

AUGUST 1980

# ILLTIFUNCTIONAL EPOXY RESINS COME OF AGE

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**1930's – Nuodex** liquid certified metalcontent driers; and **Nuact** paste-form drier stabilizer.

**1940's** – Super Ad-It universal metallic fungicide and preservative; GPD color-in-oil pigment dispersions; Exkin 1 and Exkin 2 anti-skinning agents.

**1950's** – Nuolate Tallate driers; Cal-Tint and Colortrend universal color systems for both oil and latex paints; plus Nuosperse 657 dispersing agent, Nuodex Zirconium 6, Nuvis 2, Nuvis HS, and Nuvis LB special purpose additives; Nuodex PMA 18 fungicide and preservative; Exkin 3 Zinc and Aluminum stearates; and Kromosperse color dispersant. **1960's** – Nuodex NA surfactant; Fungitrol-11 fungicide; Nuodex PMA-60 preservative; NuXtra driers; and Uni-Cal 66 color systems for industrial coatings.

1970's – Colortrend Cue, a consumer oriented color system; Aqueous and Non-aqueous Industrial Colorants, Polyester Colorants; Nuact NOPB lead-free drier stabilizer; Nuosperse HOH latex pigment dispersion; Nuosept-95 preservotive; Nuosperse PAC low-foam water-soluble pigment dispersant; Aqua Thix™ thixotropic thickening agent.

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Volume 52

Number 667

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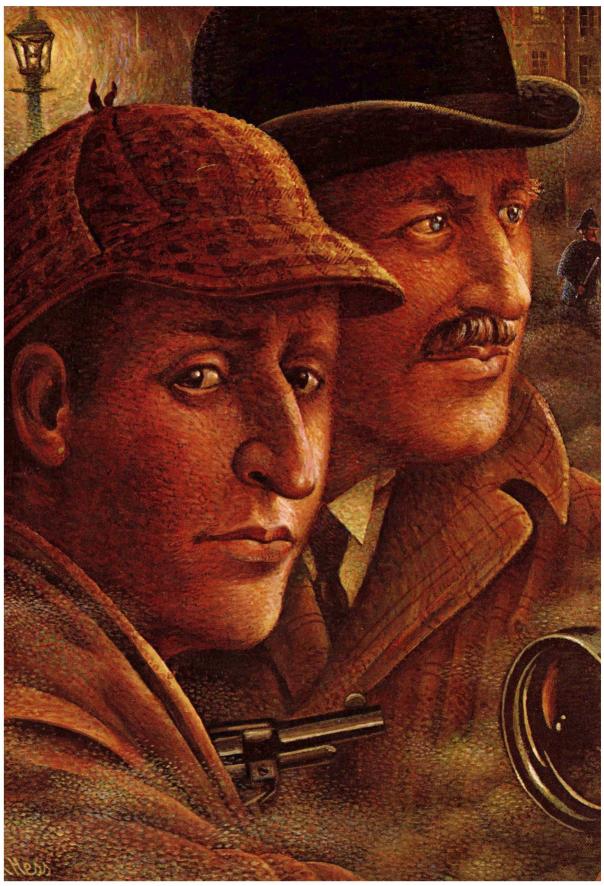
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# Comment

#### **Technical Project Work—The Beat Goes On**

Each year the Federation sponsors a meeting of members of the Technical Advisory Committee with Society Technical Committee Chairmen for the purpose of reviewing projects currently underway and exploring the potential of suggested new programs.

Newcomers attending these meetings invariably express surprise at the amount of project work going on. They've heard the gloomy litany about the dwindling numbers of members participating in Technical Committee activity, in their own Society as well as in others; but they find that, while there may be fewer people active in project work, there's no shortage of imaginative ideas for Technical Committee undertakings.

As summarized in the most recent reports on Society technical programs (see pages 72-73), there are many projects of substantial technological significance, either in progress or in the planning stage. The scope of the activity is reflected in the topics of Society project work scheduled to be reported on at the 1980 Federation Annual Meeting in Atlanta: Renewable Resources (Chicago); Extender Pigments (Louisville); Flash Rust Inhibitors (New England); Freeze-Thaw Stability (Montreal); and Exposure Performance of Primers Conforming to CARB Regs for 1984 (Golden Gate).

Indeed, the demise of Technical Committee activity has been exaggerated; it is still very much alive and contributing to the industry's well-being as it has for a good many years—TAK

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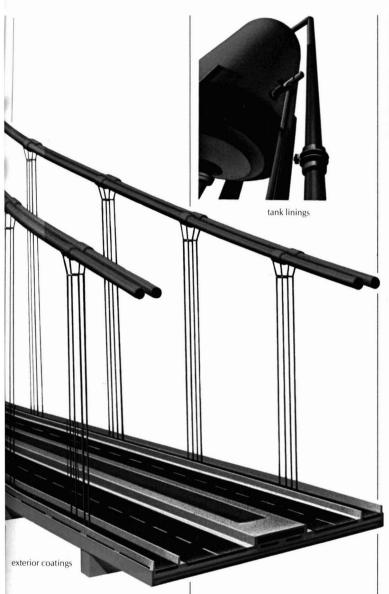
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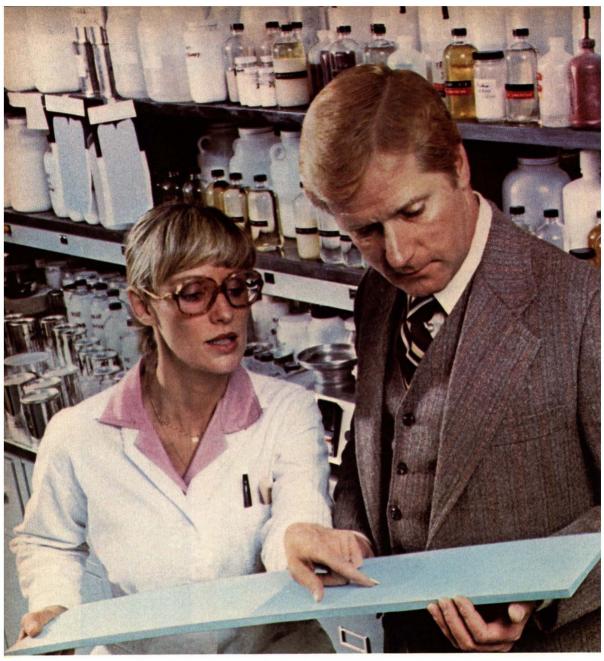
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# Abstracts of Papers inThis Issue

REACTIVITY OF ETHERIFIED AMINOPLAST RESINS— J.O. Santer and G.J. Anderson

Journal of Coatings Technology, 52, No. 667, 33 (Aug. 1980)

An important trend within the coatings industry is to formulations which cure at lower temperatures and/or in shorter times. For aminoplast-crosslinked thermosetting formulations, improved cure response can be achieved by modifications of both the aminoplast and the primary film-former (vehicle). This paper discusses the effect of such modifications and describes experiments in which aminoplast resins were reacted with hydroxyl-functional model vehicles in order to gain insight into the effective functionality/reactivity of the various aminoplasts and to indicate routes towards more efficient low temperature cure.

