ₅	+	++	50
CH ₂ CH ₂ C ₆ H ₅	++	0	20

(a) Activity: (++) Effective; (+) Some; (0) None.

(b) Solubility measured at 25°C by ultraviolet absorption analysis.

**Table 8—3(2H)-Isothiazolone Candidate
Antifoulant Toxicants**

R	LC ₅₀ ^a (ppm × 10)	Barnacle ^b	Aqueous Solubility ^c (ppm)
C ₈ H ₁₇ (n)	1	++	2
CH ₂ CH(C ₂ H ₅)C ₆ H ₅ (n)	2	++	30
C ₆ H ₄ Cl(4)	6	0	3
CH ₂ CH ₂ C ₆ H ₅	10	0	20
C ₆ H ₅	30	++	10
C ₁₀ H ₂₁ (n)	30	0	1

(a) Determined with *Enteromorpha* spores.

(b) Activity: (++) Effective; (+) Some; (0) None.

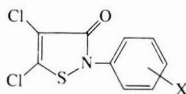
(c) Solubility measured at 25°C by ultraviolet absorption analysis.

Variation of the length and branching of an alkyl group at R has a pronounced effect on biological activity, as well as on solubility. The length of a straight chain alkyl group (Table 4) has a significant effect on aqueous solubility. In this regard, the preferred carbon chain length is eight or greater. In the alkyl(n) series, the combination of bioactivity and solubility indicates octyl(n) as the preferred antifoulant candidate.

The structure-activity effect of octyl group branching is rather unpredictable (Table 5). The straight chain octyl(n) and the 2-ethylhexyl analogs have preferred bioactivity; the former of these has the more suitable aqueous solubility (2 vs. 30 ppm).

Various aryl substituents are compared to octyl(n) in Table 6. High bioactivity was shown by this group, with phenyl and benzyl being the preferred aryl groups, comparable to octyl(n). On the basis of aqueous solubility, phenyl and octyl(n) are the preferred candidates.

Substitution on phenyl has a strong deactivating effect on bioactivity (Table 7). Of this series, only phenyl and 4-chlorophenyl were rated effective. The solubilities of both are in the range of the commercial controls (<10 ppm).

**Table 7—4,5-Dichloro-2-phenyl-3(2H)-isothiazolone;
Structure-activity Effect of Substituted Phenyl**

X	Algae ^a	Barnacle ^a	Aqueous Solubility ^b (ppm)
H	++	++	10
4-Cl	++	0	3
3-Cl	+	0	—
4-C ₂ H ₅ (n)	0	+	—
4-NO ₂	0	0	—
3,4-Cl	0	0	—
3-Cl, 4-CH ₃	0	0	—

(a) Activity: (++) Effective; (+) Some; (0) None.

(b) Solubility measured at 25°C by ultraviolet absorption analysis.

ANTIFOULANT CANDIDATES

The synthetic information and the structure-activity effects observed have led to the selection of certain 3(2H)-isothiazolones as the preferred antifoulant candidates (Table 8). Since the activity (LC₅₀) against *Enteromorpha* spores, a laboratory evaluation, is a more precise assessment of marine algae control than possible with the membrane screen, this data is presented for the selected analogs. The membrane screening results and laboratory follow-up evaluations indicate the octyl(n) derivative as the favored 3(2H)-isothiazolone for the marine antifoulant application. Field evaluations, in actual paint formulations and on ships in service, conducted by International Paint Co., have confirmed this selection, and 4,5-dichloro-2-n-octyl-3(2H)-isothiazolone⁷ is undergoing commercial development.

SUMMARY

Evaluation as antifoulant toxicants by use of a membrane screening technique has identified the 3(2H)-isothiazolone class of compound as a potential source of toxiphore against marine fouling organism. A wide range of structural modifications, involving systematic substitution on the isothiazolone ring at the 4 and 5-carbons and on nitrogen, was studied. This investigation has shown 4,5-dichloro-2-n-octyl-3(2H)-isothiazolone to be the preferred antifoulant candidate, having high biological activity, especially against algae, and water solubility in the practical range (2 ppm).

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Chelation As a Means of Developing Crosslinking Emulsions

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Chelate-containing emulsions which crosslinked through the use of multivalent metal ions were investigated using 2-acetoacetoxypropyl methacrylate (2-AAPMA) as the functional monomer. The emulsions were prepared in adequate yields and with excellent tolerance to the addition of electrolytes through the use of a nonionic/anionic blend of surfactants.

The crosslinking ability of poly (2-AAPMA) was substantiated by torsional braid analysis which indicated that coatings containing $ZnCl_2$ cured at room temperature and after thermal treatment. Subsequently, the effectiveness of such curing agents as $MgCl_2$, $NiCl_2$, $Al_2(SO_4)_3$, $CoCl_2$, $ZnCl_2$, $Zn(OAc)_2$, and $SnCl_2$ was evaluated using conventional physical testing of both latex and solvent-cast films of the same polymer.

Among the various crosslinking agents, $Zn(OAc)_2$ at a stoichiometric level of 20% provided the most preeminent results at room temperature, yielding latex-cast coatings with excellent hardness, gloss, solvent and water resistance, and tensile strength. After thermal curing at $125^\circ C$, latex coatings containing higher levels of $ZnCl_2$ demonstrated very efficient crosslink densities, undergoing virtually no solvent softening after 500 dimethylformamide double rubs.

Unexpectedly, the physical properties of the emulsion systems were superior to that of the analogously cured, solvent-cast systems.

required in typical, light to moderate demand, industrial applications. While several functional monomers and polymer classes have demonstrated varying degrees of acceptability for such applications,¹ perhaps one of the most versatile and commercially successful approaches for the development of crosslinkable emulsions has been the utilization of systems containing carboxylic acid functionality.

While carboxylic acid-containing emulsions are usually combined with such external crosslinking agents as melamine² or epoxy resins,³ useful products have also been developed by crosslinking the carboxylic acid functionality with various multivalent metal ions. For example, coatings of a 95/5-ethyl acrylate/acrylic acid copolymer emulsion crosslinked with $Cr(OAc)_3$ demonstrated a tensile strength of 96 Kg/cm^2 and an elongation of 330% while coatings of the same emulsion which did not contain a metal ion crosslinking agent demonstrated a tensile strength of 3.7 Kg/cm^2 and an elongation of 1370%.⁴

Because the carboxylate-metal bond of ionically-cured systems is particularly susceptible to displacement by nucleophilic agents, coatings crosslinked by means of carboxylate-metal bonds may be converted into the uncrosslinked form by immersion in dilute alkali.⁵ However, by replacing the carboxylic acid groups with appropriate chelating substituents, an ionically cross-linkable system results which inherently possesses greater bond integrity since the crosslinked segments incorporate the ionic species in a more thermodynamically stable, five- or six-membered ring structure.

Ionic Crosslinking by Means of Chelation

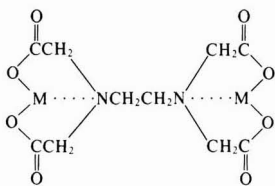
The term chelate refers to those cyclic structures which arise from the union of metal ions with certain organic groups (ligands). Typical examples of chelate compounds include hemin, an iron chelate; chlorophyll, a magnesium chelate; copper glycine; and metal complexes of ethylenediaminetetraacetic acid (EDTA) which forms multiple fused, five-membered, chelate rings, i.e.,

INTRODUCTION

Carboxyl-Functional, Crosslinkable Emulsions

Over the past two decades numerous approaches have been investigated by the coatings industry in order to develop crosslinkable emulsions which possess the application, curing, and general performance characteristics

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EDTA-metal complexes display excellent stability, and neither the copper II or nickel compound is dissociated by sodium or ammonium hydroxide.⁶ Also, the oxalate ion will not remove chelated calcium from the EDTA complex.

Even though chelate stability is usually described in terms of both kinetic and thermodynamic models,⁷⁻¹⁰ the integrity of the chelate species can be further illustrated by comparing the bond energies of various metal-ligand bonds with the bond energies of homopolar and heteropolar atoms. For example, the metal-ligand bond energies for nickel II and magnesium II acetylacetonate complexes are 71.8 Kcal/mole and 69.7 Kcal/mole, respectively, while carbon-nitrogen and carbon-sulfur bond energies are 62 Kcal/mole and 65 Kcal/mole, respectively. The integrity of the chelate bond is also indirectly observable from thermal stability measurements as illustrated by the beryllium acetylacetonate complex which boils without decomposition at 270°C at atmospheric pressure. In light of these few examples, it is ostensible that chelate-functional compounds may be employed to yield ionically crosslinked systems whose bond integrity is competitive with that found in conventional thermosetting systems.

Selection of a Chelating-Type Acrylate Monomer

As the unassociated, chelating ligands undergo transformation into cyclic structures through the combination with metal ions, a profound change takes place in many of the physical and chemical characteristics of the parent polymer. Upon chelation many polymers form insoluble, infusible powders of limited tractability. Apparently, the relatively subtle properties displayed by the organic portion of the system are masked by the more dominant properties associated with the chelate structure. Thus, selecting a chelate compound with the proper characteristics was recognized as being one of the major factors controlling the development of a viable system with acceptable coatings properties.

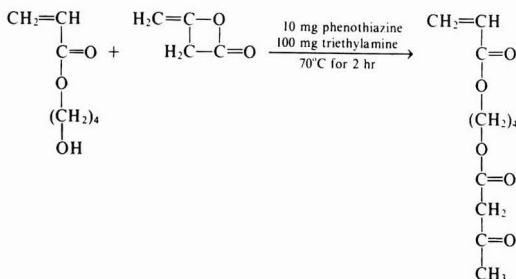
While innumerable chelate compounds have been described in the literature, derivatives of acetoacetic acid represent, perhaps, the most common and effective chelating agents for enhancing the physical properties of coatings based upon vinyl and acrylic resins.¹¹⁻¹³ For example, the addition of an acetoacetate aluminum isopropylate complex to an acrylate resin yielded cold-setting coatings with improved weather and chemical resistance¹⁴ while the addition of a similar chelating agent to epoxy and phenolic resins yielded coatings with improved mechanical properties together with increased resistance to steam and chemicals.¹⁵

Besides augmenting the physical and chemical properties of various types of polymers, a supplementary advantage for basing the crosslinking reaction on the coordi-

nating abilities of acetoacetate derivatives lies in the abundance of empirical information available on the physical and chemical nature of this chelating agent.

The selection of an olefinic-type monomer containing an acetoacetate substituent was predicated upon satisfying two requirements: (1) the derivative must be amenable to emulsion polymerization which implies that the monomer must possess low water solubility, display no tendency to complex with the surfactant, possess bonds which are stable towards hydrolysis, display low chain transfer ability, and possess suitable reactivity ratios for copolymerization with acrylates; and (2) the resulting polymer should be soluble in common solvents and display acceptable coatings properties. In light of these criteria and after consideration of the properties of various monomeric derivatives reported in the literature, the acetoacetate esters of hydroxyalkyl methacrylates were selected as the most suitable candidates for the investigation.

4-acetoacetoxybutyl acrylate has been synthesized in 96% yield by condensing one mole of 4-hydroxybutyl acrylate with one mole of diketene,¹⁶ i.e.,



However, because of a gelation problem which occurred during the later stages of the condensation reaction, the reported procedure was modified by substituting 5000 ppm hydroquinone for 10 mg phenothiazine and by reducing the reaction temperature from 70° to 55°C. As a result of these modifications both 2-acetoacetoxyethyl methacrylate (2-AAEMA) and 2-acetoacetoxypropyl methacrylate (2-AAPMA) were prepared in excellent yields.

Unfortunately, preliminary attempts to polymerize 2-AAEMA through conventional free radical methods resulted in the formation of gelled polymer; presumably because hydroxyethyl methacrylate, the parent hydroxyl substrate, contained high concentrations of a divinyl-functional contaminant. Since hydroxypropyl methacrylate contained less of the divinyl-functional contaminant, premature gelation did not occur during the polymerization of the 2-acetoacetoxypropyl derivative. Therefore, 2-AAPMA was the sole derivative used to prepare the model emulsions described in all of the ensuing studies.

EXPERIMENTAL

Preparation of 2-Acetoacetoxypropyl Methacrylate

A 5000 ml, 4 neck flask was fitted with a nitrogen gas inlet tube, thermometer, agitator, and condenser. To remove condensed water the apparatus was flame dried

while purging with nitrogen gas. Upon cooling, the reactor was charged with 2976 g of 94% hydroxypropyl methacrylate, 1.96 g, distilled triethylamine, and 11.3086 g purified hydroquinone. These ingredients were heated to 55°C with agitation and under a nitrogen gas blanket. Upon reaching the 55°C reaction temperature, 1645 g of 97% diketene were added to the reaction mixture from a mixing funnel. The addition time for the diketene was 2.33 hr, and the rate of addition was such that the flask temperature never exceeded 57°C. After the diketene addition was complete, the 55°C reaction temperature was maintained for an additional 4.25 hr. Throughout this latter period, the extent of alkylacetoacetate ester formation was determined through infrared spectroscopy by observing the disappearances of the OH band at 3550 cm^{-1} and the diketene doublet at 1875 and 1910 cm^{-1} . When unreacted diketene was no longer detected, the monomer was cooled to room temperature and removed from the flask. Elemental analysis of 2-acetoacetoxypropyl methacrylate resulted in excellent agreement between the experimental and theoretical values while the experimental boiling point of 115° to 122°C at 0.5 mm was slightly higher than the reported boiling point of 110° to 111°C at 0.45 mm.¹⁷

Emulsion Polymerization Of 2-acetoacetoxypropyl Methacrylate

PREPARATION OF LATEX A (Table 4): A 3000 ml, 4 neck flask was fitted with nitrogen inlet tubes, thermometer, agitator, and condenser. The reactor was charged with 1200 g deionized water, and the system was deoxygenated by heating and sparging with nitrogen gas. After sparging for 5 min at 97°C, the water was cooled to 80°C and 36.0 g Gafac® RE-960 were added to the slowly agitated, degassed water. This mixture was agitated for 10 min whereupon 51.4 g of Triton® X-405 (70% active) were added to the flask. The pH of this surfactant/water mixture was 0.73. After 10 min of agitation, the pH was adjusted to 7.36 by the addition of 16.2 g of 28% ammonium hydroxide. After 10 min of agitation, 1200 g of 2-acetoacetoxypropyl methacrylate were added to the surfactant/water mixture, and the agitation was increased to 240 rpm to promote emulsification. Since the addition of the monomer reduced the flask temperature to 62°C, the reaction mixture was reheated to 80° before 2.4 g of ammonium persulfate were added to the pre-emulsion. After the addition of the initiator, the agitated reaction mixture was maintained at 80°C for six hours. The resulting emulsion was cooled to room temperature and filtered. The emulsion displayed the following properties: theoretical solids—50.85; % conversion—91.96; % coagulum—2.85; and resulting pH—5.53.

PREPARATION OF LATEX B (Table 4): A 1000 ml, 4 neck flask was fitted with nitrogen inlet tubes, thermometer, agitator, and condenser. The reactor was charged with 400 g deionized water, and the system was deoxygenated by heating and sparging with nitrogen gas. After sparging

for 5 min at 97°C, the water was cooled to 80°C and 40 g of Alipal®EP-120 (30% active) were added to the slowly agitated, degassed water. This mixture was agitated for 10 min whereupon 17 g of Triton X-405 (70% active) were added to the flask. The pH of this surfactant/water mixture was 5.90. After 10 min of agitation, the pH was adjusted to 7.30 by the addition of 1.5 g of 28% ammonium hydroxide. After 10 min of agitation, 400 g of 2-acetoacetoxypropyl methacrylate were added to the surfactant/water mixture, and the agitation was increased to 240 rpm to promote emulsification. Since the addition of the monomer reduced the flask temperature to 67°C, the reaction mixture was reheated to 80°C before 0.8 g ammonium persulfate were added to the pre-emulsion. After the addition of the initiator, the agitated reaction mixture was maintained at 80°C for six hours. The resulting emulsion was cooled to room temperature and filtered. The latex was free of coagulum and displayed a 71.3% conversion. To increase the percent conversion, 772.5 g latex were reinitiated by 0.219 g ammonium persulfate dissolved in 10 g deionized water. The post-initiated latex was heated at 80°C for nine hours. The latex was then cooled to room temperature and filtered. The emulsion displayed the following properties: theoretical solids—48.78; % conversion—81.11; % coagulum—2.85; and resulting pH—5.53.

Torsional Braid Analysis

The torsional braid analyzer used an 8 in. glass multifilament rod of 0.03 in. diameter.

Paint Tests

Crosshatch adhesion, Sward rocker hardness, pencil hardness, specular gloss (60°), solvent resistance, and mandrel flexibility were conducted according to standard procedures reported in the Paint Testing Manual, 15th Ed., G.G. Sward, Editor, ASTM Technical Publication 500, 1972. Tensile strength and % elongation were measured with an Instron Tester, Model 1130, using a crosshead speed of 5 cm/min.

RESULTS AND DISCUSSION

Determination of the Crosslinking Ability Of 2-acetoacetoxyalkyl Methacrylate

Since the development of a successful thermosetting emulsion was dependent upon the coordinating ability of the chelating agent, the crosslinking potential of the polymer was investigated before proceeding with the synthesis of model emulsions containing 2-AAPMA. While infrared spectroscopy or differential thermal analysis can provide some insight into the degree of crosslinking, recent results from dynamic mechanical studies suggest that the changes which occur in glass transition temperature (T_g) and shear modulus as a result of crosslinking can be determined semi-quantitatively by torsional braid analysis (TBA).¹⁸⁻²¹ Thus, in accord with the trends established from other crosslinkable systems, the degree of chelation was expected to be reflected by a

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Table 1—Torsional Braid Analysis Results^a

Polymer Composition ^b	Crosslinking Agent ^c %	Curing Profile ^d	Tg, °C	Slope of Rigidity at Tg
80/20 MMA/2-AAEMA	none	1	87	2.250
80/20 MMA/2-AAEMA	100% with ZnCl ₂	1	118	0.645
80/20 MMA/2-AAEMA	100% with ZnCl ₂	2	117	0.940
2-AAPMA	none	1	2	3.330
2-AAPMA	none	2	40	1.720
2-AAPMA	50% with ZnCl ₂	1	42	0.516
2-AAPMA	50% with ZnCl ₂	2	140 to 156	0.107
2-AAPMA	100% with ZnCl ₂	1	116	0.500
2-AAPMA	100% with ZnCl ₂	2	151	0.214
2-AAPMA	100% with ZnCl ₂	3	158	0.187

(a) For an in-depth discussion of the principles of torsional braid analysis with respect to crosslinkable systems, see Gillham, J.K., "Torsional Braid Analysis of Polymers: A Review," U.S. Nat. Tech. Inform. Series, AD Rep. No. 759412, NTIS, 1973; *Chem. Abstr.* 79:67142x.

(b) MMA = methyl methacrylate; 2-AAEMA = 2-acetoacetoxyethyl methacrylate; 2-AAPMA = 2-acetoacetoxypropyl methacrylate.

(c) Curing agent concentration represents that percentage of the total amount of acetoacetate groups available for crosslinking with the metal ion.

(d) Curing Conditions: 1 = vacuum dried 24 hr at 23°C; 2 = stoved 4 hr at 125°C; and 3 = stoved 24 hr at 125°C.

relative increase in Tg and modulus as the crosslinking reaction progressed.

The crosslinking study utilized solution coatings of poly(2-AAPMA) and an 80/20—methyl methacrylate/2-AAEMA copolymer containing either 50 or 100% of the total concentration of ZnCl₂ required to combine with all of the available acetoacetate groups. The samples were vacuum dried for 24 hr at 23°C or thermally cured for 4 hr at 125°C. The results from the torsional braid analysis are presented in terms of Tg and slopes of rigidity at Tg. A

Table 3—Anionic/Nonionic Surfactant Study for the Emulsion Polymerization of 2-acetoacetoxypropyl Methacrylate

Surfactant Mixture ^a	Solids Content (%)	Yield (%)	Coagulum (%)	Initial pH ^b	Salt Tolerance ^c
Gafac RE-610 Triton X-405	34.19	88.5	1.7	7.45	>1.42
Gafac RE-960 Triton X-405	34.15	89.5	0.8	7.30	>1.42
Alipal EP-120 Triton X-405	33.74	66.2	8.3	5.60	>1.42
Gafac RE-960 Triton X-405	50.41	91.9	2.8	7.34	>1.42
Alipal EP-120 Triton X-405	48.88	67.6	0.5	5.40	>1.42
Alipal EP-120 Igepal CO-997	48.86	67.6	0.0	5.30	>1.42
Alipal EP-120 Triton X-405	50.17	—	100.0	7.34	—
Gelvatol 20-60	—	—	—	—	—
Alipal EP-120 Triton X-405	49.13	86.6	8.5	7.31	>1.42

(a) Triton X-405 = octylphenol polyether alcohol containing 40 moles of ethylene oxide. Igepal CO-997 = nonylphenoxy poly(oxyethylene) ethanol.

Gelvatol 20-60 = medium viscosity poly(vinyl alcohol) protective colloid.

(b) Initial pH = pH of reaction mixture at time of initiation.

(c) Salt Tolerance = millimoles of CaCl₂/g latex solids.

lower value in the slope of the rigidity is indicative of a greater degree of crosslinking since a more tightly bound system tends to display a lower rate in the change of the rigidity with temperature. The results of these studies are presented in Table 1. From these data the following conclusions were drawn:

(1) At a 100% stoichiometric concentration of ZnCl₂, the 80/20-methyl methacrylate/2-AAEMA copolymer displayed a 30°C increase in Tg and a 58 to 72% reduction in the rate of change of the rigidity with temperature. Little difference could be discerned between the room temperature dried sample and the stoved sample.

Table 2—Anionic Surfactant Study for the Emulsion Polymerization Of 2-acetoacetoxypropyl Methacrylate

Surfactant	Composition	Solids Content (%)	n-dodecyl mercaptan (%)	Yield (%)	Coagulum (%)	Salt Tolerance ^a
Triton 770	Sodium alkylaryl polyether sulfate	32.89	0.0	81.8	8.6	0.045
Triton X-200	Sodium alkylaryl polyether sulfonate	33.23	0.0	80.6	4.7	0.108
DuPont WAQE	Sodium dodecyl sulfate	33.19	1.0	78.5	3.6	0.068
Aerosol® 22	Tetrasodium N-(1,2-dicarboxyethyl) N-octadecylsulfosuccinamate	33.34	0.6	33.3	43.0	0.133
Triton GR-7	Dioctyl sodium sulfosuccinate	33.62	0.6	60.0	0.0	0.136
Gafac RE-610	Anionic phosphate ester of poly(oxyethylene) alcohols	33.66	0.6	90.0	2.9	0.074
Gafac RE-960	Anionic phosphate ester of poly(oxyethylene) alcohols	33.66	0.6	91.0	2.1	0.128
Alipal EP-120	NH ₄ salt of sulfated alkylphenoxypoly(oxyethylene) ethanol	33.43	0.6	65.3	8.5	>1.42

(a) Salt Tolerance = millimoles of CaCl₂/g latex solids. Aerosol is a registered trademark of American Cyanamide Co.

(2) After room temperature drying and at a 50% stoichiometric concentration of $ZnCl_2$, poly(2-AAPMA) displayed a 40°C increase in T_g and an 85% reduction in the slope of the rigidity. An equivalent sample containing 100% of the stoichiometric concentration of $ZnCl_2$ displayed a 114°C increase in T_g and an 85% reduction in the slope of the rigidity.

(3) After thermal curing and at a 50% stoichiometric concentration of $ZnCl_2$, poly(2-AAPMA) displayed a 100° to 116°C increase in T_g and a 94% reduction in the slope of the rigidity. An equivalent sample containing 100% of the stoichiometric concentration of $ZnCl_2$ displayed a 109°C increase in T_g and an 88% reduction in the slope of the rigidity.

(4) Thermally curing the sample beyond 4 hr at 125°C produced no discernible change in properties.

The results from the dynamic mechanical study indicated that poly(2-AAPMA) was a suitable candidate for the investigation of both room temperature cured and thermally cured emulsions. At 100% of the stoichiometric amount of curing agent, no significant differences could be discerned in T_g or rigidity increases between the room temperature dried samples and the thermally cured samples.

Emulsion Polymerization Of 2-Acetoacetoxypropyl Methacrylate

In planning the development of a viable chelating emulsion, priority consideration was given to optimizing those parameters which reduced the foreseeable impasses, thereby enhancing the possibilities of success. Thus, initial emphasis was placed on developing emulsions which displayed high degrees of conversion, low agglomerate levels, and high tolerance to the addition of various salts.

Since the emulsifier was the major constituent which influenced most of these parameters, a study was conducted to determine the most auspicious surfactant for the emulsion polymerization of 2-AAPMA. The polymerizations utilized a batch monomer addition technique, 2% of each surfactant, 0.2% initiator, 80°C reaction temperature, and a six hour reaction time. The results of this study are presented in *Table 2*.

Of the surfactants which were surveyed, the anionic phosphate esters of poly(oxyethylene) alcohols, Gafac RE-610 and Gafac RE-960, provided the greatest degree of conversion and lowest coagulum formation. These same surfactants are employed for the synthesis of high-yield, coagulum-free emulsions of vinyl acetate. Since particle surfaces of poly(vinyl acetate) and poly(2-AAPMA) are hydrophilic in nature, it is possible that some preferential interaction occurs between these particular surfactants and particle surfaces displaying a sphere of hydration. While the surfactant manufacturer indicates excellent electrolytic compatibility for these products, neither surfactant would impart adequate salt tolerance to the latex.

Alipal EP-120, the ammonium salt of a sulfated alkylpenoxy poly(oxyethylene)ethanol, imparted excellent tolerance to the addition of $CaCl_2$. However, the resulting emulsion displayed a reduced yield and an

Table 4—Chelating Emulsions Suitable for Evaluation

Latex Code	AAPMA ^a (%)	Gafac RE-960 (%)	Alipal EP-120 (%)	Triton X-405 (%)	Solids Content (%)	Yield (%)	Relative Particle Size	Salt Tolerance ^b
A	100	3.0	—	3.0	50.9	90.7	fine	1
B	100	—	3.0	3.0	49.5	81.0	fine	2

(a) AAPMA = 2-acetoacetoxypropyl methacrylate.
 (b) 1 = $CaCl_2$, $Zn(OAc)_2$, $CoCl_2$, $SnCl_2$, $NiCl_2$, $ZnCl_2$, at 100% crosslink densities.
 2 = $CaCl_2$, $Zn(OAc)_2$, $CoCl_2$, $SnCl_2$, $NiCl_2$, $ZnCl_2$, $Al_2(SO_4)_3$, $MgCl_2$, at 100% crosslink densities.

excessive level of coagulum. These results suggest that a significant disparity existed in the interaction ability of this surfactant molecule with the particle surface (resulting from an improper hydrophilic lipophilic balance) and/or that the chelate monomer was kinetically unsuited to the conditions employed during polymerization.

A search of the literature revealed that some of the variables which influence salt tolerance and the degree of conversion have been investigated. For example, when a nonionic/anionic blend of surfactants was used to effect emulsion polymerization, the resulting acrylic latex displayed a 95 to 100% degree of conversion and would tolerate additions (up to 30% each) of $CaCl_2$, $AlCl_3$, and $FeCl_3$.²² In studies of other emulsion systems, surfactants which possessed poly(oxyethylene) chains capable of orientations perpendicular to the particle surface were found to impart an increased degree of salt tolerance.²³

In light of these considerations, a nonionic/anionic surfactant study was conducted to determine if nonionic surfactants would provide a synergistic stabilizing effect when blended with Gafac RE-610, Gafac RE-960, and Alipal EP-120, respectively. Again, the polymerizations utilized a batch addition technique of monomer, 2% nonionic and 2% anionic surfactants, 0.2% ammonium persulfate, 0.6% n-dodecylmercaptan, 80°C reaction temperature, and six hour reaction time. The results of this study are presented in *Table 3*.

Since nonionic surfactants containing 30 or more ethylene oxide units can be used as the primary emulsifying agents in the polymerization of acrylate monomers, Triton X-405 with 40 ethylene oxide units was chosen as a representative candidate. In comparison with the latex prepared from Gafac RE-610, the surfactant blend of Triton X-405 and Gafac RE-610 produced a latex with a 1.5% decrease in the degree of conversion and a 1.2% decrease in the amount of coagulum. More significantly, however, the blend of surfactants imparted a marked increase in the salt tolerance. The latex was found to tolerate 1.42 millimoles of $CaCl_2/g$ latex which was the amount of calcium ion required to convert all of the free acetoacetate groups into the ion chelated form. The latex would also accept 0.569 millimoles of $Zn(OAc)_2/g$ latex.

The addition of Triton X-405 to Gafac RE-960 resulted in a similar reduction in the degree of conversion and coagulum level; however, the $Zn(OAc)_2$ tolerance increased to 0.808 millimoles/g latex. This increase in salt tolerance for the emulsion containing Gafac RE-960 as compared to the emulsion prepared with Gafac RE-610

Table 5—Evaluation of Latex B^a Catalyzed with MgCl₂—Dried for 35 Days at Room Temperature

Curing Agent Concentration ^b (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/mil	Mandrel Flexibility (pass) (in.)	Tensile Strength (Kg/cm ²)	Elongation (%)	Water Blush Time (sec)
0	3.0	12	B	67	70	0.125	33.8	496	240
10	2.0	8	B	59	42	0.5	15.2	508	130
20	1.8	7	B	65	42	0.25	30.2	218	65
30	2.2	10	B	71	49	0.25	32.1	271	50
40	1.8	8	B	61	54	0.25	30.2	315	45
50	1.8	11	B	49	63	0.25	25.5	320	30
60	2.0	11	B	19	55	0.25	27.7	223	30
70	2.5	9	B	11	48	0.25	24.2	211	15
80	1.75	9	B	4	57	0.25	33.6	245	10
90	1.75	9	B	4	51	0.25	32.7	206	4
100	1.75	9	B	3	50	0.25	31.7	211	4

(a) See Table 4 for a description of the emulsion.

(b) Curing agent concentration represents that percentage of the total amount of acetoacetate groups for which metal ions were available for crosslinking.

probably arises from the effectiveness of a longer poly-(oxyethylene) chain which stabilizes against particle flocculation. Unfortunately, neither of these nonionic/anionic systems would tolerate Al³⁺ and Mg²⁺ ions since these ions possess high electrical potential energies and can very efficiently reduce the zeta potential at the plane of shear.

The addition of Triton X-405 to Alipal EP-120 provided no discernible change in degree of conversion or amount of coagulum. However, the latex stabilized by this nonionic/anionic combination of surfactants was found to accept the following metal ions in the concentrations required to combine with 100% of the available acetoacetate groups: Ca²⁺, Zn²⁺, Co²⁺, Ni²⁺, Mg²⁺, and Al³⁺.

To produce more workable emulsions, the two most acceptable systems were scaled up from 33% solids to about 50% solids. The results in Table 3 indicate that the solids increase for the emulsions containing the Gafac RE-960/Triton X-405 and Alipal EP-120/Triton X-405 surfactant combinations had no significant effect on the

degree of conversion, percent coagulum, or salt tolerance of these latexes.

Even though Alipal EP-120 is recommended for the emulsion polymerization of water associative monomers like vinyl acetate, the inability to obtain higher degrees of polymer conversion reflected some kinetic or thermodynamic ineffectiveness between this surfactant and the 2-AAPMA monomer. In an attempt to increase the surfactant efficiency, Igepal® C0-997, a nonionic surfactant with 100 ethylene oxide units, was substituted for Triton X-405 in the Alipal EP-120 system. The results in Table 3 indicate that this substitution had no effect on the degree of conversion or salt tolerance of the latex.

Besides the addition of nonionic surfactants, another conventional method for producing high yield, salt-stable emulsions involves the utilization of protective colloids. For example, the emulsion polymerization of vinyl acetate in the presence of a poly(vinyl alcohol) protective colloid resulted in the formation of a latex which

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Table 6—Evaluation of Latex B^a Catalyzed with MgCl₂—Cured Two Hours at 125°C

Curing Agent Concentration ^b (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/mil	Mandrel Flexibility (pass) (in.)	Water Blush Time (sec)
0	3.1	17	B	81	71	pass 0.375	375
10	1.8	9	B	60	150	fail 1.00	900
20	2.0	10	B	63	190	fail 1.00	270
30	2.1	9	B	63	200	fail 1.00	75
40	1.75	12	B	55	242	pass 0.25	70
50	1.75	12	B	29	228	pass 1.00	55
60	1.6	12	B	10	275	pass 0.75	55
70	1.6	11	B	9	271	pass 0.5	55
80	1.0	12	F	5	440	pass 0.5	20
90	1.0	11	H	3	380	pass 0.5	20
100	0.95	11	H	3	368	pass 0.5	20

(a) See Table 4 for a description of the emulsion.

(b) Curing agent concentration represents that percentage of the total amount of acetoacetate groups for which metal ions were available for crosslinking.

Table 7—Evaluation of Latex B^a Catalyzed with SnCl₂—Dried for 35 Days at Room Temperature

Curing Agent Concentration ^b (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/mil	Mandrel Flexibility (in.)	Tensile Strength (Kg/cm ²)	Elongation (%)	Water Blush Time (sec)
0	3.0	12	B	67	70	pass 0.125	33.8	496	240
10	3.75	5	<6B	82	32	fail 1.00	26.2	370	360
20	3.7	5	<6B	60	29	fail 1.00	26.0	211	150
30	3.0	6	<6B	67	103	fail 1.00	32.7	313	55
40	3.5	6	<6B	66	91	fail 1.00	29.2	247	35
50	1.1	7	<6B	68	281	fail 1.00	35.2	336	30
60	1.1	9	B	68	272	fail 1.00	39.7	369	30
70	0.75	10	B	62	424	fail 1.00	50.2	219	30
80	0.4	9	HB	65	800	fail 1.00	52.1	228	30
90	0.6	8	HB	47	566	fail 1.00	42.6	124	25
100	0.75	5	6B	39	466	fail 1.00	33.7	114	25

(a) See Table 4 for a description of the emulsion.

(b) Curing agent concentration represents that percentage of the total amount of acetoacetate groups for which metal ions were available for crosslinking.

displayed excellent mechanical and freeze-thaw stability, a high degree of conversion, and excellent stability to the addition of 10% each of H₂SO₄, NaCl, and MgCl₂.²⁴

In light of these considerations, a commercially produced protective colloid, Gelvatol® 20-60, was evaluated for the emulsion polymerization of 2-AAPMA. While protective colloids can be substituted for a majority of the surfactant, the evaluation utilized 1% Gelvatol 20-60 in combination with 1% Alipal EP-120 and 2% Triton X-405. This substitution of 1% protective colloid for 1% anionic surfactant resulted in complete coagulation of the latex after a three hour reaction period.

As a final attempt to improve the efficiency of the

Alipal EP-120/Triton X-405 surfactant combination, the effect of a neutralizing buffer was investigated. While the emulsion polymerization of acrylate monomers is usually insensitive to changes in pH, a buffer is sometimes included to insure optimal surfactant efficiency. The water/surfactant mixture was buffered from a pH of 5.4 to 7.3 with ammonium hydroxide. As indicated by the results in Table 3 (the last entry), adjustment of the pH resulted in a 19% increase in the degree of conversion and an 8% increase in the amount of coagulum. Thus, the buffered Alipal EP-120/Triton X-405 combination produced a latex with a yield approaching that of the emulsion employing the Gafac RE-960/Triton X-405 surfactant blend. Both of these nonionic/anionic-based latexes satisfied the basic requirements necessary for evaluation as model, chelating emulsions.

Gelvatol is a registered trademark of the Monsanto Corp.

Table 8—Evaluation of Latex B^a Catalyzed with SnCl₂—Cured Two Hours at 125°C

Curing Agent Concentration ^b (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/mil	Mandrel Flexibility (in.)	Water Blush Time (sec)
0	3.1	17	B	81	71	pass 0.375	375
10	7.0	7	HB	79	30	fail 1.00	3600
20	3.1	4	HB	52	66	fail 1.00	3600
30	4.0	6	HB	45	67	fail 1.00	2640
40	3.0	9	HB	48	76	fail 1.00	1920
50	2.5	8	HB	27	96	fail 1.00	1080
60	1.6	8	HB	58	pass 500 hardness = 6B	fail 1.00	600
70	1.8	8	HB	54	pass 500 hardness = 6B	fail 1.00	540
80	1.3	8	F	41	pass 500 hardness = 5B	fail 1.00	480
90	1.4	8	2H	37	pass 500 hardness = 3B	fail 1.00	360
100	1.4	5	3H	40	pass 500 hardness = B	fail 1.00	—

(a) See Table 4 for a description of the emulsion.

(b) Curing agent concentration represents that percentage of the total amount of acetoacetate groups for which metal ions were available for crosslinking.