## NOMOGRAMS FOR SOLUBILITY PARAMETER—A. Jayasri and M. Yaseen

Journal of Coatings Technology, 52, No. 667, 41 (Aug. 1980)

A simple equation which defines the effect of temperature on solubility parameter ( $\delta$ ) has been derived using the equation of Fedors and Watson. This equation has also been represented by a nomogram for finding  $\delta$  values at various temperatures provided the critical temperature and one value of  $\delta$  of a substance are known. The basic equation showing the correlation of  $\delta$  with molar volume, internal molar energy, and heat of vaporization has also been transformed into two nomograms for finding  $\delta$  at 25°C.  $\delta$  values obtained from these nomograms have been found comparable with those reported elsewhere.

CONDENSATION POLYMER FORMULATION IN THE AGE OF OLIGOMERS-L.H. Brown

Journal of Coatings Technology, 52, No. 667, 47 (Aug. 1980)

A review of the theoretical concepts of Carothers and Flory, applied by Kilb, Bernardo and Bruins, Patton, Finney, Earhart and others, shows that condensation polymers for solution coatings can be formulated according to incipient gelation criteria with reasonable assurance of success.

Today, with emphasis on oligomeric polymers for high solids, water-dispersible and powder coatings, the former concepts have less value.

For polyesters, Patton's constant (K = Mo/Ea) becomes less meaningful than theoretical  $\overline{M}n$  and  $EW_{OH}$ (weight of polymer per excess carbinol group). Reactions of the base polyesters with alkoxy-functional melamines, siloxanes or other modifiers during cure have changed the precepts on which formulations are based. This is true especially for the current rapid cures required for prefinished metal.

Application of the new criteria is facilitated by equations which can be handled by programmable calculators or computers. Modification by silicone oligomers has been reduced to a mathematical treatment, and examples show the utility of the method.

#### NONDESTRUCTIVE DRYING OF AMINO RESIN SOLU-TIONS—S.M. Kambanis and J. Rybicky

Journal of Coatings Technology, 52, No. 667, 61 (Aug. 1980)

Techniques for the removal of solvent from unmodified and etherified urea-formaldehyde resins were investigated. Vacuum drying of 50  $\mu$  thick films at temperatures of approximately 50°C and pressures of about 0.2 kPa for the duration of 1 hr was found to provide good and practical drying conditions. The drying was shown to have no observable effect on the structure of resin. The free formaldehyde content in dried resin was found to be less than 0.006%.

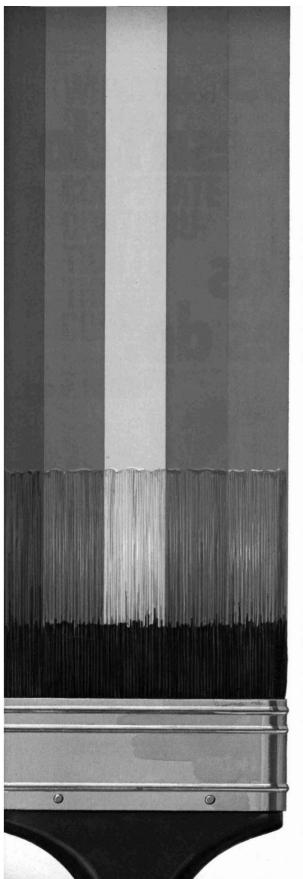
#### MULTIFUNCTIONAL EPOXY RESINS COME OF AGE-M. DIBENEDETTO

Journal of Coatings Technology, 52, No. 667, 65 (Aug. 1980)

Multifunctional epoxy resins such as epoxy cresol and epoxy phenol novolacs and triglycidyl para-aminophenol have long found utility in the molding, casting, and electrical areas of application. In combination with anhydride or aromatic amine curing agents, these resins were traditionally heat cured to form networks of extremely high crosslink density. This phenomenon was responsible for the high heat deflection temperatures and the excellent chemical resistance properties for which such systems were and still are known.

Today, however, the use of multifunctional resins need not be restricted to applications which require high gelation and post-cure temperatures. Through careful formulation, systems based on multifunctional resins show promise in room temperature cured coating applications. Since they possess the same high crosslink density as their casting and molding counterparts, these room temperature cured coating systems should afford excellence in the areas of chemical resistance, surface hardness, and thermal stability.

Numerous application possibilities for such systems exist in the areas of maintenance and marine coatings and energy transmission service.



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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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## Government / Industry

#### NABADA Issues Guidelines On "Empty" Drum Residue

The National Barrel and Drum Association has issued guidelines for residue left in "empty" drums and has recommended their adoption by all emptiers, dealers, and reconditioners of used 55-gal steel drums.

The final guidelines suggest the following:

Non-Hazardous and Non-Toxic Residues—Maximum amount of residue of free-flowing material that may be left in a drum shall not exceed 1% of the marked capacity of the container (e.g., 55-gal drum—½ gal). Maximum amount of non-freeflowing material or viscous material that may be left in a drum shall not exceed 2% of the marked capacity of the container (e.g., 55-gal drum—2 gal).

Hazardous and Toxic Residues—Drums must be neutralized, decontaminated, cleaned or purged, and the maximum amount of material that may be left in drums shall be 1% of the marked capacity of the container.

For further information, contact Morris Hershon, President, NABADA, 910 17th St., N.W., Washington, D. C. 20006. (202) 296-8028.

#### Bacteria May Be Solution To Toxic Chemical Wastes

Bacteria may be one way to destroy unwanted toxic chemicals on land and water, according to researchers at Battelle's Columbus (Ohio) Laboratories.