Choice of Model, Chelating Emulsions

Of the emulsions just described, along with others which were prepared, the systems which were most acceptable for the ensuing ionic crosslinking study as judged from the overall film appearance and properties, amount of coagulum formation, degree of conversion, tolerance to salt, and requirements for surfactant are those presented in *Table 4*. Emulsions A and B, containing 6% combined surfactant, were considered the prime candidates for the initial evaluation of the crosslinking potential of these systems.

Selection of the Curing Agents

The choice of metal ion curing agent was made by weighing such factors as the thermodynamic stability of the resulting complex, availability, toxicity, and chemical and physical suitability of the various ions. Unfortunately, the available thermodynamic data provided only qualitative guidelines for selection of suitable candidates because of the lack of agreement among much of these data.²⁵⁻²⁷

Thus, in light of their acknowledged capacity to form stable coordination complexes and because of their tractability with the emulsion environment, the following metal salts were selected as curing agents for preliminary evaluations in the chelating emulsions: $MgCl_2$, $NiCl_2$, $CaCl_2$, $Al_2(SO_4)_3$, $CoCl_2$, $ZnCl_2$, $Zn(OAc)_2$, and $SnCl_2$.

The chloride form of each metal was selected for the investigation since halide ions are less pernicious to the latex stability than are other anions. However, $Al_2(SO_4)_3$ was substituted for $AlCl_3$ in the study since the sulfate form does not react with water. Also, because the chloride ion is a weak base and may not demonstrate a marked tendency to remove a methylene hydrogen from the acetoacetate group, $Zn(OAc)_2$ was included in the series to investigate the effect that a stronger base might have on the crosslinking ability.

Determination Of the Optimum Concentration Of Crosslinking Agent

In conventional crosslinkable systems, the addition of a stoichiometric amount of curing agent to the base polymer usually results in an increase in those physical properties which are directly related to the crosslink density achieved by the resulting mixture. For example, the formation of a network structure is usually reflected by increases in tensile strength, modulus, and solvent resistance properties.

In chelating systems this direct relationship between certain physical properties and crosslink density may not be so ostensible since the presence of higher concentrations of salt may result in a reduction in some film properties. For example, unless the resulting increase in crosslink density can totally compensate for the increased hydrophilicity imparted by the salt, successive increases in salt concentrations may drastically reduce the water resistance of the coating. Therefore, in anticipation of a possible compromise in certain diametrically opposed properties, a salt concentration study was conducted to determine the amount of curing agent required to pro-

duce an optimization of the primary coating properties.

The investigation utilized both latex and dimethylformamide (DMF) solution forms of poly(2-AAPMA) containing $SnCl_2$ and $MgCl_2$ in amounts (based upon the stoichiometric amount of available acetoacetate groups) which provided curing agent concentrations from 10 to 100% and varying in increments of 10%. The salt catalyzed systems were coated onto glass and Bonderite® 37 steel panels with an adjustable Gardner blade and were dried for 35 days at room temperature or oven cured for two hours at 125°C. The cured coatings were then tested for Sward and pencil hardness, flexibility, impact resistance, adhesion, solvent and water resistance, tensile strength, and percent elongation. The surface gloss was also measured to provide some indication of the compatibility of each salt in the coating.

The results of the curing agent ladder study for the latex-cast system are presented in *Tables 5, 6, 7, and 8*, while the results for the solvent-based coatings cast from DMF solutions of poly(2-AAPMA) containing $SnCl_2$ are presented in *Tables 9 and 10*. All the samples reported in these tables displayed an impact resistance of less than 5 in./lb and a crosshatch adhesion of 100%. The low impact resistance presumably resulted from the somewhat low molecular weight of the polymer.

While a large number of comparisons and correlations could be made from the data presented in *Tables 5 through 10*, the following conclusions reflect the central theme of the study which was to determine the amount of curing agent required for an optimization of the primary properties of the system:

(1) *Latex coatings containing $MgCl_2$ which were dried at room temperature (Table 5).*

The addition of $MgCl_2$ to the latex provided no increase in the properties of the coating. As the amount of $MgCl_2$ was increased from 10 to 100%, based on total stoichiometry, no significant differences were observed in film Sward hardness, pencil hardness, solvent resistance, flexibility, tensile strength, or elongation. However, as the $MgCl_2$ concentration increased, the water resistance and gloss decreased. Obviously, these data indicate that little or no reaction occurred between Mg^{2+} ions and the acetoacetate groups of the polymer.

(2) *Latex coatings containing $MgCl_2$ which were cured 2 hr at 125°C (Table 6).*

In contrast with the room temperature dried coatings, the thermally cured coatings displayed an increase in pencil hardness and solvent resistance as the crosslink density increased. Films containing 80% of the stoichiometric amount of curing agent displayed a pencil hardness of F and a solvent resistance of 440 double rubs/mil. Apparently, the crosslink density was still below the threshold required to produce completely insoluble polymer.

(3) *Latex coatings containing $SnCl_2$ which were dried at room temperature (Table 7).*

With successive increases in the amount of $SnCl_2$, the solvent resistance, Sward and pencil hardness, and ten-

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Table 9—Evaluation of Solvent-Cast Coatings Containing SnCl₂—Dried for 35 Days at Room Temperature

Curing Agent Concentration ^a (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/mil	Mandrel Flexibility (in.)	Tensile Strength (Kg/cm ²)	Elongation (%)	Hardness After 30 min Immersion in Water at 23°C
0	1.3	8	B	91	86	pass 0.25	5.0	2935	<6B
10	1.0	7	5B	77	90	pass 0.25	20.5	1174	<6B
20	1.25	5	5B	82	108	pass 0.25	19.3	623	<6B
40	1.6	6	2B	63	350	pass 0.25	36.2	466	<6B
60	1.5	7	B	60	400	pass 0.25	36.3	419	<6B
80	2.0	7	HB	60	225	pass 1.00	43.8	348	<6B
100	1.4	9	HB	31	271	fail 1.00	59.1	303	<6B

(a) Curing agent concentration represents that percentage of the total amount of acetoacetate groups for which metal ions were available for crosslinking.

sile strength of the coatings increased. A marked increase in these properties occurred at 70% curing agent concentration. However, the degree of crosslinking was not sufficient to produce completely insoluble polymer.

In contrast with films containing MgCl₂, the gloss of coatings containing SnCl₂ showed little variance with increasing concentrations of curing agent. Apparently SnCl₂ was more compatible with the system than was MgCl₂.

The water resistance of the system was not increased by crosslinking, and above a curing agent concentration of 20% the water sensitivity increased markedly.

(4) *Latex coatings containing SnCl₂ which were cured for 2 hr at 125°C (Table 8).*

After thermal curing, the coatings containing 60% of the stoichiometric amount of SnCl₂ displayed excellent solvent resistance. After 500 DMF double rubs, the coating remained intact and displayed only slight plasticization by the solvent.

In contrast to the room temperature dried samples, the water resistance of the coatings was increased by cross-

linking. However, the coatings did become more water sensitive with successive increases in salt concentrations.

(5) *Solvent-cast coatings containing SnCl₂ which were dried at room temperature (Table 9).*

Since in solution the metal ions can achieve a more intimate degree of comingling with the polymer, solvent-borne coatings were expected to display a significant improvement in properties as compared to the latex coatings. At a SnCl₂ concentration of 40%, a substantial increase in pencil hardness, solvent resistance, and tensile strength was observed. In the analogous latex coatings, an equivalent property increase was not observed until the SnCl₂ concentration reached 70%.

(6) *Solvent-cast coatings containing SnCl₂ which were cured 2 hr at 125°C (Table 10).*

After thermal curing, the coatings containing 20% of the stoichiometric amount of SnCl₂ displayed a marked increase in Sward and pencil hardness and solvent resistance. The coatings were found to be insoluble in the solvent from which they were cast and experienced only

Table 10—Evaluation of Solvent-Cast Coatings Containing SnCl₂—Cured Two Hours at 125°C

Curing Agent Concentration ^a (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/mil	Mandrel Flexibility (in.)	Hardness After 30 min Immersion in Water at 23°C
0	1.2	13	HB	85	94	fail 1.00	<6B
10	1.25	15	B	81	310	fail 1.00	<6B
20	2.1	22	2H	84	pass 500 hardness = 6B	fail 1.00	<6B
40	3.25	25	2H	79	pass 500 hardness = 6B	fail 1.00	<6B
60	3.25	24	2H	88	pass 500 hardness = 6B	fail 1.00	<6B
80	3.25	34	2H	70	pass 500 hardness = 6B	fail 1.00	<6B
100	3.25	8	2H	24	pass 500 hardness = 6B	fail 1.00	<6B

(a) Curing agent concentration represents that percentage of the total amount of acetoacetate groups for which metal ions were available for crosslinking.

Table 11—Effect of Various Metal Ions on the Properties of Room Temperature Dried, Latex Films

Curing Agent Concentration (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/mil	Mandrel Flexibility (in.)	Tensile Strength (Kg/cm ²)	Elongation (%)	Water Blush Time (sec)
0	3.0	12	B	67	70	pass 0.125	33.8	496	240
20 CaCl ₂	2.2	21	6B	55	45	fail 1.00	20.9	4	20
60 CaCl ₂	2.0	14	<6B	34	44	fail 1.00	27.7	3	7
80 CaCl ₂	3.0	9	<6B	21	30	fail 1.00	30.4	3	5
20 Zn(OAc) ₂	1.9	27	F	77	pass 500 hardness=6B	fail 1.00	102.1	8	2400
60 Zn(OAc) ₂	1.9	10	6B	34	98	fail 1.00	80.8	7.2	15
80 Zn(OAc) ₂	2.25	7	F	22	91	fail 1.00	53.0	14	15
20 CoCl ₂	3.5	14	6B	41	27	fail 1.00	36.5	13	75
60 CoCl ₂	2.4	16	6B	22	35	fail 1.00	31.1	4.1	15
80 CoCl ₂	1.2	11	6B	26	47	fail 1.00	29.0	3	55
20 NiCl ₂	2.7	6	H	33	91	fail 1.00	50.1	436	30
60 NiCl ₂	1.9	8	2H	4	73	fail 1.00	19.9	560	90
80 NiCl ₂	3.0	16	4H	4	53	fail 1.00	9.3	355	90
20 ZnCl ₂	2.75	18	6B	67	131	fail 1.00	50.1	359	150
60 ZnCl ₂	2.0	8	<6B	76	pass 500 hardness=6B	pass 0.25	19.9	560	150
80 ZnCl ₂	2.3	5	<6B	62	255	pass 0.25	9.3	390	150
20 Al ₂ (SO ₄) ₃	2.1	18	HB	44	109	pass 0.1875	26.3	109	135
60 Al ₂ (SO ₄) ₃	1.25	9	6B	2	86	pass 0.1875	36.3	24	15
80 Al ₂ (SO ₄) ₃	1.25	9	6B	2	84	pass 0.1875	44.1	61	10
20 SnCl ₂	3.7	5	<6B	60	29	fail 1.00	26.0	211	150
60 SnCl ₂	1.1	9	B	68	272	fail 1.00	39.7	369	30
80 SnCl ₂	0.4	9	HB	65	800	fail 1.00	52.1	228	30
20 MgCl ₂	1.8	7	B	65	42	pass 0.25	30.2	218	65
60 MgCl ₂	2.0	11	B	19	55	pass 0.25	27.7	223	30
80 MgCl ₂	1.75	9	B	4	57	pass 0.25	33.6	245	10

plasticization after 500 DMF double rubs. In the analogous latex coatings, an equivalent property increase was not observed until the SnCl₂ concentration reached 60%. Unfortunately, the degree of crosslinking did not compensate for the water sensitivity imparted by the salt.

Effects of Various Metal Ions On the Film Properties of Chelating Emulsions

Based upon the results obtained from the curing agent concentration study, the remaining crosslinking agents were evaluated at 20, 60, and 80% of the concentration required to consume the stoichiometric amount of acetate groups. The 20% level was selected because of its less pernicious effect on water sensitivity while the 60 and 80% levels were chosen because of the greater effect which higher concentrations of curing agent have on those properties which reflect the formation of a crosslinked matrix.

The evaluation utilized both latex and DMF solution forms of poly(2-AAPMA). The solutions were prepared by dissolving polymer which had been isolated from the latex. Because the various salts displayed a preferred compatibility with the type of surfactant combination used to prepare the emulsion, latex A (Table 4) was evaluated with CaCl₂, Zn(OAc)₂, CoCl₂, and NiCl₂ while latex B (Table 4) was evaluated with SnCl₂, MgCl₂, ZnCl₂, and Al₂(SO₄)₃. The resulting salt-catalyzed emulsions were coated onto glass and Bonderite 37 steel

panels with an adjustable Gardner blade and were dried for 35 days at room temperature or oven cured for two hours at 125°C. The cured coatings were then tested for Sward and pencil hardness, gloss, flexibility, impact resistance, adhesion, solvent and water resistance, tensile strength, and elongation. While the crosslinking ability of these systems was best reflected by increases in tensile strength, solvent resistance, and coating hardness, the other test measurements were included to provide an overview of the physical capabilities of these systems. Tables 11 and 12, respectively, present the results for ambient and thermally cured coatings derived from latex forms while Tables 13 and 14, respectively, present the results for ambient and thermally cured coatings derived from solution forms.

PARAMETERS AFFECTING CROSSLINKING AND CONCLUSIONS DRAWN

If the numerous parameters examined in this study were correlated with each result obtained, various sets of conclusions could be drawn concerning the theoretical, empirical, and applied aspects of these systems. However, since the principal objective of this survey was to examine the feasibility of producing chelating emulsions, the correlations and conclusions that follow are those which deal primarily with the variables associated with the formation of a crosslinked matrix. The results obtained from coatings derived from solution forms are

Table 12—Effect of Various Metal Ions on the Properties of Thermally Cured, Latex Films

Curing Agent Concentration (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/mil	Impact (in.-lb)	Mandrel Flexibility (in.)	Water Blush Time (sec)
0	3.1	17	B	81	71	5	pass 0.375	375
20 CaCl ₂	3.0	6	HB	52	56	5	fail 1.00	55
60 CaCl ₂	3.0	3	<6B	7	73	5	fail 1.00	25
80 CaCl ₂	5.0	3	<6B	15	30	5	fail 1.00	25
20 Zn(OAc) ₂	5.0	9	4H	64	pass 500 hardness=6B	5	fail 1.00	3600
60 Zn(OAc) ₂	2.6	8	3H	26	pass 500 hardness=6B	5	fail 1.00	220
80 Zn(OAc) ₂	2.0	5	F	42	245	5	fail 1.00	180
20 CoCl ₂	4.5	10	3H	51	64	5	fail 1.00	240
60 CoCl ₂	2.4	16	6H	10	179	5	fail 1.00	50
80 CoCl ₂	2.5	8	6H	19	130	5	fail 1.00	45
20 NiCl ₂	4.0	14	2H	88	pass 500 hardness=6B	5	fail 1.00	60
60 NiCl ₂	2.2	5	5H	15	pass 500 hardness=6B	5	fail 1.00	120
80 NiCl ₂	2.5	—	7H	—	—	5	fail 1.00	150
20 ZnCl ₂	3.6	9	5H	86	pass 500 hardness=6B	5	fail 1.00	150
60 ZnCl ₂	4.2	9	6H	93	pass 500 hardness=6H	5	fail 1.00	390
80 ZnCl ₂	3.5	15	7H	88	pass 500 hardness=7H	5	fail 1.00	390
20 Al ₂ (SO ₄) ₃	3.6	12	4H	57	196	5	fail 1.00	150
60 Al ₂ (SO ₄) ₃	2.4	6	5H	1	134	5	fail 1.00	30
80 Al ₂ (SO ₄) ₃	3.0	8	6H	1	170	5	fail 1.00	30
20 SnCl ₂	3.1	4	HB	52	66	5	fail 1.00	3600
60 SnCl ₂	1.6	8	HB	58	pass 500 hardness=6B	5	fail 1.00	600
80 SnCl ₂	1.3	8	F	41	pass 500 hardness=5B	5	fail 1.00	480
20 MgCl ₂	2.0	10	B	63	190	5	fail 1.00	270
60 MgCl ₂	1.6	12	B	10	275	5	pass 0.75	55
80 MgCl ₂	1.0	12	F	5	440	5	pass 0.5	20

presented only for comparative purposes since the crosslinking ability of solvent-cast films has been substantiated in other studies.^{17,28,29}

(1) *The effectiveness of various metal ions as crosslinking agents for latex films dried under ambient conditions (Table 11).*

Of the properties measured in Table 11, the degree of crosslinking is reflected best by the solvent resistance and tensile strength data. The film hardness data, which reflect in part the modulus of the system, also provides some indication of the formation of a network structure. If the various metal salts were ranked for their effectiveness in enhancing film hardness, solvent resistance and tensile strength,³⁰ the ratings would indicate that Zn(OAc)₂ was more consistent than the other salts in providing an overall enhancement of the coating properties. In comparison with coatings containing the other salts, the samples containing 20% Zn(OAc)₂ were found to display superior Sward hardness, gloss, solvent and

water resistance, and tensile strength. The tensile strength data, which increased three-fold as compared to the control, together with the solvent resistance data, which indicated that the coating was insoluble in a solvent for the polymer, reflected the achievement of a high degree of crosslinking. However, the preeminence displayed by Zn(OAc)₂ was found to be concentration dependent since Zn(OAc)₂ levels greater than 20% resulted in a reduction in the properties of the coating.

That these property increases were governed by more than just the nature of the Zn²⁺ ion was demonstrated by the somewhat mediocre results displayed by coatings containing 20% ZnCl₂. Obviously, these data indicate that the nature of the anion also has a marked effect on the crosslinking ability of chelating systems. As compared to the chloride ion, the more basic acetate anion has a greater tendency to abstract a methylene proton from the acetoacetate derivative. The formation of the acetoacetate anion would greatly enhance the rate of the chelate reaction.

Table 13—Effect of Various Metal Ions on the Properties of Room Temperature Dried, Solvent-Cast Films

Curing Agent Concentration (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/ml	Mandrel Flexibility (in.)	Tensile Strength (Kg/cm ²)	Elongation (%)	Hardness After 30 min Immersion in H ₂ O at 23° C
0	1.3	8	B	91	86	pass 0.25	5.0	2935	<6B
20 CaCl ₂	2.2	2	<6B	52	34	pass 0.1875	2.0	2047	<6B
60 CaCl ₂	8	2	<6B	48	22	pass 0.1875	2.0	2053	<6B
80 CaCl ₂	8	4	<6B	21	21	pass 0.1875	23.9	43	<6B
20 Zn(OAc) ₂	3.75	12	HB	88	23	fail 1.00	25.6	15	<6B
60 Zn(OAc) ₂	3.0	12	2H	60	42	fail 1.00	25.1	1	<6B
80 Zn(OAc) ₂	2.5	6	2H	5	42	fail 1.00	30.1	35	<6B
20 CoCl ₂	2.25	7	<6B	76	37	pass 0.1875	8.7	285	<6B
60 CoCl ₂	3.0	4	6B	12	13	pass 0.1875	18.1	370	<<6B
80 CoCl ₂	3.0	3	B	14	13	pass 0.1875	14.7	575	<<6B
20 NiCl ₂	3.1	3	<6B	47	31	pass 0.1875	7.7	1315	<6B
60 NiCl ₂	3.3	2	<6B	69	33	pass 0.1875	9.8	1032	<6B
80 NiCl ₂	3.5	2	6B	18	42	fail 1.00	11.4	420	<<6B
20 ZnCl ₂	3.6	4	<6B	73	23	pass 0.1875	4.2	1877	<<6B
60 ZnCl ₂	3.5	3	<6B	67	37	pass 0.1875	10.5	2168	<<6B
80 ZnCl ₂	3.5	2	<6B	6	32	pass 0.1875	0.3	3180	<<6B
20 Al ₂ (SO ₄) ₃	3.4	8	B	75	32	pass 0.1875	19.0	137	<6B
60 Al ₂ (SO ₄) ₃	4.5	2	5B	28	19	pass 0.1875	12.9	91	<<6B
80 Al ₂ (SO ₄) ₃	4.5	3	5B	23	17	pass 0.1875	17.3	27	<<6B
20 SnCl ₂	1.25	5	5B	82	108	pass 0.25	19.3	623	<<6B
60 SnCl ₂	1.5	7	B	60	400	pass 0.25	36.3	419	<B
80 SnCl ₂	2.0	7	HB	60	225	pass 1.00	43.8	348	<6B
20 MgCl ₂	4.0	3	<6B	46	20	pass 0.1875	5.3	2207	<6B
60 MgCl ₂	2.5	3	<6B	25	21	pass 0.1875	8.3	510	<<6B
80 MgCl ₂	5.5	2	<6B	29	20	pass 0.1875	2.7	1960	<<6B

Increasing the amount of ZnCl₂ from 20 to 60% caused a marked improvement in the solvent resistance of the coating. The crosslinking density provided by this 40% increase in curing agent concentration was sufficient to prevent dissolution of the polymer.

Thus, the two salts which provided the greatest increase in latex film properties after drying at room temperature were Zn(OAc)₂ at 20% concentration and ZnCl₂ at 60% concentration. SnCl₂ at 80% concentration also demonstrated a significant reinforcing effect on latex films.

(2) *The effectiveness of various metal ions as crosslinking agents for latex films cured for 2 hr at 125° C (Table 12).*

Of the properties measured in Table 12, the degree of crosslinking is reflected best by the solvent resistance data and, to a lesser degree, by film hardness data. The thermally cured samples became too brittle to evaluate for tensile strength.

Again, because of the inconsistencies in the order of effectiveness for the various ions, specific, interrelated conclusions are difficult to draw. Little or no correlation exists between the salt order for Sward hardness after room temperature drying and the salt order for Sward hardness after thermal curing. However, a greater correlation seems to exist between the salt order for solvent resistance obtained from the two methods of curing.

Of the various curing agents tested, Zn(OAc)₂ and

ZnCl₂ were again consistent in their ability to produce coatings with excellent solvent resistance. The latex coatings containing ZnCl₂ at 60 and 80% concentrations retained their original hardness after 500 DMF double rubs. This excellent solvent resistance reflects an inordinately high efficiency of crosslinking.

After thermal curing little difference could be discerned in the solvent resistance provided by Zn(OAc)₂, NiCl₂, and SnCl₂ at 60% concentrations. While each of these systems experience some reduction in film hardness after 500 DMF double rubs, the degree of crosslinking provided by these salts was sufficient to produce coatings which were insoluble in a solvent for the polymer.

Thus, four salts were found to be effective crosslinking agents for thermally cured, latex films: ZnCl₂, which imparted such high crosslink densities that the coating was unaffected by solvent, and Zn(OAc)₂, NiCl₂, SnCl₂ all of which imparted good solvent resistance to the coating.

(3) *The effectiveness of various metal ions as crosslinking agents for solvent-cast films dried under ambient conditions (Table 13).*

Since the evaluation of the crosslinking capacity of solvent-cast coatings served mainly as a control, the results obtained from these systems (Table 13) are included primarily as a means of comparison with the latex systems.

A juxtapositional comparison of the results obtained from the latex-cast coatings (Table 11) with the results

Table 14—Effect of Various Metal Ions on the Properties of Thermally Cured, Solvent-Cast Films

Curing Agent Concentration (%)	Coating Thickness (mils)	Rocker Hardness	Pencil Hardness	Specular Gloss (60°)	DMF Double rubs/mil	Impact (in.-lb)	Mandrel Flexibility (in.)	Hardness After 30 min Immersion in H ₂ O at 23° C
0	1.2	13	HB	85	94	5	fail 1.00	<6B
20 CaCl ₂	2.5	9	HB	46	58	5	fail 1.00	<6B
60 CaCl ₂	5.2	—	6B	12	23	5	fail 1.00	<6B
80 CaCl ₂	5.0	—	6B	18	21	5	fail 1.00	<6B
20 Zn(OAc) ₂	3.5	24	3H	74	pass 500 hardness=6B	5	fail 1.00	F
60 Zn(OAc) ₂	2.3	20	5H	56	pass 500 hardness=2B	5	fail 1.00	HB
80 Zn(OAc) ₂	1.8	12	3H	3	pass 500 hardness=6B	5	fail 1.00	HB
20 CoCl ₂	2.1	15	HB	66	152	5	fail 1.00	<6B
60 CoCl ₂	2.0	7	F	5	130	5	fail 1.00	6B
80 CoCl ₂	2.1	3	2H	5	128	5	fail 1.00	6B
20 NiCl ₂	3.0	11	6B	67	41	5	fail 1.00	<6B
60 NiCl ₂	3.0	7	6B	30	40	5	fail 1.00	<6B
80 NiCl ₂	2.0	4	6B	15	45	5	fail 1.00	<6B
20 ZnCl ₂	3.9	11	6B	78	pass 500 hardness<6B	5	fail 1.00	<6B
60 ZnCl ₂	3.9	6	<6B	83	pass 500 hardness<6B	5	pass 0.5	<6B
80 ZnCl ₂	4.6	4	<6B	83	93	5	pass 0.5	<6B
20 Al ₂ (SO ₄) ₃	3.25	18	HB	75	66	5	fail 1.00	2B
60 Al ₂ (SO ₄) ₃	6.0	4	HB	15	57	5	fail 1.00	HB
80 Al ₂ (SO ₄) ₃	3.6	3	6B	15	pass 500 hardness=6B	5	fail 1.00	4B
20 SnCl ₂	2.1	22	2H	84	pass 500 hardness=6B	5	fail 1.00	<6B
60 SnCl ₂	3.25	24	2H	88	pass 500 hardness=6B	5	fail 1.00	<6B
80 SnCl ₂	3.25	34	2H	70	pass 500 hardness=6B	5	fail 1.00	<6B
20 MgCl ₂	2.0	13	HB	42	pass 500 hardness=6B	5	fail 1.00	6B
60 MgCl ₂	2.5	—	3H	8	pass 500 hardness=6B	5	fail 1.00	5B
80 MgCl ₂	0.4	—	HB	5	1100	5	fail 1.00	3B

obtained from the solvent-cast coatings (Table 13) reveals that a marked difference exists between the order of salt effectiveness in latex films and the order of salt effectiveness in solvent-cast films. While the magnitude of these differences prevents establishing direct correlations between the nature of the metal ion and the properties of these film types, the salt order obtained by rating the effectiveness of the various metal ions to enhance hardness, solvent resistance, and tensile strength³⁰ intimates that Zn(OAc)₂ and SnCl₂ were again consistent in augmenting the coatings properties. The crosslinking ability of these salts was manifested by the four- and five-fold increases in tensile strength displayed by the samples containing SnCl₂ and Zn(OAc)₂ at 20% concentrations, respectively.

Interestingly, the latex coatings containing Zn(OAc)₂ at 20% concentration were found to possess properties which were superior to the analogously-cured, solvent-cast coatings; for example, coatings cast from latex form displayed only a slight loss in film hardness after 500 DMF double rubs and provided a tensile strength of 102.1 Kg/cm² while the analogously-cured, solvent-cast coatings displayed a solvent resistance of only 23 double rubs/mil and demonstrated a tensile strength of 25.6 Kg/cm². In light of these results it appears that the environment offered by the latex system was more conducive for promoting the crosslinking reaction than was the environment offered by the solvent system. The enhanced crosslinking ability of emulsion systems was also demonstrated in ZnCl₂-containing coatings. At a

concentration of 60% $ZnCl_2$, the latex coating displayed a slight loss in film hardness after 500 DMF double rubs and provided a tensile strength of 19.9 Kg/cm² while the analogously-cured, solvent-cast coating displayed a solvent resistance of 37 double rubs/mil and demonstrated a tensile strength of 10.5 Kg/cm².

This unexpected trend in which latex coatings were found to be superior to solvent-cast coatings in tensile strength and solvent resistance extended throughout this latter evaluation.

The reduced tensile strength and solvent resistance of the solution-cast coatings could result from plasticization caused by residual solvent or from insufficient crosslink density. A difference in crosslink densities between the two systems could arise if solvent association with the ion or ligand retarded the chelate reaction.

The solvent-cast system which displayed the greatest property increase contained $SnCl_2$ at a concentration of 60%. However, the properties displayed by this preeminently rated solvent-cast system did not approach the properties displayed by the latex system containing $Zn(OAc)_2$ at a concentration of 20%. This comparison between the top rated members from each coating type further corroborates the superior crosslinking ability of latex coatings.

(4) *The effectiveness of various metal ions as crosslinking agents for solvent-cast films cured for 2 hr at 125° C (Table 14).*

In line with the previous conclusion, the solvent resistance data presented in Table 14 suggest that the crosslink density achieved by the solvent-cast coatings was less than the crosslink density achieved by the latex coatings. For example, at a concentration of 80% $ZnCl_2$, the latex coatings displayed no decrease in film hardness after 500 DMF double rubs while the solvent-cast coatings would tolerate only 93 DMF double rubs. At a concentration of 60% $ZnCl_2$, the latex coatings again displayed no decrease in film hardness after 500 DMF double rubs while the solvent-cast coatings showed some solvent softening.

This marked difference in chelating ability between solvent-cast systems and latex systems may arise from the chemical and physical disparities which exist between these two forms. For example, in latex systems water may provide a better environment for the enolization of the chelate group, or perhaps water provides a more favorable entropy and enthalpy effect for chelate formation. A physical factor, which could contribute to the property differences displayed by these two systems, may emanate from the loci of the crosslinks generated by each system. The latex particle may become encapsulated by a sheath of metal ions which provides an inordinately high surface crosslink density and which resists penetration by the solvent.

In contrast with the room temperature dried, solvent-cast coatings, the thermally cured samples containing $Zn(OAc)_2$, $ZnCl_2$, $Al_2(SO_4)_3$, $SnCl_2$, and $MgCl_2$ displayed good solvent resistance. While these coatings became somewhat plasticized upon exposure to solvent, the crosslink density afforded by these salts was sufficiently high to prevent dissolution of the coating.

PROJECT OVERVIEW

In summary, a chelate-functional, acrylate-type monomer was prepared using a one-step condensation of diene with 2-hydroxypropyl methacrylate. The resulting 2-AAPMA monomer was polymerized through conventional emulsion techniques, and after a partial optimization of certain variables (surfactant type and concentration, pH, and post-addition of initiator) model, chelatable emulsions were produced which displayed good degrees of conversion, low levels of coagulum, and excellent tolerance to salt.

The crosslinking ability of these emulsions was characterized by torsional braid analysis which indicated that the crosslinking reaction occurred after 24 hr at room temperature or after four hours at 125°C when the coatings contained 100% of the stoichiometric amount of $ZnCl_2$.

The coatings properties of the model, chelatable emulsions were investigated after room temperature drying and after thermal curing using $MgCl_2$, $NiCl_2$, $Al_2(SO_4)_3$, $CoCl_2$, $ZnCl_2$, $Zn(OAc)_2$, and $SnCl_2$ as crosslinking agents. While each of these metal ion curing agents displayed some property improvement which indicated a reinforcement of the coating, $Zn(OAc)_2$ and $ZnCl_2$ were the most consistent and effective curing agents for providing those property changes which reflected the formation of a crosslinked network.

$Zn(OAc)_2$ at 20% of the stoichiometric concentration was found to be the most preeminent curing agent for crosslinking latex-cast films at room temperature. The coatings which resulted displayed excellent rocker hardness, gloss, solvent and water resistance, and tensile strength.

While emulsion coatings containing $ZnCl_2$, $Zn(OAc)_2$, $NiCl_2$, and $SnCl_2$ displayed excellent property increases after thermal curing, the superior crosslinking ability of $ZnCl_2$ at 60 and 80% of the stoichiometric concentration was demonstrated by the 100% solvent resistance of the cured emulsion coating.

Unexpectedly, the solvent resistance of the cured, emulsion systems was found to be superior to the analogously cured solvent-cast coatings.

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SAUL SPINDEL
Corrosion

is Vice-President and Technical Director of the D/L Laboratories and has been associated with the paint industry since 1947. In addition to serving as a Past-President of the New York Society, Mr. Spindel is a member of the Technical Advisory and the Finance Committees. He is also active on the ASTM Committee C-24 on Building Seals and Sealants.



S. LEONARD DAVIDSON
Environmental Control
Delegate

**National Paint and Coatings Association
And Government Agencies**

is a Regulatory Affairs Specialist for NL Chemicals/NL Industries, Inc. Past-President of the Federation for 1971, he also acted as President of the New York Society in 1965. He was honored by the Society, who presented him with the Roy H. Kienle and PaVaC Awards in 1960 and 1968, respectively. Mr. Davidson serves as Society Representative to the Federation. In addition, he has chaired the Inter-Society Color Council and the Bruning Awards Committee. He was also the recipient of the ISCC's Bruning Award in 1969 and the Distinguished Service Award in 1972.



COM-1-H-WH
M-M-A-I-C
N-M-D-R-S

Society Meetings

Birmingham

Dec. 6

Dr. G. Anderson, of the Paint Research Association, spoke on "THE PRA—PAST, PRESENT, AND FUTURE."

Defining the PRA as an international institute for contract research on surface coating problems, Dr. Anderson reviewed the contributions of the association in the past ten years.

Focusing on various departments in the institute, Dr. Anderson cited the information department as one of the finest in the UK. He also reported on many of the laboratory tasks of PRA. Contracts on the evaluation of paint raw materials, the use of organo-aluminum compounds as driers of alkyd resins, replacements for cobalt and lead as driers, and work on industrial microbiology were discussed. High infrared curing and a survey of instruments for the control of blast coatings were other topics mentioned.

In conclusion, Dr. Anderson expressed great faith in the future of surface coatings, but he predicted that well-informed customers would have a greater influence on the industry.

B.J. ADDENBROOKE, *Secretary*

C-D-I-C

Nov. 12

Lloyd Reindl, of Inland Div. of GMC, spoke on "THE PRODUCTS AND HISTORY OF THE INLAND DIVISION."

Mr. Reindl's talk followed a plant tour of the Dayton, Ohio facility where injection molding and related processes involved in the manufacture of automobile instrument panels were observed.

ROBERT D. THOMAS, *Secretary*

C-D-I-C

Dec. 10

Harry Poth and Herb Fenburr were named Honorary members of the C-D-I-C Society.

Violet Stevens, of Dow Chemical Co., spoke on "THE USE OF METHYLENE CHLORIDE AND 1,1,1,-TRICHLOROETHYLENE AS PAINT SOLVENTS."

Ms. Stevens pointed out that these two solvents offer promise because of their exempt status, due to negligible photochemical activity. Consideration should also be given to them because there already exists an extensive data base regarding their toxicological properties, she said.

ROBERT D. THOMAS, *Secretary*

Detroit

Dec. 11

Dr. Elliott Levy, of Drew Chemical Corp., spoke on "FLASH RUST IN WATER-BASED PAINTS."

After an introduction to the methods of flash rust inhibition, Dr. Levy discussed paint additives formulated to inhibit flash rusting. These additives are for water-borne industrial coatings, are buffered at a Ph of 9-10, and have a shelf life of one to two years, he said.

JOHN J. GENTILIA, *Secretary*

Montreal

Dec. 15

Ray Metzinger, of Atlas Electric Devices Co., spoke on "ACCELERATED WEATHERING: YESTERDAY, TODAY, AND TOMORROW."

After discussing the development of the Weather-Ometer, Mr. Metzinger described the conditions which can be controlled and duplicated by the instrument. He explained that there are three basic light sources: enclosed violet carbon arc lamp; sunshine carbon arc lamp; and the Xenon arc lamp. The Xenon arc lamp exhibited the closest match to sunlight, he said. In addition, this light source is the least affected by line voltage and its energy output can be controlled.

Mr. Metzinger recommended that test cycles (light and dark) should be individually developed to duplicate natural conditions. Evaluations in Weather-Ometers should always include a standard of known outdoor performance, he continued. His presentation concluded with an explanation of a FS-40 (fluorescent sun) device which is rich in UV, to be used when rapid screening is necessary.

Q. Is it possible to judge the performance of a coating solely on the Weather-Ometer results?

A. This is not recommended when the coating is evaluated by itself, however, it can be done when a known standard is included in the evaluation.

Q. Why not boost the light source energy to further accelerate the weathering?

A. This is not a good idea. The acceleration is already obtained because the light source has a constant output comparable to sunlight at noon in June.

Q. Where and how do you recommend the use of FS-40?

A. This instrument should not be used indiscriminately. It is only recommended as a screening process. Known standards should be included at all times.

Q. Is it possible to change the carbon arc lamp for a Xenon arc lamp?

A. Yes, depending on the model and type, as long as it is less than 12 years old.

R. KUHNEN, *Secretary*

Northwestern

Dec. 4

Jack Christie, of Hunter Laboratories, Inc., spoke on "USING COLORIMETERS FOR BATCH COLOR CORRECTION."

Mr. Christie described how Tri-Stimulus Colorimetry can be used to characterize a standard color, and to predict the type and quantity of additional pigment needed to correct an off-color match.