The researchers are conducting genetic engineering studies on ways microorganisms can be used to transform or degrade toxic chemicals—such as DDT or 2,4-D—into easily disposable products.

According to Battelle's Drs. George Kidd and George Pierce, the capability of bacteria ingesting toxic chemicals or pollutants and converting them to other substances has vast potential for industry. They believe, for instance, that microorganisms may be developed to control oil spills or pollution, dispose of industrial wastes, reclaim land, or extract useful chemicals.

Currently, Battelle researchers are using gene-to-bacterium transfer techniques to program bacteria with the traits necessary to ingest toxic chemicals.

"With this technique," Drs. Kidd and Pierce explained, "a donor organism with a desired quality is selected. Within the organism, specific genes encoding for that trait are identified. These genes are isolated and transferred to a different selected host bacterium. The bacterium—a single, undifferentiated cell—readily accepts the donor genes and transmits the new characteristics to its daughter cells."

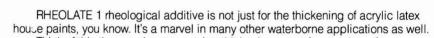
In the Battelle experiments, researchers have isolated the soil microorganism *Pseudomonas* bacteria. These bacteria, which can be found on devastated land, use the defoliant 2,4-D as their carbon source.

Researchers have isolated the genes that "eat up" the 2,4-D and have developed bacteria capable of ingesting it. The bacteria then becomes an innocuous product or may even be converted into salable items such as fertilizers.

The experiments, conducted in special containment laboratories at Battelle, are aimed at improving bacteria that can be used in industrial processes.

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# PRELIMINARY PROGRAM

## 1980 ANNUAL MEETING and PAINT INDUSTRIES' SHOW



## October 29, 30, 31 Atlanta Civic Center, Atlanta, Georgia

FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

## **Preliminary Program**

## WEDNESDAY, OCTOBER 29

#### OPENING SESSION (10:30-11:00)

Fifty-eighth Annual Meeting of the Federation of Societies for Coatings Technology opened by President Elder C. Larson

Invocation and In Memoriam

Welcome: Thad T. Broome, President of Southern Society for Coatings Technology

Hugh Lowrey, Chairman of the Program Committee

Berger Justen, Chairman of the Host Committee Deryk R. Pawsey, Chairman of the Paint Industries' Show Committee

Introduction of Federation Officers

Introduction of Distinguished Guests

#### E. W. FASIG KEYNOTE ADDRESS (11:00-12:00)

THE CHALLENGES OF THE 80's IN COAŢINGS AND GRAPHIC ARTS—Harvey F. George, Executive Vice-President and Research Director, Gravure Research Institute, Port Washington, NY.

#### PROTECTION OF RESOURCES THROUGH CORROSION CONTROL (1:30-4:30)

THE FOUR C's—CALAMITY, CORROSION, COATINGS, AND COMPOSURE—Donald Tuomi, Physics and Electronics Dept., Borg-Warner Corp., Des Plaines, IL.

SALT SPRAY TESTING FOR SHORT-TERM EVALUA-TION OF COATINGS—Bernard R. Appleman, Federal Highway Administration, Washington, DC, and Paul G. Campbell, National Bureau of Standards, Washington, DC.

SOME SUBSTRATE AND ENVIRONMENTAL INFLU-ENCES ON THE CATHODIC DELAMINATION OF ORGANIC COATINGS—Henry Leidheiser, Jr. and Wendy Wang, Center for Surface and Coatings Research, Lehigh University, Bethlehem, PA. (A Roon Awards competition paper) INVESTIGATION AND MONITORING OF THE COR-ROSION CONTROL PROCESS OF COATINGS—Clive H. Hare, Clive H. Hare, Inc., Stoughton, MA.

MECHANISM OF FILM FORMATION OF ALKYL SILICATE ZINC-RICH COATINGS—Thomas Ginsberg, Chemicals and Plastics Div., Union Carbide Corp., Bound Brook, NJ.

TECHNIQUES OF ELECTROCHEMICAL CORROSION MEASUREMENT—W. M. Peterson, Princeton Applied Research Corp., Princeton, NJ.

#### EFFICIENT UTILIZATION OF RESOURCES AND RESEARCH (1:30-4:00)

RENEWABLE RESOURCES FOR THE COATINGS IN-DUSTRY—Chicago Society for Coatings Technology. Presented by T. Kirk Hay, The Sherwin-Williams Co., Chicago, IL.

RECYCLING WASTE EFFLUENT STREAMS WITH UL-TRAFILTRATION—Carl R. Hoffman, Abcor, Inc., Wilmington, MA.

KINETIC PARAMETER CONSIDERATIONS FOR MAX-IMIZING STABILITY AND MINIMIZING CURE TEM-PERATURE OF THERMOSETTING COATINGS. SUL-FONIUM SALTS AS LATENT THERMAL INITIATORS FOR CATIONIC POLYMERIZATION—S. Peter Pappas, Polymers and Coatings Dept., North Dakota State University, Fargo, ND, and Loren W. Hill, Monsanto Plastics and Resins Co., Indian Orchard, MA. (A Roon Awards competition paper)

A COMPUTER METHOD FOR PREDICTING EVAPO-RATION OF MULTICOMPONENT AQUEOUS SOLVENT BLENDS AT ANY HUMIDITY—Albert L. Rocklin, Shell Development Co., Houston, TX, and David C. Bonner, Shell Oil Co., Houston, TX. (A Roon Awards competition paper)

APPLICATION OF SIMPLEX LATTICE DESIGN EX-PERIMENTATION TO COATINGS RESEARCH—Kenneth K. Hesler and John R. Lofstrom, DeSoto, Inc., Des Plaines, IL. (A Roon Awards competition paper)

## **THURSDAY, OCTOBER 30**

#### PRI SEMINAR ON COATINGS RESEARCH PROGRAMS FOR THE 80's (9:00-12:00)

OVERVIEW OF PRI'S NEW DIRECTIONS—Peter V. Robinson, Glidden Coatings and Resins Div., SCM Corp., Strongsville, OH.

MILDEW RESEARCH BY CONSORTIUM—Charles C. Yeager, Registration Consulting Associates, Pacifica, CA., and Program Manager of PRI's Mildew Consortium. SYNTHESIS AND EVALUATION OF ANCHORED FUNGICIDES—Charles U. Pittman, University of Alabama, University, AL.

OLIGOMER RESEARCH FOR HIGH SOLIDS COAT-INGS—Zeno W. Wicks, Jr., Polymers and Coatings Dept., North Dakota State University, Fargo, ND, and Loren W. Hill, Monsanto Plastics and Resins Co., Indian Orchard, MA.

AQUEOUS COATINGS RESEARCH—Raymond R. Myers, PRI Research Director and University Professor, Kent State University, Kent, OH.

## **THURSDAY, OCTOBER 30**

(Continued)

#### MANUFACTURING COMMITTEE SEMINAR ON SAFETY IN THE PAINT PLANT (9:00-12:00)

Moderator-Gilbert E. Cain, of Hercules, Inc., Wilmington, DE.

PROPERTIES AND HANDLING OF FLAMMABLE MATERIALS—Nelson W. Lamb, Hercules, Inc., Wilmington, DE.

LOSS PREVENTION TECHNIQUES IN MATERIALS HANDLING-Gabriel Malkin, Consulting Engineer, West-field, NJ.

IN-PLANT SAFETY PRACTICES AND PROCEDURES— Francis C. Gaugush, The Sherwin-Williams Co., Cleveland, OH.

DEVELOPING AND IMPLEMENTING SUCCESSFUL LOSS-CONTROL TECHNIQUES—Donald W. Smith, Pratt & Lambert, Inc., Buffalo, NY.

Seminar will also feature 20-minute motion picture on general safety techniques and practices.

Moderator and speakers will assemble as a panel for a 45minute open-discussion period to conclude seminar.

Presentation of the Morehouse Industries Golden Impeller Award will be made at this session.

#### SEMINAR ON FORMULATION (9:00-12:00)

FORMULATION STUDIES OF WATER-REDUCIBLE SILICONE ALKYDS—Lynne M. Parr, Resins and Chemicals Div., Dow Corning Corp., Midland, MI.

TITANIUM DIOXIDE: ITS PERFORMANCE IN FLAT LATEX PAINT—Robert Rauch, Tioxide of Canada Ltd., Sorel, Quebec, Canada.

EXTENDER PIGMENTS IN LATEX WALL PAINTS— Louisville Society for Coatings Technology. Presented by Donald W. Collier, Porter Paint Co., Louisville, KY.

FLASH RUST INHIBITORS: AN EVALUATION OF SOME AMINES AND ORGANIC SALTS IN AN AQUEOUS ACRYLIC COATING—New England Society for Coatings Technology. Presented by N. Bradford Brakke, Lilly Chemical Products, Templeton, MA.

FACTORS INFLUENCING FREEZE-THAW STABILITY IN FLAT LATEX PAINTS—Montreal Society for Coatings Technology. Presented by Robert Kuhnen, Tioxide of Canada Ltd., Sorel, Quebec, Canada.

ORGANO-SILANES AS ADHESION PROMOTERS FOR ORGANIC COATINGS—Peter Walker, Ministry of Defence, Aldermaston, Reading, England (A Roon Awards competition paper).

#### TOUR OF ATLANTA-10:00 am

(See Spouses' Activities)

#### TESTING AND PERFORMANCE (2:00-5:00)

EXPOSURE PERFORMANCE EVALUATION OF COM-MERCIALLY AVAILABLE, NON-TOXIC, WATER-BORNE, CORROSION-INHIBITIVE PRIMERS CON-FORMING TO CARB REGULATIONS FOR 1984—Golden Gate Society for Coatings Technology. Presented by Patricia Shaw, Esselte Pendaflex Corp., Emeryville, CA.

OBJECTIVE USE OF SUBJECTIVE COMPARISONS— G. M. Deighton, Laporte Industries Limited, Grimsby, S. Humberside, England. (Presented on behalf of OCCA: Oil and Colour Chemists' Association—UK.)

EVAPORATION AND VAPOR DIFFUSION RESIS-TANCE IN PERMEATION MEASUREMENTS BY THE CUP METHOD—Erik Nilsson and Charles M. Hansen, Scandinavian Paint and Printing Ink Research Institute, Horsholm, Denmark. (Presented on behalf of SLF: Federation of Scandinavian Paint and Varnish Technologists.)

SHORT-TERM EVALUATION TECHNIQUES FOR LIQ-UID AND PARTICLE BEHAVIOR IN COATINGS SYS-TEMS—Frank Zurlo, Byk-Mallinckrodt, Melville, NY.

ANALYSIS OF WATER-BORNE COATINGS FOR THE QUANTITATIVE DETERMINATION OF WATER AND CO-SOLVENTS—James A. Vance, Vance Laboratories, Indianapolis, IN. (A Roon Awards competition paper)

COLOR MEASUREMENT OF WET PAINT-Don W. Parker, Tromac, Inc., Michigan City, IN.

#### INFORMATION: A RESOURCE FOR THE 80's (2:00-4:30)

ON-LINE SEARCHING OF DATABASES OF INTEREST TO THE COATINGS INDUSTRY—Richard Caputo, Lockheed Information Systems, Arlington, VA.

COMPUTER-ASSISTED DESIGN OF COATINGS FORMULAS—Edwin J. Lowrey, Precision Paint Corp., Atlanta, GA.

COMPUTERIZED COLOR CONTROL-Robert T. Marcus, Mobay Chemical Corp., Pittsburgh, PA.

COMPUTERIZED PROCESS CONTROL IN THE COAT-INGS INDUSTRY—Robert W. McFee, Glidden Coatings and Resins Div., SCM Corp., Huron, OH.

TECHNICAL COMPUTER APPLICATIONS IN THE COATINGS INDUSTRY: A BIBLIOGRAPHY, 1967-79—Helen Skowronska, Consultant, Cleveland, OH.

#### EDUCATIONAL COMMITTEE PRESENTATION ON FSCT-SPONSORED CORRESPONDENCE COURSE FOR COATINGS TECHNOLOGY (2:00-3:00)

CORRESPONDENCE COURSE ON THE SCIENCE AND TECHNOLOGY OF SURFACE COATINGS: AN OVER-VIEW—B. George Bufkin, Department of Polymer Science, University of Southern Mississippi, Hattiesburg, MS.