He emphasized that the characterization of the standard color with a colorimeter is tedious and requires a lot of time. Readings are made at 16 different wave lengths for each of the 3 elements of the color scales (L-Black and White, a-Green to Red, b-Yellow to Blue). Similar scales are then developed for variations of the standard color that occur when the quantity of each of the pigments that contribute to the eventual color is varied in step-wise fashion.

A similar plot is established for the batch of paint that is out-of-spec. When this scan is superimposed upon the standard scan, it is possible to determine which pigment needs to be added, and an approximation of the quantity of pigment needed can be made.

G. DALE ERNST, *Secretary*

Pacific Northwest

Dec. 13

Twenty-five year pins were awarded to Doug Dicker, of Dicker-Martens, and Larry Storey, of Walker Brothers.

Bob Nickells, of Argosy Protective Coatings, spoke on "TCC FLASH PAINT."

ART BRAGG, *Secretary*

Philadelphia

Dec. 13

A moment of silence was observed in memory of Dr. Willard A. Madson, retired from E.I. du Pont de Nemours & Co., who died recently.

Among the honored guests in attendance from the Federation staff were Frank J. Borrelle, Executive Vice-Presi-

dent; Tom Kocis, Field Director; and Rosemary Falvey, Director of Administration.

Mrs. Falvey was presented with an onyx desk set and perpetual calendar as a token of appreciation for her twenty years of service.

Mr. Kocis accepted a check of \$500 for the Paint Research Institute, presented by Society Representative, John Stigile.

Mr. Borrelle congratulated the Society of the occasion of its 60th anniversary and acknowledged its many contributions to the paint industry. Citing the accomplishments of individual members, he also recognized the efforts and achievements of various Society committees. Particular attention was given to the many prize winning papers presented by the Society. Mr. Borrelle concluded his presentation by defining the merits of Society membership.

MATTHEW J. HANRAHAN, *Secretary*

Piedmont

Dec. 19

Ruth K. Harmon-Stangs, of Drew Chemical Corp., spoke on "FOAM CONTROL."

Aided by a film, "Foam and Foam Control," Mrs. Harmon-Stangs highlighted new trends in methods of controlling foam in aqueous systems pertaining to the coatings industry. Slides depicting charts of recommended defoamer types used in coatings systems were also presented.

SARA ROBINSON, *Treasurer*

Rocky Mountain

Dec. 10

Dr. Daniel Teitelbaum, of the Center for Toxicology, Man and Environment, Inc., spoke on "THE WORKSMART PROGRAM."

Dr. Teitelbaum defined "Worksmart" as an educational/informational program designed to instruct employees in the proper handling of chemicals used in manufacturing. The effect of some of these chemicals on health is also covered in the program, he said. Dr. Teitelbaum will notify members of the next available "Worksmart" program.

S. CROUSE, *Secretary*

Western New York

Nov. 13

Michael Dunn, of H. Kohnstamm and Co., spoke on "TOXICITY."

Mr. Dunn discussed the many changing governmental regulations which must be met by industry. In California, Rule 66 has been replaced by CARB and, nationally, OSHA and EPA are continually issuing new directives and regulations, he said. According to Mr. Dunn, the

Constituent Society Meetings and Secretaries

BALTIMORE (Third Thursday — Eudowood Gardens, Towson). MITCHELL DUDNIKOV, Harry T. Campell Sons, Executive Plaza IV, Hunt Valley, Md. 21031.

BIRMINGHAM (First Thursday—Calthorpe Suite, Edgbaston). B.J. ADDENBROOKE, Croda Paints Ltd, Bordesley Green Rd., Birmingham B9 4TE, England.

CHICAGO (First Monday — meeting sites in various suburban locations). JOHN L. PETTY, Sherwin-Williams Co. 10909 S. Cottage Grove, Chicago, Ill. 60628.

C-D-I-C (Second Monday — Sept., Nov., Jan., Mar., May in Columbus; Oct., Dec., Feb., Apr. in Cincinnati, Kings Island Inn). ROBERT D. THOMAS, Perry & Derrick Co., P.O. Box 12049, Cincinnati, Ohio 45212.

CLEVELAND (Second Tuesday — meeting sites vary). THOMAS TUCKERMAN, Coatings Research Group, Inc., 2340 Hamilton Ave., Cleveland, Ohio 44114.

DALLAS (Thursday following second Tuesday — Vic's Gallery Restaurant). RAY O. MARETT, Western Specialty Ctg., 4400 Singleton Blvd., Dallas, Tex. 75212.

DETROIT (Fourth Tuesday — meeting sites vary). JOHN J. GENTILIA, Union Carbide Corp., 26500 Northwestern Hwy., Southfield, Mich. 48037.

GOLDEN GATE (Monday before third Wednesday — Sabella's Restaurant, San Francisco). DON MAZZONE, Fuller O'Brien Corp., 405 E. Grand Ave., S. San Francisco, Calif. 94080.

HOUSTON (Second Wednesday — Marriott Hotel, Astrodome). DUANE FIELDS, Conchemco, Inc., 2503 W. 11th St., Houston, Tex. 77008.

KANSAS CITY (Second Thursday—Cordon Bleu). RICHARD WARREN, Cook Paint & Varnish Co., P.O. Box 389, Kansas City, Mo. 64141.

LOS ANGELES (Second Wednesday — Steven's Steak House). ROMER E. JOHNSON, Dorsett & Jackson, Inc., 3800 Noakes St., Los Angeles, Calif. 90023.

LOUISVILLE (Third Wednesday — Essex House). J. DURHAM, Celanese Coatings Co., Technical Center, P.O. Box 8248, Louisville, Ky. 40208.

MEXICO (Fourth Thursday—meeting sites vary). JUAN IRAZABAL, Grafex De Mexico S.A., Calz. Tulyehaulco 4615, Mexico, D.F., Mexico.

MONTREAL (First Wednesday — Bill Wong's Restaurant). R. KUHNEN, Tioxide of Canada Ltd., P.O. Box 580, Sorel, Que., Canada J3P 5P8.

NEW ENGLAND (Third Thursday — Fantasia Restaurant, Cambridge). JOHN E. FITZWATER, Jr., Polyvinyl Chemical Ind., 730 Main St., Wilmington, Mass. 01887.

NEW YORK (Second Tuesday — Landmark II, East Rutherford, N.J.). DONALD E. BRODY, Skeist Laboratories, Inc., 112 Naylon Ave., Livingston, N.J. 07039.

NORTHWESTERN (Tuesday after first Monday—Edgewater Inn). G. DALE ERNST, Tennant Co., P.O. Box 1452, Minneapolis, Minn. 55440.

PACIFIC NORTHWEST (Portland Section — Tuesday following second Wednesday; Seattle Section — the day after Portland; British Columbia Section — the day after Seattle). RICHARD A. STEWART, Stainco, Box 67638, Stn. "O", Vancouver, B.C. V5W 3V1.

PHILADELPHIA (Second Thursday — Valle's Steak House). MATTHEW HANRAHAN, Lilly Industrial Ctg., 1991 Nolte Dr., Paulsboro, N.J. 08066.

PIEDMONT (Third Wednesday — Howard Johnson's Coliseum, Greensboro, N.C.). JAMES N. ALBRIGHT, JR., Lilly Co., P.O. Box 2358, High Point, N.C. 27261.

PITTSBURGH (First Monday — Skibo Hall, Carnegie-Mellon University Campus). RICHARD TRUDEL, Mobil Chemical Co., 1000 Westhall St., Pittsburgh, Pa. 15233.

ROCKY MOUNTAIN (Monday prior to second Wednesday — Gusthaus Ridgeview, Wheatridge, Colo.). STEVE CROUSE, Kwal Paints, P.O. Box 5231, T.A. Denver, Colo. 80217.

ST. LOUIS (Third Tuesday — Salad Bowl Restaurant). JOHN KEMPER, Sinnett Lacquer Mfg. Co., 1378 Kingsland Ave., St. Louis, Mo. 63133.

SOUTHERN (Gulf Coast Section — Second Tuesday; Central Florida Section — Thursday after third Monday; Atlanta Section — Third Thursday). FRANKLIN RECTOR, Indurall Coatings, 3333 Tenth Ave. N., Birmingham, Ala. 35234.

TORONTO (Second Monday — Town and Country Restaurant). G.E. ABBODD, Canadian Industries, Ltd., 1330 Castlefield Ave., Toronto, Ont., Canada, M6B 4B3.

WESTERN NEW YORK (Second Tuesday — Holiday Inn, Cheektowaga, N.Y.). JAY A. ROBEY, Spencer-Kellogg Div., P.O. Box 210, Buffalo, N.Y. 14225.

TOSCA regulations have been especially worrisome for the chemical industry due to the high cost of proving the non-toxicity of new chemical compounds not on the EPA Inventory list.

Mr. Dunn detailed the many specific standards that have been set for various pigments. In addition, limits have been placed on the heavy metal content (e.g. lead and arsenic) in paints, he related. The presentation concluded with a discussion of the regulations which cover the use of pigments, such as carbon black and phthalocyanine blue, in food, drugs, and cosmetics.

JAY A. ROBEY, *Secretary*

Western New York Dec. 11

Sam Bellatierre, of Tenneco, Inc., spoke on "HEAVY METAL ADDITIVES FOR COATINGS."

Mr. Bellatierre explained that many applications of heavy metals in coatings are now prohibited by law. Mercury, for example, is not permitted in solvent-thinned paints. This has led to an extensive search for fungicides and preservatives that are not based on mercury, he said. Mr. Bellatierre reported that several nonmercurial candidates exist, but that none are as effective as those containing mercury.

A similar situation exists with lead

driers which uniquely improve coating gloss and toughness, he continued. A number of drier systems will match the performance of lead driers under laboratory conditions. However, lead is superior when the paint is applied at lower temperatures. According to the speaker, only bismuth approaches the performance of lead at lower temperatures. In baked systems, bismuth and cerium/zirconium give results comparable to lead. Mr. Bellatierre concluded his presentation by relating that, in water-dilutable systems, the speed of dry seems to depend more on the resin than on the drier system.

JAY A. ROBEY, *Secretary*

Future Society Meetings

Baltimore

(Mar. 20)—Manufacturing Committee Seminar on "WASTE MANAGEMENT"

(Mar. 28)—BALTIMORE COATINGS SHOW. Hilton Inn.

(Apr. 17)—Educational Committee program

(May 15)—Technical Steering Committee program on "WATER-BORNE COATINGS"

Cleveland

(Mar. 18)—"WASTE DISPOSAL AND THE ENVIRONMENT"—Speaker to be announced.

(April 15)—Plant Tour—Davies Can Co.

(May 20)—"PAINTING AND DECORATING FROM THE FEMALE POINT OF VIEW"—Spouses' Night.

Detroit

(Mar. 25)—Past-Presidents' Night; "NEW CONCEPTS IN FORMULATION OF GLOSS LATEX COATINGS"—John Bax, of Pacific Scott Bader, Inc.

(Apr. 22)—"DEFOAMING AGENTS"—Eileen E. Reich, of Air Products and Chemicals, Inc.

(May 13)—Joint meeting with Detroit PCA.

Louisville

(Mar. 19)—"PLANT SAFETY"—Gil Kane, of Hercules Incorporated.

(Apr. 16)—FSCT SLIDE PRESENTATION—Federation Officers.

New York

(Mar. 11)—Mini Workshops: "INTRODUCTORY COATINGS CHEMISTRY AND FILM FORMATION"; "RELATION BETWEEN STRUCTURE OF POLYMERS AND PROPERTIES"; "PAPERWORK IN A PAINT FACTORY" (panel discussion); and "PUMPS."

Northwestern

(March 4)—Annual Symposium.

Chicago

(Mar. 3)—Society Manufacturing Committee Program; and "INTERFACIAL CHEMISTRY IN CORROSION-INDUCED PAINT ADHESION LOSS"—Ray Dickie, of Ford Motor Co.

(April 7)—Society Technical Committee Program; and "EVAPORATION BEHAVIOR OF ORGANIC CO-SOLVENTS IN WATER-BORNE COATINGS"—R. F. Eaton, of Union Carbide Corp.

Pittsburgh

(Mar. 3)—"SUCCESS FOR THE PAINT CHEMIST"—Milt Glaser, of Midland Div., Dexter Corp.

(Apr. 7)—"NONLEAD PIGMENTS FOR CORROSION CONTROL"—Clive Hair, of Clive Hair, Inc.

(May 5)—"SYMPOSIUM ON GOVERNMENT AND COATINGS" (one-half day programs).

(June 2)—"CONSUMERISM"—Jean Fox.

C-D-I-C

(Mar. 10)—Society Technical Committee Program.

(Apr. 14)—"NEW CONCEPTS IN THE FORMULATION OF GLASS LATEX COATINGS"—John Bax, of Pacific Scott Bader, Inc.

(May 12)—"MISCIBILITY CHARACTERISTICS OF SOLVENT/WATER MIXTURES"—Larry Culver, of Eastman Chemical Co.

Montreal

(Mar. 5)—JOINT MEETING with the Quebec Paint Industries Association.

(Apr. 2)—Speaker to be announced.

(May 7)—"GOVERNMENT SPECIFICATIONS IN THE PAINT INDUSTRY"—Joe Roberge, of B.N.Q., and Frank Eadie, of C.G.S.B.



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 KAIL, JAMES E.—DeSoto, Inc., Des Plaines, Ill.
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 MAU, THOMAS—Cargill, Inc., Dolton, Ill.
 NIEMIEC, MATTHEW L.—Rockford Chemical Coatings, Rockford, Ill.
 ONI, MATTHEW A.—Allans Paint Co., Chicago.
 PFOHL, RAYMOND R.—The Enterprise Companies, Wheeling, Ill.
 ROBERTS, JACK F.—Enterprise Chemical Coatings, Chicago.
 SCHMID, STEVEN R.—DeSoto, Inc., Des Plaines.
 SCHNEIDER, WARREN—Cargill, Inc., Carpentersville.
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 THIELMANN, LEO—Air Products Chemical, Hillside, Ill.
 WEILAND, RONALD E. JR.—Cargill, Inc., Carpentersville, Ill.

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Associate

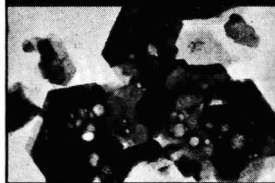
ACKERMAN, H. D.—Ack Lac-Ack Sales & Chemical Co., North Kansas City, Mo.

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 JOSE L. ARAMIZ—Pinturas Siglo S.A., Snadalajara, Jalisco.
 JOSE VERA BARRERA—General Paint Co., Tlalnepantla, Edo De Mexico.
 ROBERTO BATALLAR G.—Isomex S.A., Mexico, D.F.

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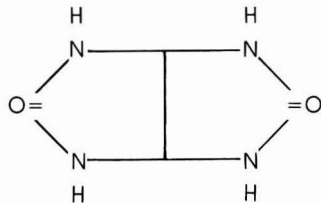
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BERUARDO MARTINEZ M.—Pinturas Pittsburgh, Mexico Distritofederal, Mexico.
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JOSE L. PADILLA DE A.—International Paints, Mexico.
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PRISCILIANO ZARAGOZA—Productos Rival, S.A. Guadalajara, Jalisco Mexico.

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JUAN MANUEL CAMPO B.—Ciba-Geigy Mexicana, Mexico, D.F.
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FRANCISCO RESENDIZ RICO—Cia. Sherwin-Williams, S.A., Mexico.
MA. DEL CARENEN VALDEZ—Colorquim S.A. De C.V., Mexico.

NORTHWESTERN

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JOHNSON, BRUCE A.—Valspar Corp., Minneapolis.
BRYAN, G. BRUCE JR.—Glidden Company, Minneapolis.

- ERICKSON, CHARLES J.—Conwed Corp., Cloquet, Minn.
MADY, ROBERT E.—Stewart Paint Co., Minneapolis.
MCGEE, KATHRYN L.—Valspar Corp., Minneapolis.
OLSON, ROBERT J.—Valspar Corp., Minneapolis.
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QUANDAH, RUTH A.—3M Corp., Prairie du Chien, Wis.
STENE, MARK D.—Sherburn Paint Corp., Sherburn, Minn.

Associate

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CAMPBELL, RICK—American Hoechst, Elk Grove Village, Ill.
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KARLSTAD, RICHARD H.—Whittaker Corp., Minneapolis.
LARSON, GARY C.—Worum Chemical, St. Paul, Minn.
MILLS, EDWARD N.—Minnesota Solvents, Roseville, Minn.
POWELL, WILLIAM T.—NL Chemicals, Blomington, Minn.
TUINENGA, JOHN P.—Frost Paint and Oil, Minneapolis.
WIRTH, JOSEPH R.—Midway Can Co., St. Paul.

Educator and Student

- CULHANE, WILLIAM—P&C Dept. NDSU, Fargo, N.D.
JESSEPH, STEVEN P.—North Dakota State University, Fargo.
PATEL, PARMANAND P.—North Dakota State University, Fargo.
VETTER, GLEN O.—Polymer-Coatings, Fargo.
WATSON, BEN C.—North Dakota State University, Fargo.

PIEDMONT

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- DRUM, JAMES A.—Nation Wide Research, Durham, N.C.
GREENGARD, RICHARD—Reliance-Universal, Inc., High Point, N.C.
JARVIS, JOHN LEE JR.—The Lilly Co., High Point.
PLYLER, MICHAEL HULL—Reliance-Universal, Inc., High Point.
SHORE, GARY E.—Chemical Coatings, Inc., Hudson, N.C.
SMITH, CALVIN—Reliance-Universal, Inc., High Point.

Associate

- BEHAN, JOHN M.—A. B. Kohl Sales, Newport News, Va.
HARPER, RICHARD—Drew Chemical Corp., Clemson, S.C.
SPEIR, RICHARD O.—Columbia Div., Atlanta, Ga.
WEST, EDWARD C.—General Steel Drum, Charlotte.

Proposed Amendments To Federation By-Laws and Standing Rules

The following proposed amendments to the By-Laws and Standing Rules of the Federation of Societies for Coatings Technology will be presented at the Board of Directors meeting on May 16, 1980, in Minneapolis.

The proposed change to By-Laws Article III, Section B, will be presented for first reading. If approved, it will then be presented for adoption at the Board of Directors meeting on October 28, 1980, in Atlanta.

Proposed changes to the Standing Rules require one reading only. Therefore, the revision to SR VII, Section A, will be presented for adoption.

The proposed addition of Section F to By-Laws Article III received first reading at the Board meeting on October 2, 1979, and will be presented for adoption on May 16.