## FRIDAY, OCTOBER 31

#### CONSERVATION IN THE APPLICATION AND CURING OF INDUSTRIAL PRODUCT FINISHES (9:00-10:30)

ENVIRONMENTAL SOLUTIONS FOR THE SPRAY PAINTING INDUSTRY—Arvid C. Walberg, Arvid C. Walberg & Co., Downers Grove, IL.

CURING COATINGS WITH AN INERT-OVEN SOLVENT RECOVERY SYSTEM—Ronald D. Rothchild, Airco Industrial Gases, Murray Hill, NJ.

CURING OF VARNISHES BY MICROWAVES—Henri Valot, National Center for Scientific Research, Thiais, France, (Presented on behalf of FATIPEC: Federation of Associations of Technicians in the Paint, Varnish, Lacquer, and Printing Ink Industries of Continental Europe.)

#### INNOVATION IN POLYMERS (9:00-10:30)

SYNTHESIS OF BLOCKED MDI ADDUCTS, THEIR DSC EVALUATION AND EFFECT OF PIGMENTATION— Taki J. Anagnostou, Wyandotte Paint Products, Inc., Troy, MI, and Ernest Jaul, Silicones & Urethane Intermediates Div., Union Carbide Corp., South Charleston, WV. (A Roon Awards competition paper)

USE OF THIIRANE-FUNCTIONAL MONOMERS AS A MEANS OF DEVELOPING CROSSLINKABLE EMUL-SIONS—B. George Bufkin and John R. Grawe, Department of Polymer Science, University of Southern Mississippi, Hattiesburg, MS, Robert M. O'Brien, Mobil Chemical Co., Pittsburgh, PA, and Samuel A. Brown, of Celanese Chemical Co., Summit, NJ. (A Roon Awards competition paper)

PRIMARY AMINE ZWITTERION CO-POLYMERS— Zeno W. Wicks, Jr., and Chiew-Wah Koay, Polymers and Coatings Department, North Dakota State University, Fargo, ND. (A Roon Awards competition paper)

#### MATTIELLO LECTURE (10:30-11:45)

PHYSICAL CHEMISTRY OF CATHODIC ELECTRO-DEPOSITION—Percy E. Pierce, Manager of Physical/Analytical Research, PPG Industries, Inc., Allison Park, PA.

#### FEDERATION LUNCHEON (11:45)

Ballroom West, Atlanta Hilton Hotel

Presentation of the George Baugh Heckel, Paint Show, and Union Carbide Awards.

Winners of other Federation Awards to be announced.

"You CAN Do Something About It"—Address by Dr. Kenneth McFarland, Dean of America's Public Speakers.

#### ENVIRONMENTAL CONTROL COMMITTEE PANEL DISCUSSION ON WASTE MANAGEMENT BY EPA REGULATIONS—WHAT ANSWERS DO YOU NEED? (2:00-4:00)

Moderator-S. Leonard Davidson, N L Industries, Inc., Hightstown, NJ.

Norman Groves, Reliance Universal, Inc., Louisville, KY.

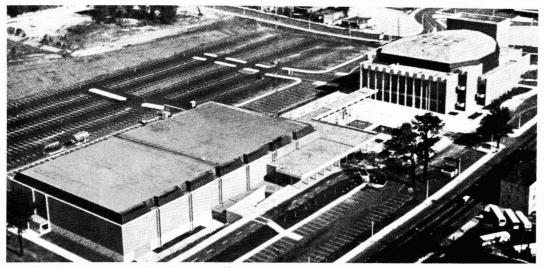
Hugh Williams, Jr., The Sherwin-Williams Co., Cleveland, OH.

#### ANNUAL BUSINESS MEETING (4:00-5:00)

Annual Business Meeting of the Federation

Presentation of these Awards: American Paint & Coatings Journal/A. F. Voss . . . Materials Marketing Associates . . . Program Committee . . . Armin J. Bruning . . . Roon Foundation . . . Ernest T. Trigg.

Installation of Officers, 1980-81



The Atlanta Civic Center will be site of 1980 Federation Annual Meeting and Paint Industries' Show. Exhibit Hall is in foreground, auditorium at upper right

## **OTHER CONVENTION INFORMATION**

#### PAINT INDUSTRIES' SHOW

The 45th Annual Paint Industries' Show will run concurrently with the Annual Meeting in the Atlanta Civic Center. The only national exposition of materials and equipment used in the formulation, testing, and manufacture of coatings, the Show will be open from 12:30 pm to 5:30 pm on Wednesday, October 29; 9:30 am to 5:00 pm on Thursday, October 30; and 9:30 am to 4:00 pm on Friday, October 31.

Participating supplier companies will have their top technical representatives on hand to discuss the latest developments with registrants at this year's event, the largest in Paint Show history.

#### REGISTRATION

Registration fees for the Annual Meeting and Paint Show are on a Federation member and nonmember basis:

	Member	Nonmember	Spouses
Advance*	\$40	\$55	\$25
In Atlanta	\$45	\$60	\$30
One-Day	\$25	\$35	_

\*Special registration for retired members and their spouses only: Advance registration will be \$20 each.

Registration form is included in this issue and has also been mailed to all members.

Note: Purchase of luncheon tickets is optional for both advance and on-site registration.

#### LUNCHEON

The Federation Luncheon will be held on Friday, October 31, at the Atlanta Hilton Hotel.

Presentations will be made to the recipients of the George Baugh Heckel Award (outstanding individual who has contributed to the advancement of the Federation), the Flynn Awards (firms judged to have the best exhibit booths in the 1980 Paint Industries' Show), and the Union Carbide Coatings Technology Award (for extraordinary achievement in coatings technology).

Featured speaker will be Dr. Kenneth McFarland, known as the "Dean of America's Public Speakers," who will address the topic, "You CAN Do Something About It!"

#### SPOUSES' PROGRAM

A schedule of activities has been planned each day for the spouses attending the Annual Meeting, and a Hospitality Room will be maintained at the Atlanta Hilton Hotel.

A get-acquainted Wine and Cheese Social is scheduled for Wednesday afternoon.

On Thursday, a continental breakfast will precede an all-day tour of Atlanta and an ante-bellum plantation.

Continental breakfast will be available again on Friday morning.

Registration fees for the Spouses Activities are \$25 in advance and \$30 on-site.

#### **HEADQUARTERS HOTEL**

The Atlanta Hilton will be headquarters hotel. Other hotels with blocks of rooms set aside for the Annual Meeting are the Marriott, Hyatt Regency, Holiday Inn Downtown, Atlanta American, Ladha Downtown, Downtowner Motor Inn, and the Inntown Motor Hotel.