ARTICLE III—Organization Section B, Paragraph (1)

WHEREAS the Federation's Executive Committee passed the following action on October 6, 1979:

"That the President, or duly appointed representative, and the Research Director of PRI submit progress reports to and attend the two meetings of the Board of Directors and further that this statement be made a provision of the Standing Rules and be referred to the By-Laws Committee for preparation of the proper resolutions," be it

RESOLVED that By-Laws Article III, Section B, Sub-Paragraph n, be revised as follows [additions in *italic*, deletions in parenthesis ()] :

- (n) Receive written reports on their activities from the Federation officers, (and) Committee Chairmen, and both the President, or duly appointed representative, of the Paint Research Institute and its Research Director semiannually; and from Constituent Societies annually.

Comment: The By-Laws Committee favors this revision.

ARTICLE SR VII—Open Meetings Section A, Paragraph (2)

WHEREAS the Federation's Executive Committee passed the following action on October 6, 1979:

"That the President, or duly appointed representative, and the Research Director of PRI submit progress reports to and attend the two meetings of the Board of Directors and further that this statement be made a provision of the Standing Rules and be referred to the By-Laws Committee for preparation of the proper resolution," be it

RESOLVED that Standing Rules Article SR VII, Section A, Sub-Paragraph (2) be revised as follows [additions in *italic*, deletions in parenthesis ()] :

- (2) The President of the Federation shall extend an invitation to appropriate officers of the Canadian Paint Manufacturers Association, the Mexican Association of Paint & Ink Manufacturers, the National Paint & Coatings Association, (and the Paint Research Institute) and both the President of the Paint Research Institute and its Research Director to attend meetings of the Board. The President may also invite officers of other organizations which he deems to be appropriate.

Comment: The By-Laws Committee favors this revision.

ARTICLE III—Organization New Section F

WHEREAS counsel has recommended that a By-Law be adopted authorizing the Federation to indemnify its officers and directors, and

WHEREAS the Board of Directors at its January 28, 1978 meeting requested the By-Laws Committee to offer a resolution which would provide such a statement, be it

RESOLVED that a new section "F" be added to Article III of the By-Laws which shall read as follows:

"F. INDEMNIFICATION OF DIRECTORS, OFFICERS AND OTHERS

The Federation shall indemnify, to the extent determined by the Board of Directors, any person who, by reason of serving as a director, officer, employee, or agent of the Federation is made a party to any legal, administrative, investigative or similar proceeding. Indemnification may be against all or a portion of any amounts such person is required to pay (including expenses) as a result of such a proceeding. This right of indemnification shall not deprive such person of any other rights of indemnification to which a person may be entitled and shall inure to the benefit of such person's heirs, executors and administrators."

Technical Articles in Other Publications

Compiled by the Technical Information Systems Committee—H. Skowronska, Chairman

Farbe und Lack (in German)

Published by Curt R. Vincentz Verlag, Schiffgraben 41-43,
Postfach 6247, 3000 Hannover 1 Germany

Vol. 85 No. 10

October 1979

- Monitor—"Comments on the Situation in the Paint Industry"; 819.
Kollek, H. and Brockmann, W.—"Simple Methods for Measuring the Adhesive Properties of Metallic Surfaces (Reflectance Photometry)"; 820-824.
Rieche, G.—"Plastic Coatings for Covering Cracks in Mineral Building Materials"; 824-831.
Weder, J.—"Automatic Determination of Pot Life of Two-Component Systems"; 832-834.
Helmen, T. and Hess, E.—"Importance of Suntest Apparatus for Short Term Weathering of Lacquers"; 835-841.
Brushwell, W.—"Epoxy Resin Technology" (Literature review); 842-846.
Muller, W.—"Value Analysis—A Method for Determining Quality of Paints and Varnishes"; 846-847.
German Standard Draft DIN 53 181: Binders for Paints and Varnishes; Interval of Resins by the Capillary Method; 854-855.
German Standard Draft DIN 55 954: Binders for Paints and Varnishes; Test of Miscibility of Resins; 856.
German Standard Draft DIN 55 955: Binders for Paints and Varnishes; Test of Solubility and Diluting Properties of Resins and Resin Solutions; 857-858.

Vol. 85 No. 11

November 1979

- Florus, G.—"Aqueous Dispersion Paints and Plasters for Exteriors"; 913-915.
Funke, W. and Handloser, G.—"Flow of Water-Borne Paints during Application. Influence of Temperature and Air Humidity"; 916-919.
Ladstadter, E. and Gessner, W.—"Quantitative Determination of Luminous Reflectance, Flow Quality and Sheen with GR-COMP-Gonioreflectometer"; 920-924.
Schneider, W.—"Thermoanalytical Tests and Properties of Solvent-Free Coatings"; 925-929.
Bar, R.—"Lacquers Used for Coating Absorbers of Solar Panels"; 930-932.
Herz, C.P. and Eichler, J.—"New Photoinitiators for UV Ray Curing"; 933.
Brushwell, W.—"Technical Developments in Electro-dip Coating"; 934-937.
Blazevac, A.—"Punched Card Controlled Synthetic Resin Plant"; 938-940.
German Standard Draft DIN ISO 3248: Paint and Varnishes; Determination of effect of heat; p. 945.
German Standard Draft DIN 5033 Part 9: Colorimetry; White Standard for colorimetry and reflectometry; p. 944.

Paint Manufacture and Resin News

Published by Wheatlands Journals Ltd., 157 Hagden Lane,
Watdord WD1 8LW, U.K.

Vol. 49 No. 1

January/February 1979

- Feyt, L. and Bauwin, H.—"Action Mechanisms of Flow Modifiers"; 18, 20-1, 23.
Boxall, J. and von Fraunhofer, J.A.—"Mathematics for Paint Technologists, Part III"; 24, 26.
Ellinger, M.L.—"R & D Report—Anticorrosion and Marine Paints (Literature review)"; 27-28, 31.

Vol. 49 No. 2

March 1979

- Ramsbotham, J.—"Solvent System Design for Epoxy Resin. Part I"; 26-29.
Boxall, J. and von Fraunhofer, J.A.—"Mathematics for Paint Technologists. Part IV"; 41-42, 44.

Vol. 49 No. 3

April 1979

- Ramsbotham, J.—"Solvent System Design for Epoxy Resin, Part II"; 18, 20-21.
Bedford, D. and Mason, C.R.—"Coal Tar Products in Protective Coatings"; 22-24.

Vol. 49 No. 4

May 1979

- Boxall, J. and von Fraunhofer, J.A.—"Mathematics for Paint Technologists. Part V-1"; 17, 35.
"Trends in Adhesive Technology" (Based on presentation by D.G. Coe); 19-22.
Hirzel, W.—"Cleaning Tanks and Vessels"; 33-35 (First published in Seifen, Ole, Fette, Wasche)

Vol. 49 No. 5

June 1979

- Ferguson, J.P.—"Trends in Formulating Paints and Printing Inks"; 13, 33.
Ellinger, M.L.—"Accelerated Weathering (Literature review)"; 22-23.
Kinmonth, R.A. and Norton, J.E.—"Spectral Energy Distribution and Degradation of Organic Coatings"; 25-29.
Boxall, J. and von Fraunhofer, J.A.—"Mathematics for Paint Technologists. Part V-2"; 34-35.

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Krishnamurti, N., Shirsalker, M.M., and Suisamban, M.A.—“Epoxy-based Primers”; 11,13,16.

Anon.—“Water and Effluent Treatment in Paint Industry”; 15-16.
 Androm, R.T.—“Resin Plant Problems and Some Solutions”; 27-29.

Summaries of talks presented at June 1979 Conference of Paintmakers Association: Howd, G.T.—“Raw Materials—Outlook for Supply and Prices”; 9. Kerr, P.—“Titanium Dioxide—Outlook for Supply and Prices”; 10. “Economics Force Changes in the Pigment Industry”; 11,13.

Boxall, J. and von Fraunhofer, J.A.—“Mathematics for Paint Technologists. Part V-3”; 17,18.

Anon.—“Expanding Role for Epoxy Resins”; 10,12.

Green, J.R.—“Effects of Oil Prices and Toxicity Laws (on U.K. synthetic resin industry)”; 13-14, 16.

Topp, J.—“Water-Based Synthetic Polymers for Adhesive Applications”; 18,20.

Double Liaison — Chimie des Peintures (In French)

Published by EREC, 68 Rue Jean-Jaures, 92800 Puteaux, France

Kresse, W.—“Market Analysis—Trends for Production and Use of Carbon Black in Europe and United States of America”; 30-36.

Sebban, G.—“Use of Dispersions in Non-polluting Coatings”; 43-47.
 Kondrachoff, W.—“Ecological Problems in Relation to the Sale of Pigments”; 49-54.

Journal of the Oil and Colour Chemists' Association

Published by Oil and Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex, HA0 2SF England

Rascio, V., Giudice, C.A., Benitez, J.C., and Presta, M.—“Ship's Trials of Oleoresinous Antifouling Paints. II. Formulations with Medium and Low Toxicant Contents”; 282-292.

Sreeves, J.E. and Whitfield, L.—“Epoxy/Polyester Powder Coatings. Economic and Technical Considerations in their Usage”; 293-298.
 Vorster, O.C.—“Emulsion Paints in Building”; 299-302.

Dulligal, E.A.—“Consultation before Painting Avoids Litigation After”; 303-308.

Harrison, J.B.—“Challenge to Coatings in a Changing World: Predestination of Prophesy?”; 331-336.

Walker, P. and Haighton, A.—“Surface Coatings for the Nuclear Industry”; 337-350.

Atherton, D.—“Original and Maintenance Painting Systems for North Sea Oil and Gas Platforms”; 351-358.

Ramsbotham, J.—“Solvent Loss from Paint Films”; 359-365.

Luthardt, H.J. and Burckhardt, W.—“Water-Borne Industrial Thermo-setting Systems”; 375-385.

Egerton, T.A. and King, C.J.—“Influence of Light Intensity on Photoactivity in TiO₂ Pigmented Systems”; 386-391.

Percy, E.J. and Nouwens, F.—“Aqueous Coil Coatings”; 392-400.
 Varley, D.M. and Bower, H.H.—“Dispersion of Pigments: What Is the Ultimate?”; 401-409.

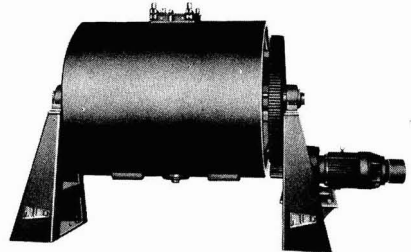
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XVth FATIPEC Congress in Amsterdam, June 8-13, to Feature Papers On Coatings Industry Problems in Ecology, Energy, and Economy

The XVth Congress of FATIPEC will be held from June 8-13, 1980, at the International Congressentrum RAI, in Amsterdam, Netherlands. (FATIPEC is the Association of Technicians in the Paint, Varnish, Lacquer, and Printing Ink Industries of Continental Europe).

The theme of the Congress is "Activities of the Coatings Industry in the Framework of Ecology, Energy, and Economy Problems" and papers presented during the four days of program sessions will address this subject. The program is as follows:

PLENARY AND SELECTED PAPERS

"Imide-Structure Resins in Electrostatic Spray Powder Coatings"—P. Pappardo, S. Vargiu (Italy).

"Prevention of Environmental Pollution: a Contribution by the Dutch Paint and Printing Ink Industry"—T. Doorgeest, R. Cirkel (Netherlands).

"Role of Titanium Dioxide in the Protection of Paint Media"—R.R. Blakely (United Kingdom).

"Ecological Implications and Energy Requirements Resulting from the Development of Titanium Dioxide Pigments"—R.S. Darby (United Kingdom).

"How the Titanium Oxide Industry Can Help the Coatings Industry Save

Energy"—J.M. Rackham (United Kingdom).

"Environmentally Acceptable and Energy Efficient Epoxy Coatings"—R.S. Bauer. (Mr. Bauer is associated with Shell Development Co., Houston, and will present this paper on behalf of the Federation).

"Governmental Pollution Regulations in Sweden and their Consequence in the Coatings Industry"—M. Winell (Scandinavia).

"Ecological Demands for New Marine Paints"—N.A. Ghanem (Egypt).

"Technological Market Segmentation: The Challenge of the 1980's"—H. Ellerehorst, G. Ader (United Kingdom).

Also papers by: R. Sussmeyer (Belgium); C.M. Bruneau (France); A. Lorine (Hungary); I. Tonini (Switzerland); and L. Dulog (West Germany).

DISCUSSION PAPERS

Ecology, Environmental Issues

"Modern Developments in Testing and Formulation of Can Coatings"—E.M.A.A.J. van Acker (Netherlands).

"Solvents and Environmental Problems Raised by Oven-Cured Paints and Varnishes"—M. Motoiu (Roumania).

Also papers by: E. Knappe (Switzerland); K. Mahner (West Germany); J.

Sickfeld (West Germany); F. Lucherini, and Z. Mihalko (Soviet Union).

Ecology, Waterborne Coatings

"Action of Surfactant in Pigment Dispersion"—N. Kawashima, O. Hiruta, K. Meguro (Japan).

"Some Pigmentation Effects in Latex Paints"—S. Skledar (Yugoslavia).

"Gloss-Emulsion Paints based on TiO₂ Pigments Precoated with Polymeric Dispersants"—J.H. Bieleman, Z. Stark, A. Capelle, J. Knoef (Netherlands).

"Cationic Autophoresis, a New Possibility"—J. Kunnen (Netherlands).

"Place of Polybutadienes in Cathodic Electrocoating Paints"—A. Toth (France).

"Influence of the Hydroxylic Groups Content of an Oleophenolic Binder on the Properties of Primers, Non-Pollution, Applied by Electrosetting"—T. Domide, T. Pintilie, R. Serban (Romania).

"Reactive Solubilizers for Acid Functional Water Soluble Polymers"—F.T. Klein, M.E. Woods (West Germany).

Also papers by: K. Gabert, A. Vigouroux (France); B. Pepin (France); H.J. Traenckner (West Germany); R. Zimmerman (West Germany); C. Robu (Romania); J. Parmentier; and J.C. Laout (France).

Ecology, High Solids Coatings

"Functional Group Analysis and Curing Reactions of Methoxylated Melamine Formaldehyde Crosslinkers"—J.H. Van Dijk, W. Dankelman, C.J. Groenboom (Netherlands).

"High Solids Industrial Coatings"—F.Th. Nouwens (Netherlands).

"Higher Solids in Automotive Top-Coats"—I. Wagstaff (Netherlands).

"Retention Properties of Organic Coatings"—P. Alessi, I. Kikic, and G. Torriano (Italy).

Also a paper by C. Bertrand (France).

Ecology, Powder Coatings

"Influence of Resin Composition on Automotive Powder Surfactant Properties"—A. van de Werff (Netherlands).

"Dry on Wet—a New High Solids, Low Polluting Coating System"—J.A. Hewson, R.W. Mahar, B.P. Windibank (United Kingdom).

(Continued on page 101)

Industry Seminar to Focus on Colorimetry, March 4-6

"Colorimetry and Optics of Pigmented Systems" is the topic of a comprehensive two-and-one-half day, non-commercial seminar offered by the Colorimetry Laboratory Pigments Dept., CIBA-GEIGY Corp., on March 4-6 at the Marriott Hotel, Columbus, Ohio.

The seminar will provide practical information for objective evaluation of the optical behavior of pigments in various materials. It is designed to acquaint students with the application of the science of colorimetry and color measurement and the theories of colorant formulation and pigment dispersion to the solutions of typical problems encountered in the industry.

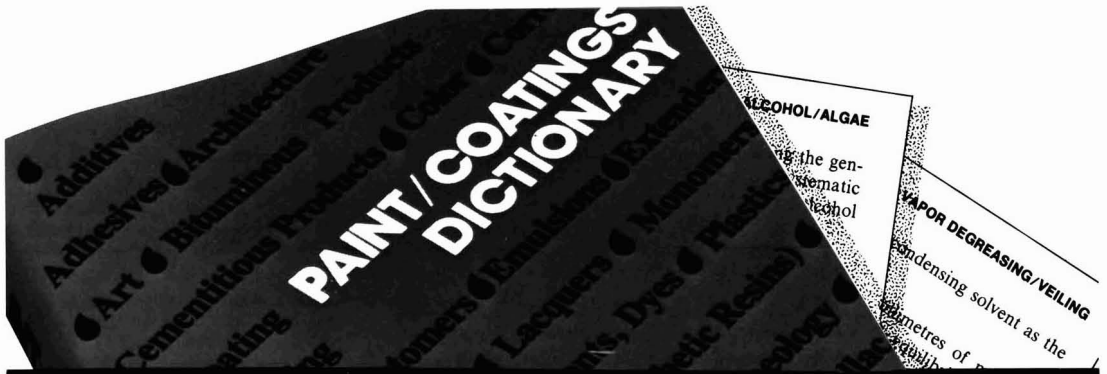
Speakers Dennis Osmer, of CIBA-GEIGY's Pigment Dept., and Ruth Johnston-Feller, a consultant to the firm, will place emphasis on "how to" ways and methods for improving product quality,

increasing productivity, and decreasing costs.

Attendees, previously ranging from division vice-presidents to new technicians, generally have moderate exposure in the application fields, but not in the science of color. Of particular interest to students are the evening sessions, which provide opportunities to receive assistance on individual special application problems, as well as particular color problems or areas.

Tuition cost is \$185, which includes textbook, notebooks, instruction, and luncheons. Participants are responsible for hotel and transportation arrangements and cost.

For additional information, contact Dennis Osmer, CIBA-GEIGY Corp., Colorimetry Laboratory, Pigments Dept., Ardsley, N.Y. 10502.



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technicians in all the coatings-related fields.

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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)		\$
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1980 FATIPEC Congress to Look at Ecology, Energy, Economy

(Continued from page 98)

"On the Formation of Two-Phase Coatings From a Mixture of Powdered Polymers"—H. Murase, Prof. W. Funke. (West Germany).

"Continuous Production of Powder Coatings"—F. Erben. (West Germany).

Also papers by: R. Dhein (West Germany); H.F. Lauterbach (Switzerland); H. Riemer (West Germany); and K.M. Oesterle (Switzerland).

Economy by Materials Saving

"Increased Durability in Aircraft Coatings by the Use of Pigmented Polymer Beads"—G.P. Bierwagen. (Sherwin-Williams Co., Chicago).

"Penetration of Stains and Drying Oils into Softwood"—J. Koskelainen, B. Lindberg. (Denmark).

"Effect of Transparent Iron Oxide on the Mechanical and Thermomechanical Properties of Transparent Finishes for Wood during Natural and Artificial Weathering"—K. Zabel. (Netherlands).

"Improved Protection of Structural Steelwork"—N.R. Whitehouse. (United Kingdom).

"Cathodic Protection of Coated Mild Steel in Sodium Chloride Solutions"—J.D. Scantlebury, D.A. Eden, M.J. Schofield. (United Kingdom).

"Some New Aspects in the Relation Between Pigment and Binder in Corrosion Protecting Coating Systems"—A. Koopmans. (Netherlands).

"New Rust-Inhibiting Paints of Low Toxicity"—G. Torriano, A. Papo. (Italy).

"Methods of Measuring Oxygen Permeability of Organic Coatings"—Prof. W. Funke, H. Zatloukal, B. Harfelder, R.D. Flaig. (West Germany).

"Role of Surface Morphology on Paint Adhesion to Aluminum Substrates"—J.J. Mills, K.C. Holub. (Martin Marietta Corp., Baltimore).

"An Attempt to Correlate the Electrochemical Properties and the Adhesion on Steel of Some Macromolecular Films"—H. Jullien, Prof. W. Funke, U. Zoril. (West Germany).

"New Possibilities in Offshore Maintenance"—L.A. Kik. (Netherlands).

Also papers by: G. Kruska (Switzerland); M. Piens (Belgium); and L. Capeller (West Germany).

Economy in Research

"The Innovation Indices—Models to Improve the Effectiveness of R&D Laboratories"—Milton A. Glaser. (Dexter Corp., Waukegan, Ill.)

"Information, a Resource for the Coatings Industry"—P. Houmes (Netherlands).

Also papers by: R. Ammann, E. Edelmann, A. Helynck, and F. Rosendahl. (Netherlands).

Energy Saving

"UV Curing Resins for Tinplate Decoration"—A. van Neerbos, C.A.M. Hoefs, E.A. Giezen. (Netherlands).

"Properties and Applications of UV Dual Cure Systems"—A. Noomen. (Netherlands).

"Study of Initiation Systems Enabling the Curing of Unsaturated Polyester Resins by Visual Radiation"—Prof. J. Mleziva, V. Cermak. (Czechoslovakia).

"Dynamic Thermomechanometry of Networks from Acrylated Epoxy Resins"—G. Giuliani, A. Priola. (Italy).

"Dispersion—the Neglected Parameter"—W. Carr. (United Kingdom).

"Rheological Properties of Mill-Bases and Energy Consumption in Dispersing Operation"—R. Lapasin, D. Mallamaci, E. Medaglia. (Italy).

"Optimal Efficiency of Titanium Dioxide Mill Base Formulation"—F. Biglieri. (Italy).

"Influence of the Pigment Binder Ratio on Efficient Paint Milling"—H.J. van Der Stoep, R. J. Hoogenhout. (Netherlands).

Also papers by: E. Haering (West Germany); H. Valot (France); J.P. Aubert (France); H. Wilde (West Germany), B. Medinger (Switzerland) and P. Mennesson (France).

Inks

"Oil Beads for Publication Inks: An Energy and Economy-Efficient Alternative"—E.J. Queen. (Columbian Chemicals Div., Tulsa).

"Interaction between Damping Solution and Printing Ink Raw Materials"—C. van Esch. (Netherlands).

GENERAL INFORMATION

Registration

To register for the Congress, please obtain a registration form from the Congress Secretariat at the following address: XVth FATIPEC Congress, c/o Organisatie Bureau Amsterdam BV, Europaplein 14, 1978 GZ Amsterdam, Netherlands. The fees are Dfl. 550 (approx. \$275) for participants and Dfl. 200 for spouses. The Secretariat will also arrange for hotel reservations for which a deposit of Dfl. 200 is required.

Exhibition

During the Congress an international exhibition, "Formula '80," will be held in the adjacent RAI exhibition halls. No

admission charge for Congress participants.

Social Program

An informal welcoming reception will be held at the RAI center on Sunday evening. An official reception will be hosted by the municipal authorities of Amsterdam and the Ministry of Economic Affairs on Tuesday evening.

Wednesday will feature an all-day excursion to the Zaanse Schans, an area with clusters of windmills and old wooden houses.

The Congress will close on Friday evening with a dinner dance at the RAI center.

For the spouses there will be Monday, Tuesday, and Thursday tours of Amsterdam, Gouda and Schoonhoven, and the Aalsmeer Flower Auction and The Hague.

Organization

The organization of the XVth Congress is the responsibility of the Dutch FATIPEC member association, N.V.V.T.

Dr. M.F. Kooistra is the 1978-80 President of FATIPEC. In this capacity he also serves as the President of the Congress and the Technical - Scientific Committee. A.M.C. Kalmeijer is the President of the Organizing Committee. The address of the Congress Secretariat is given under "Registration" above.



M. F. Kooistra

Lehigh University to Hold Corrosion Course in May

A short course, "Corrosion Principles and Their Application to Protection Coatings," will be offered by Lehigh University, Bethlehem, Pa., May 19-23.

The course is designed for industrial scientists and engineers who are concerned with controlling corrosion or designing products that must withstand a corrosive environment. Special emphasis will be placed on corrosion control through organic or metallic coatings.

The registration fee, \$595, covers all program activities and materials.

Information concerning the course is available from Dr. Henry Leidheiser, Director, Center for Surface and Coatings Research, Lehigh University, Bethlehem, Pa. 18015.

Cleveland Society Stages Conference on Coatings Advances in March

The Education Committee of the Cleveland Society for Coatings Technology will present its 23rd Annual Technical Conference, "Advances in Coatings Technology," March 24-25, at Baldwin-Wallace College in Berea, Ohio.

According to Chairman George R. Pilcher, the conference will be divided into four half-day symposia: "Recent Developments in Deposition Technology"; "High-Performance Technologies"; "Corrosion Concepts and Analysis"; and "Low Temperature/Ambient Cure."

The first morning session, "Recent Developments in Deposition Technology," will be chaired by Carl J. Knauss, of Kent State University; the afternoon session, "High-Performance Technologies," by Charles K. Beck, of Premier Industrial Corp.

During the second day, the morning session, "Corrosion Concepts and Analysis," will be chaired by Dr. Theodore Provder, of Glidden Div., SCM Corp.; and F. Louis Floyd, of Glidden, will chair the afternoon session, "Low Temperature/Ambient Cure."

The following papers will be presented:

March 24

Recent Developments in Deposition Technology

"Vacuum Deposition: State of the Art and Future Challenges"—T.R. Riley, of Parmat, Inc.

"Electrodeposition: A Total System"—L.L. Foy, of Sherwin-Williams Co.

"Cathode Reactions and Metal Dissolution in Cationic Electrodeposition"—D.G. Anderson and E.J. Murphy, of DeSoto, Inc.

Symposium on High-Performance Technologies

"High Temperature Polymers for Heat-Resistant Coatings"—Dr. N. Bilow, of Hughes Aircraft Corp.

"Ceramic Coatings"—Dr. G.J. Roberts, of Ferro Corp.

"Coatings for Plastics"—M.L. Pollock, of Bee Chemical Co.

"The Theoretical and Practical Factors Regarding Foam Control in Water-

Borne Coatings"—E.M. Antonucci, of Drew Chemical Corp.

"Amezedrine: Milestone or Millstone?"—Dr. K.L. Scott, of Case Western Reserve University.

March 26

Symposium on Low Temperature/Ambient Cure

"Autooxidation and the Role of Metal Catalysis"—P.A. Kuschner, of The Sherwin-Williams Co.

"How Low Can You Go?"—Dr. P.J. Schirmann, of American Cyanamid Co. "Quantitative Reaction Kinetics via Thermal Analysis"—T.H. Grentzer, R.M. Holsworth, and Dr. T. Provder.

"Sulfonate Ionomers: A New Approach to Polymer Properties"—R.D. Lundberg, of Exxon Research and Engineering Co.

Additional information can be obtained from the Education Committee Chairman, George R. Pilcher, The Sherwin-Williams Co., 601 Canal Rd., Cleveland, Ohio 44113.

Paint Research Institute Programs Supported by 1979 Contributions

Additional funds for the 1979 research projects of the Paint Research Institute, received during the fourth quarter, supplemented funds committed by the Federation in support of PRI research efforts.

1979 Contributors to PRI

Societies

Baltimore; Chicago; C-D-I-C; Cleveland; Dallas; Detroit; Golden Gate; Houston; Kansas City; Louisville; Montreal; Pacific Northwest; Philadelphia; Piedmont; St. Louis; Southern; and Toronto.

Corporations and Foundations

Body Bros., Inc.; M.F. Cachat Co; Canada Colors & Chemicals Ltd.; Canadian Industries Ltd.; Dar-Tech, Inc.; Davies Can Co.; DeSoto, Inc.; Diamond Shamrock Corp.; Dominion Colour Co. Ltd.; Dow Chemical Co.; Drew Chemical Corp.; Ferro Corp.; Gulf Canada Ltd.; Harshaw Chemical Co.; Inmont Canada Ltd.; Inmont Corp.; International Minerals & Chemicals Corp.; Jamestown Paint & Varnish Co.; Kalcor Coatings Co.; L.V. Lomas Chemical Co. Ltd.; Lord Corp.; Mameco International; McNalley-Webber Co.; Midland Div., Dexter Corp.; Mobil Chemical Canada Ltd.; Monsanto Co.; Benjamin

Moore & Co. Ltd.; N L Industries Foundation; Nacan Products Ltd.; Ohio Solvents; Palmer Supplies of Cleveland; Pfizer, Inc.; The Q-Panel Co.; Reichhold Chemicals Ltd.; Rohm and Haas Co.; Rust Oleum Corp.; SCM Corp. (Glidden Coatings & Resins Div.); Seegot, Inc.; Selectone Paints Ltd.; Shell Chemical Co.; Silberline Manufacturing Co., Inc.; Donald McKay Smith, Inc.; Tioxide of Canada Ltd.; Tremco, Inc.; Union Carbide Corp.; and Henry Reichhold Foundation.

Associations

Ontario Paint Association.

Individuals

William Asbeck; James W. Broggini; William Dunn; Ross Galati; Cynthia Kress; Raymond R. Myers; Fred G. Schwab; Betty Sharpe; Kenneth C. Waldo; and Anonymous.

Mildew Consortium

Buckman Laboratories, Inc.; Cosan Chemical Corp.; E.I. du Pont de Nemours & Co., Inc.; National Paint and Coatings Association; PPG Industries, Inc.; SCM Corp. (Glidden Coatings & Resins Div.); and Troy Chemical Corp.

Southern Society to Hold Annual Meeting in Atlanta

"Personal Growth in Coatings Technology," will be the theme during the annual meeting of the Southern Society for Coatings Technology at the Terrace Inn, Atlanta, Ga., March 12-14.

The program will include motivational talks on coping with stress, self-improvement, positive thinking, estate planning, and human relations. Panels of industry experts will also discuss production problems and internal and external purchasing relationships.

The University of Southern Mississippi will conduct a day-long seminar on

Coatings Fundamentals, including such topics as "Formulating with Water-Borne Vehicles," "Experimental Design," and "Test Methods and Instrumentation."

The annual meeting pre-registration (prior to March 1) is \$57.50; spouses' registration is \$20. Registration for the fundamentals seminar on March 12 is \$20.

For additional information, or to register, please contact Peter F. Decker, Union Carbide Corp., 17 Executive Park Dr., N.E., Atlanta, Ga. 30359.

NDSU to Sponsor Coatings Science Courses in June

The Department of Polymers and Coatings of North Dakota State University, Fargo, N.D., will offer two two-week courses in Coatings Science in June 1980 for chemists in the coatings and allied industries.

(June 2-13)—“Basic Coatings Science”—Designed for the person with narrow experience in the coatings industry, this course covers basic principles. It will be taught on a level of a senior college course, and requires at least two years of college chemistry.

(June 16-27)—“Advanced Coatings Science”—Covering the scientific principles of importance in high solids, waterborne, radiation cure coatings, this course will relate these to the practical problems of the industrial coatings scientist. Since the course will be taught on a graduate school level, applicants should have substantial experience in the coatings or related field, a strong background in chemistry, or have participated in the Basic course.

For additional information, contact Dr. Zeno W. Wicks, Jr., Polymers and Coatings Dept., North Dakota State University, Fargo, N.D. 58105.

PPG Offers Fellowships to NDSU

PPG Industries, Inc. will fund a research fellowship program at the Polymers and Coatings Dept. of the North Dakota State University, for undergraduate chemistry majors. Applications will be accepted from junior year students interested in entering the NDSU graduate program after completing their undergraduate degree.

Recipients will carry out organic or physical chemistry research related to the basic scientific problems of the coatings industry. The \$1200 stipend will cover the 10 weeks of the summer of 1980. Such funding is intended to illustrate the interest of industry in increasing the availability of graduates for future employment.

Further information can be obtained by writing Dr. Zeno W. Wicks, Jr., Polymers and Coatings Dept., North Dakota State University, Fargo, N.D. 58105.

Corrosion Failures is Subject of Battelle Conference

The identification and prevention of corrosion failures will be discussed at a conference, sponsored by Battelle Columbus Laboratories, in Houston, March 24 and 25.

This conference is aimed at acquainting engineers with the mechanisms of corrosion, how they are manifested, and how they can be prevented. Various forms of corrosion in a variety of structural alloys will be examined. Speakers from Battelle and industry will discuss technologies with emphasis on case histories of corrosion failures and preventions.

Topics will include: forms of corrosion; general, localized, and intergranular corrosion; pitting, impingement, and cavitation; environmental cracking—stress corrosion cracking, hydrogen embrittlement, and corrosion fatigue; high-temperature corrosion-erosion; and corrosion control—cathodic protection inhibitors and coatings.

A panel discussion will also be held to address specific concerns of registrants.

Additional information may be obtained from Ruth Anne Gibson, Battelle's Columbus Laboratories, 505 King Ave., Columbus, Ohio 43201.

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People

McCloskey Varnish Co., Philadelphia, Pa., has appointed **Peter H. Hess** President and General Manager of the firm's western headquarters, located in Los Angeles. In his new position, he will be responsible for all operations of the Calif. facility.

Dr. David D. Taft, Group Vice-President, has been appointed a Director of Henkel Corp., Minneapolis, Minn. Most recently, Dr. Taft assumed responsibility for Henkel Chemicals Canada, in addition to his duties with chemical specialties, textile, consumer products, leather, and export divisions.

Arthur T. Gronet will assume the responsibilities of Manager of Research and Development of Armstrong Products Co., Warsaw, Ind. His new duties will include the formulation and direction of research and development programs for the company.

Dan K. Farstad, Senior Vice-President of Operations, has retired from the Spencer Kellogg Div. of Textron, Inc. after 29 years of service. A Past-President of the National Paint and Coatings Association, he was the 1979 recipient of the organization's Industry Statesman Award. Mr. Farstad was responsible for several special projects which have affected the long-term growth of the division.

The duPont Co., Wilmington, Del., has named **N. Douglas Cassel** Product Manager, automotives, for the colored pigment products div. in the Chemicals, Dyes, and Pigments Dept. Assuming his new duties, he will be responsible for supervising marketing of colored pigment products to the automotive industry.

DeSoto, Inc., Des Plaines, Ill., has announced the following appointments: **Kazys Sekmakas** has been promoted to the newly created position of Staff Scientist; **Dennis A. Centofante** to Manager of Operations, Chemical Coatings Div.; **Terry Szesny** to Elgin Plant Manager, Chemical Coatings Div.; **Myron J. Bezdicek** to Supervisor, interior trade sales laboratory; **Wayne E. Gyssler** to Supervisor, exterior trade sales laboratory; and **Raj Shah** to Senior Research Chemist in the Polymer Development Dept. Messrs. Sekmakas, Centofante, Szesny, Bezdicek, and Gyssler are members of the Chicago Society.



P. H. Hess



D. D. Taft



R. Harman-Stangs



W. M. Kraus

In a series of appointments, Drew Chemical Corp., Boonton, N.J., has named **James A. Thorstad**—District Representative; **Denis Breton**—Director of Sales for the Water and Waste Treatment and Specialty Chemicals Div.; and **Ruth K. Harman-Stangs**—Market Specialist. Mr. Thorstad is a member of the Chicago Society.

Richard K. Lucas has been named Market Manager, Wood, for the Chemical Coatings unit of Glidden Coatings & Resins, Div. of SCM Corp., in Cleveland. He previously served as Market Manager, Plastics, for the firm.

Meanwhile, **Thomas G. Christner** has been appointed Resin Plant Manager for the Midwest Consolidated Resin Plant of Glidden. He will be headquartered in Huron, Ohio.

Edward J. Goett, President and Chief Executive Officer of ICI Americas, Inc., Wilmington, Del., will retire March 31, 1980, after 25 years' service in the industry. He will be succeeded by **Robert P. Barnett**, Senior Executive Vice-President.

Applied Color Systems, Inc., Princeton, N.J., has appointed **Michael Lycosky** to the position of Business Software Product Manager. Assuming this new position, he will undertake responsibilities for the development, specification, and coordination of business program packages for customers of the company in all industries.

Dr. E. Janet Berry has become the first woman President of the American Institute of Chemists, a 5,500 member professional society of chemists and chemical engineers. Dr. Berry, who has served as President-Elect since 1977, became chief executive officer of the Institute on January 1, and will hold that office for the next two years.

M & T Chemicals, Inc., Rahway, N.Y., has appointed **Jay A. Kaplan** Senior Vice-President of Operations. In this capacity, he will take responsibility for all operating divisions of the company.

Meanwhile, **William M. Kraus** has been named General Manager of the Plastics Additives Div. of the firm. Mr. Kraus will bring to this position many years of experience in sales and marketing, technical research, and manufacturing.

As part of a new marketing organization for the colored pigment products division in the Chemicals, Dyes, and Pigments Dept. of duPont Co., Wilmington Del., **John S. Thackrah** has been named Marketing Manager. Under this reorganization, the following will hold specific product responsibilities: **N. Douglas Cassel**—Product Manager, automotive finishes and plastics; **Donald W. Hoppenjans**—Product Manager, plastics; **Richard A. Repetto**—Product Manager, paint; and **John F. Kane**—Product Technologist, ink.

Three officers and three directors have been elected by the membership of the National Association of Corrosion Engineers to serve terms beginning in March 1980. The new officers are: President—**M.D. Orton**; Vice-President, **D.A. Dutton**; and Secretary-Treasurer—**W.K. Boyd**. Named as Directors are **E.W. Haycock**, **Robert P. Brown**, and **W.J. Griest**.

The Petrochemical Div. of American Hoechst Corp., has promoted **Charles V. Sleeth** to Senior Divisional Vice-President. He will be responsible for marketing, supply, and distribution activities.