Shuttle bus service will be provided between participating hotels and the Civic Center.

#### **ROOM RESERVATIONS**

All requests for rooms and suites must be sent to the Federation Housing Bureau on the official form provided by the Federation. These have been mailed to all members and are also included in this issue. Additional forms are available from Federation Headquarters.

#### **BOARD MEETING**

The Fall Board Meeting of the Federation will be held on Tuesday, October 28, at the Hyatt Regency Hotel.

#### SPEAKERS' BREAKFAST

A breakfast and briefing for each day's program participants will be held at the Atlanta Hilton Hotel on Wednesday, Thursday, and Friday.

#### **PUBLICATION OF PAPERS**

The JOURNAL OF COATINGS TECHNOLOGY has prior rights to the publication of all papers presented at the Annual Meeting.

#### NPCA MEETS SAME WEEK

The National Paint and Coatings Association will hold its annual meeting from October 27-29 at the Atlanta Hilton Hotel.

NPCA badges will be honored for admission to the Federation Annual Meeting and Paint Show on Wednesday, October 29.

#### **PROGRAM STEERING COMMITTEE**

Chairman—Hugh Lowrey, of Indurall Coatings, Inc., Birmingham, AL; Vice-Chairman—Thomas J. Miranda, of Whirlpool Corp., Benton Harbor, MI; Umberto Ancona, of McCloskey Varnish Co., Philadelphia, PA; Fred M. Ball, of Eastman Chemical Products, Inc., Kingsport, TN; Thomas Ginsberg, of Union Carbide Corp., Bound Brook, NJ; Kenneth A. Kieselburg, of Valspar Corp., Rockford, IL; and William Mirick, of Battelle Memorial Institute, Columbus, OH.

#### **MEETINGS COMMITTEE**

Members of the Southern Society are serving on the Meetings Committee under General Chairman Berger Justen, of Justen & Associates, Tampa, FL. Chairing the various subcommittees are: Program Operations—Preston D. Smith, of Glidden Coatings & Resins, Atlanta; Information Services— Al Hendry, of A.L. Hendry Co., Tampa; Luncheon—Thad T. Broome, of Precision Paint Corp., Atlanta; Publicity—Peter F. Decker, of Union Carbide Corp., Atlanta; and Spouses' Program—Donald B. Morgan, of Spencer-Kellogg Div., Textron, Inc., Atlanta.

### 1980 Paint Industries' Show Exhibitors Atlanta Civic Center • October 29–31

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

B.A.G. Corp. BASF Wyandotte Corp. Beltron Corp. Bennett's Colorant Div. Blackmer Pump Div., Dover Corp. Brinkmann Instruments, Div. Sybron Brookfield Engineering Labs. Buckman Laboratories, Inc. Burgess Pigment Co. Byk-Mallinckrodt Chem. Prod. GmbH

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

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

Eastman Chemical Products, Inc. Ebonex Corp. Elcometer, Inc. Encapsulair, Inc. Engelhard Minerals & Chems. Div. Epworth Manufacturing Co., Inc. Fawcett Co., Inc. Federated Metals Corp., Sub. ASARCO Filter Specialists, Inc. Fricke Enterprises Co.

Gardner Laboratory, Div. Pacific Sci. Georgia Kaolin Co. 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. Hooker Chemical Corp. J.M. Huber Corp. Hunter Associates Lab., Inc.

Ideal Mfg. & Sales Corp. International Minerals & Chems. Corp. Interstab Chemicals, Inc. IWI Industries, Itasco Div.

Jaygo, Inc. Johns-Manville

Kenrich Petrochemicals, Inc. KTA-Tator Associates, Inc.

Labelette Co. Liquid Controls Corp.

3M/Chemical Resources Div. Macbeth Div., Kollmorgen Corp. Meanchem Limited Meadowbrook Corp. Merck & Co., Chemical Div. Miller Paint Equipment, Inc. MiniFibers, Inc. Modern Paint and Coatings Morehouse Industries, Inc. Myers Engineering, Inc.

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

O'Brien Industrial Equipment Co. Omya, Inc. Paint Research Institute Penn Color, Inc. Pennsylvania Glass Sand Corp. Pfaudler Co., Div. Sybron Corp. Pfizer, Inc., MPM Div. Photomarker Corp. Polychrome Corp. Polychrome Corp. Polyvinyl Chemical Industries PPG Industries, Inc. Premier Mill Corp.

Q-Panel Co. Quackenbush Co.

Reichard-Coulston, Inc. Reichhold Chemicals, Inc. Rohm and Haas Co.

Schold Machine Co. Semi-Bulk Systems, Inc. Shamrock Chemicals Corp. Shell Chemical Co. Silberline Manufacturing Co., Inc. Solem Industries, Inc. Southern Clay Products, Inc. Spencer Kellogg Textron Sun Chemical Corp. Surfatest, Inc. Sweco, Inc.

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

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

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

Wacker Chemical Co. Warren Rupp Co. Wilden Pump & Engineering Co.

Zorelco Limited

#### SHOW HOURS Wednesday, Oct. 29—12:30–5:30 pm Thursday, Oct. 30—9:30 am-5:00 pm Friday, Oct. 31—9:30 am-4:00 pm



1980 Annual Meeting and Paint Industries' Show October 29, 30, 31 Atlanta Civic Center Atlanta, Georgia

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- C 🗌 Manufacturers of Equipment and Containers
- D 🗌 Sales Agent for Raw Materials and Equipment
- E 🗌 Government Agency
- F 🗌 Research/Testing/Consulting
- G 🗆 Educational Institution/Library
- H 🗌 Paint Consumer
- J 🗌 Other

- E 🔲 Technical Sales Service
- F 🗌 Sales and Marketing
- G 🗌 Consultant
- H 🗌 Educator/Student/Librarian
- J 🗌 Other

\*Special registration for Retired Federation Members and their spouses \$20.00 each

in advance only.

**IMPORTANT:** REGISTRATION AND FULL PAYMENT MUST BE IN THE FEDERATION OFFICE NO LATER THAN WED., OCTOBER 1, 1980 TO QUALIFY FOR THIS OFFER.