In addition, the division has appointed **William C. Bowman** Divisional Vice-President—Marketing. He will be located in the division's new sales office in Houston, Tx.

Philip R. Arvidson and **Sam G. Houston** have been elected Vice-Presidents of Inmont Corp., New York, N.Y. Mr. Arvidson will have responsibility for the firm's commercial development activities. In his new position, Mr. Houston will assume responsibility for business analysis and planning.

Rohm and Haas Co., Philadelphia, Pa., has appointed **David Moffat** Product Specialist for industrial coatings. Mr. Moffat is a member of the Toronto Society.

Air Products and Chemicals, Inc., Allentown, Pa., has appointed **Ronald L. Turner** Marketing Manager for its polyvinyl alcohol business area, and **Jeffrey C. Searer** Market Manager for polymerization and adhesives in the same area.

Eric Barry, Executive Vice-President of the Canadian Paint Manufacturers Association for the past 30 years resigned on January 1, to become President of the Canadian Textiles Institute in Montreal.

He was succeeded by **Richard W. (Dick) Murry**, Technical Director of the CPMA for the past two years.

Mr. Barry joined the CPMA in 1952 as Assistant to the President and General Manager. He was named General Manager in 1958, the title changing to Executive Vice-President in 1974.

Mr. Murry was associated with the National Paint and Coatings Association in Washington, D.C., prior to joining CPMA in 1977.

A. Frank Leo has been named Senior Research Chemist for Bee Chemical Co., Lansing, Ill. In his new position, he will concentrate on refinement and development of new coating systems to meet the demanding requirements of industrial coaters. Mr. Leo is a member of the Chicago Society.

Ashland Chemical Co. has named **Robert A. York** Supervisor of Advertising. He will be headquartered in the Columbus, Ohio area.

Kirt Smith has been appointed Polymer Applications Specialist in the research and development laboratory of Freeport Kaolin Co., a division of Freeport Minerals Co., New York, N.Y. Mr. Smith was most recently employed with PPG Industries, Coatings and Resins Div.

Byk-Mallinocrodt, Melville, N.Y., has promoted **John S. Parker** to the position of Sales Support Manager. Mr. Parker will be responsible for daily sales operation in the U.S., Canada, Australia, Puerto Rico, and the Dominican Republic.

Reichhold Chemicals, Inc. has named **George C. Sweet** to the position of Senior Vice-President, Administration and Material Management. In this capacity, Mr. Sweet's duties will include material procurement and overseeing various administrative functions.

Kerr-McGee Chemical Corp., Oklahoma City, has named **J.R. Kelley** Vice-President of Chemical Marketing. He will be succeeded in his former position as Vice-President of Soda Products, Marketing, by **L.E. Erikson**.

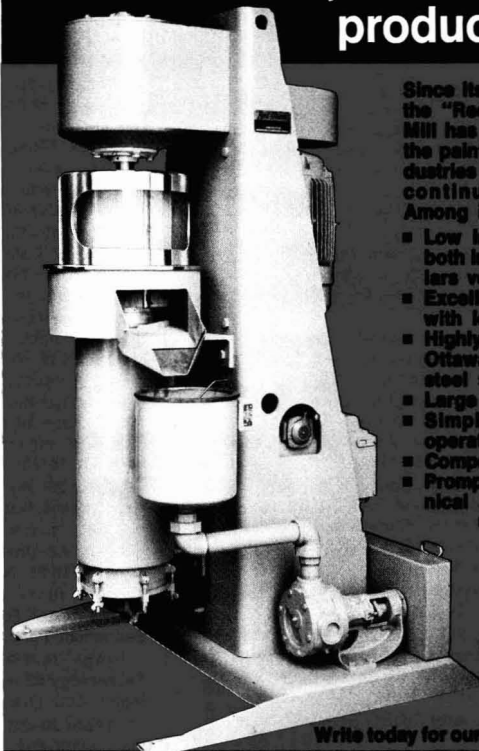
James W. Lenigan has been named Powder Coatings Account Specialist for the southern territory of Glidden Coatings & Resins, Div. of SCM Corp. He will be headquartered in Memphis, Tenn.

California Products Corp., Cambridge, Mass., has appointed **Billy E. McArdle** to Vice-President, Paint Operations. Mr. McArdle has been associated with the paint industry since 1959.

Robert A. Taylor has been appointed Technical Manager of coatings research and development for national programs in the kitchen cabinet market for Reliance Universal Inc., Louisville, Ky.

In a series of directorial appointments, Kelco, Div. of Merck & Co., Inc., has named **Dr. Ian W. Cottrell**—Director, Basic Research and Development; **Dr. David J. Pettitt**—Executive Director, Research and Development; and **Dr. Kenneth S. Kang**—Associate Director, Microbiological Sciences Research.

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Coming Events

FEDERATION MEETINGS

1980

(May 15-17)—Federation Spring Meetings. Society Officers—15th; Executive Committee—16th; Board of Directors—16th and 17th. North Star Inn, Minneapolis, Minn. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

(Oct 28)—Federation Board of Directors Meeting. Hyatt Regency, Atlanta, Ga. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

(Oct. 29-31)—58th Annual Meeting and 45th Paint Industries' Show. Atlanta Civic Center, Atlanta, Ga. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

1981

(Mar. 24-26)—"Symposium on Color and Appearance Instrumentation." Jointly sponsored by Federation of Societies for Coatings Technology, Manufacturers Council on Color and Appearance, and Inter-Society Color Council. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, Pa. 19107).

SPECIAL SOCIETY MEETINGS

1980

(Mar. 10-12)—Seventh Annual Water-Borne and Higher-Solids Coatings Symposium. Hyatt Regency Hotel, New Orleans, La. Sponsored by Southern Society and University of Southern Mississippi. (Dr. George Bufkin, Dept. of Polymer Science, University of Southern Mississippi, Southern Station, Box 276, Hattiesburg, Miss. 39401).

(Mar. 12-14)—Southern Society, 44th Annual Meeting and Convention. Terrace Garden Inn, Atlanta, Ga. (Peter F. Decker, Union Carbide Corp., 17 Executive Park Dr., N.E., Atlanta, Ga. 30359).

(Mar. 25-26)—Chicago Society, SYMCO '80, "Additives." Fountain Blue, Des Plaines, Ill. (Dennis Socha, U.S. Gypsum Co., 1000 E. Northwest Highway, Des Plaines, Ill. 60016).

(Mar. 25-26)—23rd Symposium of the Cleveland Society, "Advances in Coatings Technology." Baldwin-Wallace College, Berea, Ohio. (James A. Miller, Morgan Adhesives Co., 4560 Darrow Rd., Stow, Ohio 44224, (216) 688-111.)

(Apr. 14-15)—Louisville Society Spring Symposium, "Compliance with Government Regulations" and "Hazardous Materials Waste Disposal." Marriott Inn, Clarksville, Ind. (M. Joyce Specht, Porter Paint Co., Corporate Office, 400 South 13th St., P.O. Box 1439, Louisville, Ky. 40201).

(April 17-19)—Dallas and Houston Societies. Southwestern Paint Convention, Hilton Inn, Dallas, Tex.

(May 1)—Detroit Society FOCUS Seminar. "Environmental Challenges to Automotive Coatings." Michigan State Management Conference Center, Troy, Mich. (G. Sastry, Chrysler Corp., Chemicals Div., 5437 W. Jefferson, Trenton, Mich. 48183).

(May 1-3)—Pacific Northwest Society Annual Symposium. Hilton Hotel, Portland, Ore.

(May 8)—Kansas City Symposia, "Modernistic Approaches to Hiding." (Dennis Mathes, Cook Paint & Varnish Co., P.O. Box 389, Kansas City, Mo. 64141.)

(May 13-14)—"Coatings and Ink Expo '80," sponsored by the New England Society. Sheraton Inn and Conference Center, Boxborough, Mass. (Dame Assoc., Inc., 51 Church St., Boston, Mass. 02116, (617) 482-3596.)

(May 15)—Birmingham Society Symposium, "Effect of Current Legislation in the U.K. Paint Industry." Metropole Hotel,

Birmingham, England. (D.M. Heath, Arthur Holden & Sons Ltd., Bordesley Green Rd., Birmingham, B9 4TQ, England.)

(June 6-7)—Joint meeting of Kansas City and St. Louis Societies. Hilton Plaza Hotel, Kansas City, Mo. (Tim T. Walsh, Walsh & Associates, Inc., 500 Railroad Ave., N. Kansas City, Mo. 64116).

(Oct. 7)—Cleveland Society Manufacturing Committee Symposium, "Formulation for and Utilization of Pigment Dispersion Equipment." Cleveland Engineering and Scientific Center, Cleveland, Ohio. (G. Dubey, Cambridge Coatings, Inc., 5461 Dunham Rd., Cleveland, Ohio 44137).

1981

(Mar. 4-6)—Western Coatings Societies Symposium and Show. Disneyland Hotel, Anaheim, Ca.

OTHER ORGANIZATIONS

(Feb. 25-Apr. 11)—Paint Short Courses at University of Missouri-Rolla. Painting Contractors and Paint Inspectors—Feb. 25-29; Composition of Paints and Coatings—Mar. 3-7; Quality Control—Mar. 10-14; Fundamentals of Paint Formulation—Mar. 31-Apr. 4; Chemical Coatings Workshop—Apr. 7-11. (Norma Fleming, Continuing Education, University of Missouri-Rolla, 501 West 11th St., Rolla, Mo.).

(Mar. 3-7)—"CORROSION/80," sponsored by National Association of Corrosion Engineers, Palmer House, Chicago, Ill. (Membership Services Dept., NACE Headquarters, P.O. Box 986, Katy, Tex. 77450).

(Mar. 4-6)—National Association of Corrosion Engineers. Materials Performance and Corrosion Show. Palmer House, Chicago, Ill. (NACE, P.O. Box 986, Katy, Tex. 77450).

(Mar. 10-15)—"High Performance Coating Procedures," San Diego, Calif. (Institute of Applied Technology, Jean L. Kaplan, 1200 - 17th St., N.W., Suite 406, Washington, D.C. 20036.)

(Mar. 23-28)—Div. of Organic Coatings and Plastics Chemistry Symposia and Spring Meeting of American Chemical Society, Houston, Texas. (American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036).

(Mar. 25-27)—Society of Manufacturing Engineers Workshop on Radiation Curing in the Graphic Arts. Ramada O'Hare Inn, Chicago, Ill. (Susan E. Buhr, Technical Division, Society of Manufacturing Engineers, One SME Drive, P.O. Box 930, Dearborn, Mi. 48128).

(Mar. 26-28)—Marine Coatings Conference, Marriott Hotel, New Orleans, National Paint and Coatings Assoc.)

(Apr. 7-11)—"Nuclear Quality-Assured Coating Work," Phoenix, AZ. (Institute of Applied Technology, Jean L. Kaplan, 1200 - 17th St., N.W., Suite 406, Washington, D.C. 20036.)

(Apr. 10-11)—"Symposium on Emulsion Polymerization of Vinyl Acetate." Emulsion Polymers Institute, Lehigh University, Bethlehem, Pa.

(Apr. 14-15)—20th Annual Symposium, "New Coatings Technology for the 1980's," Marriott Twin Bridges Motel, Washington, D.C. (Washington Paint Technical Group.)

(April 20-22)—Inter-Society Color Council. Annual meeting. Downtown Holiday Inn, Rochester, N.Y. (Dr. Fred Billmeyer, Secretary, ISCC, Rensselaer Polytechnic Institute, Troy, N.Y. 12181).

(April 21-25)—"High Performance Coating Procedures," Philadelphia, Pa. (Institute of Applied Technology, Jean L. Kaplan, 1200 - 17th St., N.W., Suite 406, Washington, D.C. 20036.)

(Apr. 29-May 1)—Oil and Colour Chemists Association Australia, Victoria Section. Symposium on "Toxicity." In Melbourne on Apr. 29; Sydney on May 1. (Brian Gray, Secretary, OCCAA, 25 Shanley St., Pascoe Vale, Vic. 3044, Australia.

(May 4-5)—National Coil Coaters Association Annual Meeting, Hotel Del Coronado, Coronado, Calif. (NCCA 1900 Arch St., Phila., Pa. 19103.)

(May 5-8)—Society of Plastics Engineers, 38th Annual Technical Conference (ANTEC). New York Hilton, N.Y. (Rod Ellis, SPE, 656 W. Putnam Ave., Greenwich, Conn. (06830).

(May 12-16)—"Coatings and Polymer Characterization" Short Course. Kent State University, Kent, Ohio. (Carl J. Knauss, Chemistry Dept., Kent State Univ., Kent, Ohio 44242).

(May 12-16)—"High Performance Coating Procedures," New Orleans, LA. (Institute of Applied Technology, Jean L. Kaplan, 1200 - 17th St., N.W., Suite 406, Washington, D.C. 20036.)

(May 13-15)—Oil and Colour Chemists Association Technical Exhibition, OCCA 32, Cunard International Hotel, London, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HA0 2SF, England).

(May 19-23)—"Advanced Chemical Coatings Workshop," University of Missouri—Rolla. (Norma Fleming, Continuing Education, University of Missouri—Rolla, 501 W. 11th St., Rolla, Mo.)

(May 19-23)—Dispersion of Pigments and Resins in Fluid Media" Short Course. Kent State University, Kent, Ohio. (Carl J. Knauss, Chemistry Dept., Kent State Univ., Kent, Ohio 44242).

(June 1-6)—"Polymer Blends and Composites" Short Course. Lehigh University, Bethlehem, Pa. (Ms. Jone Svirzofsky, Lehigh Univ., Materials Research Center, Coxe Lab. 32, Bethlehem, Pa. 18015).

(June 2-13)—Basic Coatings Science Course. North Dakota State University, Fargo, N.D. Zeno W. Wicks, Jr., Polymers and Coatings Dept., NDSU, Fargo, N.D. 58105).

(June 8-13)—XVth Congress of FATIPEC, RAI Congress Centre, Amsterdam, Netherlands. "Activities of the Coatings Industry in the Framework of Ecology, Energy, and Economy Problems." (Congress Secretary is C. Kork, Oostenrijklaan 43, Haarlem, Netherlands).

(June 15-18)—Dry Color Manufacturer's Association Annual Meeting. The Greenbriar, White Sulphur Springs, W.Va. (J.L. Robinson, Dry Color Manufacturers' Association, Suite 100, 117 North 19th St., Arlington (Rosslyn) Va. 22209).

(June 16-20)—"Adhesion Principles and Practice for Coatings and Polymer Scientists" Short Course. Kent State University, Kent, Ohio. (Carl J. Knauss, Chemistry Dept., Kent State Univ., Kent, Ohio 44242).

(June 16-20)—"High Performance Coating Procedures," San Francisco, Calif. (Institute of Applied Technology, Jean L. Kaplan, 1200 - 17th St., N.W., Suite 406, Washington, D.C. 20036.)

(June 16-27)—Advanced Coatings Science Course, North Dakota State University, Fargo, N.D. (Dr. Zeno W. Wicks, Jr., Polymers and Coatings Dept., NDSU, Fargo, N.D. 58105).

(June 22-27)—73rd Annual Meeting and Exhibition, Air Pollution Control Association, "Pollution/Economics/People," Place Bonaventure, Montreal, Quebec, Canada. (Public Relation Dept., Air Pollution Control Association, P.O. Box 2861, Pittsburgh, Pa. 15230.)

(June 23-27)—"Applied Rheology for Industrial Chemists," Rheology and Coatings Laboratory, Kent State University, Kent, Ohio. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, Ohio 44242.)

(June 25-27)—"Interpenetrating Polymer Networks" Short Course. Lehigh University, Bethlehem, Pa. (Ms. Jone Svirzofsky, Lehigh Univ., Materials Research Center, Coxe Lab. 32, Bethlehem, Pa. 18015).

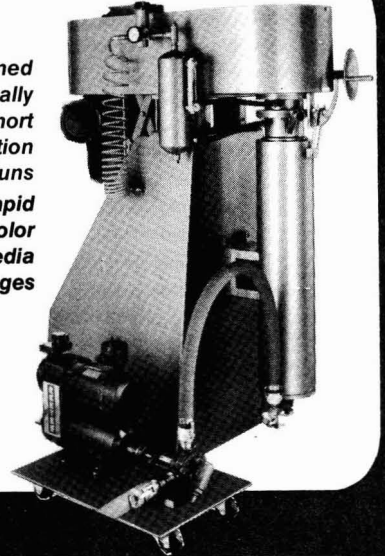
(July 1980)—Oil and Colour Chemists' Association Australia. Annual Convention. Tanunda, Australia.

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(July 14-18)—"Sixth Annual International Conference in Organic Coatings Science and Technology," Athens, Greece. (Angelos V. Patsis, State University of New York, CSB Building, Room 209, New Paltz, N.Y. 12561).

(Aug. 18-Nov. 21)—Paint Short Courses at University of Missouri-Rolla. Introductory, Composition of Paints—Aug. 18-22; Basic Quality Control—Aug. 25-29; Introduction to Paint Formulation—Sept. 8-12; Advanced Paint Formulation—Sept. 15-19; Refresher Course for Maintenance Engineers, Contractors, and Painting Inspectors—Nov. 10-14; Job Estimating Workshop for Painting Contractors—Nov. 17-21. (Norma Fleming, Continuing Education, University of Missouri-Rolla, 501 W. 11th St., Rolla, Mo.)

(Sept. 14-17)—"Advanced Coating Technology," Williamsburg, Va. (Institute of Applied Technology, Jean L. Kaplan, 1200-17th St., N.W., Suite 406, Washington, D.C. 20036.)

(Sept. 23-25)—Association of Finishing Processes of the Society of Manufacturing Engineers, 5th International Conference and Exposition on Radiation Curing. Park Plaza Hotel, Boston, Mass. (AFP/SME, Technical Administrator, Society of Manufacturing Engineers, One SME Drive, P.O. Box 930, Dearborn, Mich. 48128)

(Sept. 24-26)—National Coil Coaters Association Fall Technical Meeting, Marriott Lincolnshire Resort, Lincolnshire, Ill. (NCCA, 1900 Arch St., Phila., Pa. 19103.)

(Sept. 29-Oct. 3)—"Nuclear Quality-Assured Coating Work," Boston, Mass. (Institute of Applied Technology, Jean L. Kaplan, 1200 - 17th St., N.W., Suite 406, Washington, D.C. 20036.)

(Sept. 30-Oct 2)—The Metals Society, International Conference, "Production and Use of Coil-Coated Strip." Metropole Hotel National Exhibition Centre, Birmingham England. (T.L. Hughes, The Metals Society, 1 Carlton House Terrace, London SW1Y 5DB).

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