## **FSCT Annual Meeting and Paint Show Registration Fees**

	OF
Federation Memb	GI

**ONE BLOCK** 

CATEGORY

**IN EACH** 

\$40.00

**ADVANCE REGISTRATION** ATLANTA, GA **OCTOBER 29-31, 1980** 

Non-Member
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\$55.00

MAIL TO: FSCT, 1315 WALNUT ST., PHILADELPHIA, PA 19107



#### MAIL TO: FSCT, 1315 WALNUT ST., PHILADELPHIA, PA 19107

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## FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

## 1980 ANNUAL MEETING AND PAINT SHOW

## SPOUSES ACTIVITIES

#### Wednesday, October 29

2:00 pm—Get-Acquainted wine and cheese social in the Crystal Ballroom of the Hilton Hotel. (Wine courtesy of Pfizer, Inc.)

#### Thursday, October 30

8:00 am-Continental breakfast will be available in the Crystal Ballroom.

10:00 am—Buses will depart the Hilton Hotel for a tour of "Antebellum Roswell/Swan House." The group will be driven to Roswell, a "planned community" 20 miles from downtown Atlanta. Accompanied by a costumed guide, there will be a visit to one of the antique-furnished stately mansions that survived General Sherman's invasion. Also "Bulloch Hall" where Teddy Roosevelt's mother grew up. Lunch will be at Aunt Fanny's Cabin, a picturesque restaurant with a fascinating 130-year history. Then a visit to the Swan House, a magnificently furnished house museum on the property of the Atlanta Historical Society.

#### Friday, October 31

8:00 am-Continental breakfast in the Crystal Ballroom.

12:00 Noon—A special awards luncheon will be held in the Ballroom of the Hilton. Presentation will be made of the Heckel Award (for outstanding contributions to the Federation) and the Flynn Awards (firms judged to have the best exhibits in the 1980 Paint Show). Featured speaker will be Dr. Kenneth McFarland, a philanthropist and educator widely recognized as the "Dean of America's Public Speakers." His talk will be "You CAN Do Something About It." Tickets for the luncheon (not included in the Spouses Registration Fee) are \$10.00 each.

The spouses registration fee (\$25.00 in advance, \$30.00 in Atlanta) includes the wine party, the continental breakfasts, and Thursday tour.

#### FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY 1980 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW ATLANTA CIVIC CENTER, ATLANTA, GEORGIA OCTOBER 29, 30, 31 (Wednesday, Thursday, Friday)

MAIL TO: Coatings Federation Housing Bureau 233 Peachtree St. N.W. #200 Atlanta, GA 30303

#### APPLICATION FOR ACCOMMODATIONS

Please indicate below the type of accommodations requested and your choice of hotels. Room reservations cannot be guaranteed unless this form is received by October 8. All reservation requests must be in writing and processed through the Housing Bureau: Phone requests are not accepted.

TYPE OF ACCOMMODATION	NUMBER	RATE REQUESTED
Single (1 person)		
Double (2 persons)		
Twin (2 persons)		
Suite (parlor and 1 bedroom)		
Suite (parlor and 2 bedrooms)		

СНС	DICE OF HOTELS:
(	see notice below)
1st	
2nd	
3rd	
4th	

Hotel placement will be made in the order received. Your first choice will be assigned, if rooms are available. Otherwise, you will be assigned to hotels of your other choices. Confirmation will be sent to you directly by the hotel. Please make all changes with the Housing Bureau in writing only. All reservations will be held until 6:00 p.m., unless a later arrival time is indicated below.

Please fill in the names and addresses of all occupants of the rooms you have reserved. (Bracket those rooming together.) Incomplete information will delay assignment of rooms. Type additional names on the reverse side of this sheet. Please give accurate arrival and departure times and dates.

Type of			Dat	es of
Type of Room	Name .	Address	Arrival	Departure

		NAME			
SEND CONFIRMATION	N TO:				
PLEASE PRINT		COMPANY			
OR TYPE		ADDRESS _			
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#### IMPORTANT NOTICE

RESERVATIONS FOR THE ATLANTA HILTON WILL BE ACCEPTED FOR ARRIVAL BEGINNING WEDNES-DAY, OCTOBER 29, ONLY. ANY RESERVATIONS REQUESTING HILTON ACCOMMODATIONS PRIOR TO WEDNESDAY, WILL BE ASSIGNED TO ANOTHER HOTEL.

#### HOTEL INFORMATION AND RATES

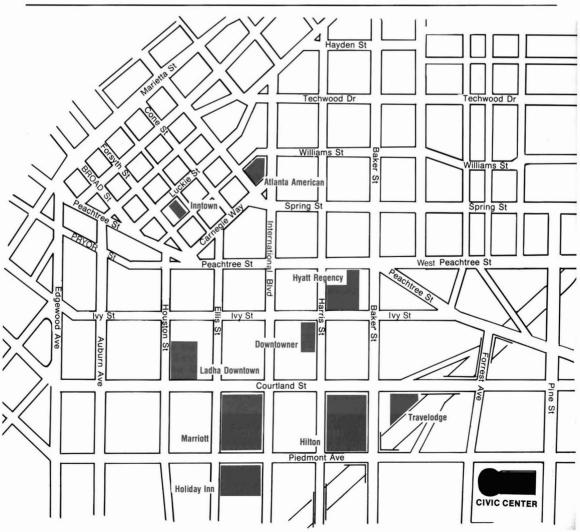
Nine hotels in Atlanta have reserved blocks of rooms for the 1980 Annual Meeting and Paint Industries' Show of the Federation. Please refer to map below.

All room rates are subject to an additional 7% charge-an Atlanta tax.

Confirmation will be sent to you directly by the hotel. Please make any changes with the Housing Bureau, in writing only.

IMPORTANT NOTICE: Reservations for the Atlanta Hilton will be accepted for arrival beginning Wednesday, October 29, only. Any reservations requesting Hilton accommodations prior to Wednesday, will be assigned to another hotel.

Hotel	Singles	Doubles/Twins	Parlor & 1 Bedroom	Parlor & 2 Bedrooms
ATLANTA HILTON (Headquarters)	\$52-75	\$68-91	\$160 up	\$230 up
HOLIDAY INN	35	39	56 up	112 up
HYATT REGENCY	46-66	58-76	145 up	225 up
MARRIOTT	45-62	57-74	130 up	190 up
ATLANTA AMERICAN (Quality Inn)	36	44	75 up	_
DOWNTOWNER MOTOR INN	37	47	150 up	_
INNTOWN MOTOR HOTEL	32	38-42	115 up	140 up
TRAVELODGE CENTRAL	30	35-40	_	-
LADHA DOWNTOWN HOTEL	36	42-46	125 up	167 up



## FEDERATION SERIES ON COATINGS TECHNOLOGY

#### Please enter my order for the following Units at the price of \$2.50 each:

Number				Price
Of Copies	Unit	Title		(@ \$2.50)
	1	"Introduction to Coatings Technology"-W. R. Fuller. (Oct. 1964) (Revised May 1973)	\$	
	2	"Formation and Structure of Paint Films"-W. R. Fuller. (June 1965)	\$	
	з	"Oils for Organic Coatings"—F. L. Fox. (Sept. 1965)	\$	
	4	"Modern Varnish Technology"—A. E. Rheineck. (May 1966)	\$	
	5	"Alkyd Resins"—J. R. Blegen. (Mar. 1967)	\$	
	6	"Solvents"—W. R. Fuller. (May 1967)	\$	
	7	"White Hiding and Extender Pigments"—W. H. Madson. (Oct. 1967)	\$	
	8	"Inorganic Color Pigments"-W. R. Fuller and C. H. Love. (Mar. 1968) Not available at this time	; 0	ut of stock.
	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)	\$	
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Two awards will be presented during the luncheon: The Heckel Award—for outstanding contributions to the Federation and the industry; and Flynn Awards—for the best six exhibits in the Paint Show.

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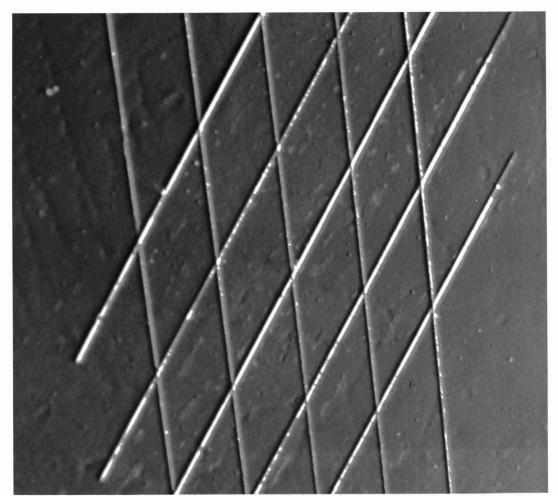
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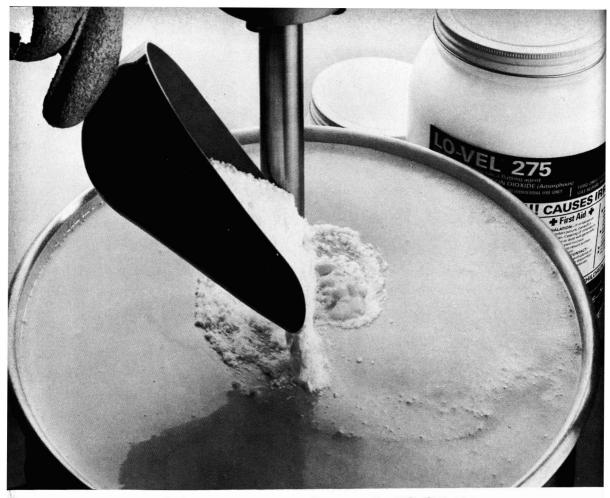
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PPG: a Concern for the Future



## Reactivity of Etherified Aminoplast Resins

J. Owen Santer and George J. Anderson Monsanto Plastics and Resins Company\*

An important trend within the coatings industry is to formulations which cure at lower temperatures and/or in shorter times. For aminoplast-crosslinked thermosetting formulations, improved cure response can be achieved by modifications of both the aminoplast and the primary film-former (vehicle). This paper discusses the effect of such modifications and describes experiments in which aminoplast resins were reacted with hydroxylfunctional model vehicles in order to gain insight into the effective functionality/reactivity of the various aminoplasts and to indicate routes towards more efficient low temperature cure.

#### INTRODUCTION

Industrial coatings crosslinked by melamine resins are usually cured in gas-fired ovens at temperatures in excess of  $250^{\circ}$  F (121° C). If proper cure could be obtained below this temperature, the ovens could be heated by low pressure steam generated from coal. Aside from the likely savings in energy consumption, this is a particularly desirable goal in view of possible limitations in the supply of natural gas.

High solids methylated aminoplast resins offer a number of advantages over conventional (butylated) resins. These include lower levels of cure volatiles and little or no solvent contribution from the aminoplast during bakeout. An often-stated disadvantage of high solids methylated resins is the requirement of high temperature for cure. With properly designed resins, this need not be the case. Because of their highly blocked (etherified) structure, coreaction of aminoplast with vehicle is likely to predominate over self-condensation. This fact can be used to advantage in designing formulations which cure rapidly and at low temperatures to highly crosslinked coatings with properties comparable to those obtained from conventional systems cured at higher temperatures.

In order to be most effective, any investigation into the reduction of cure requirements would benefit from the development of a convenient test for the measurement of relative cure speed. Consideration of several approaches to this goal has led to the selection of the gel time test. This method of cure rate measurement depends simply on the very rapid rise in viscosity at the gel point in thermoset systems. When comparing similar systems, the reactions which lead to gelation are also those which lead to cure. There is, therefore, a relationship between gelpoint and cure-point, and the time-to-gel can be regarded as an index of the rate of the cure reaction. The method has the following advantages in use:

- (1) Is convenient to run;
- (2) Gives a numerical (time) value to cure rate;
- (3) Can be run at different temperatures;

(4) Can be run on both pure resins and total formulations; and

(5) Requires only that the material be liquid at the temperature of the test.

Initially, the gel time tests were carried out to determine the self-condensation reactivities of various high solids methoxymethylated melamine resins with no vehicle present. Reactivities were measured with and without catalyst (p-toluene sulfonic acid, PTSA). Reactivities of melamine resins were also measured in formulations with acrylic vehicle having either predominantly hydroxyl or carboxyl functionality. Again, the effect of catalysis on these crosslinking reactions was investigated. Aqueous and solvent-based acrylic formulations were also compared for relative cure speed.

In a more quantitative investigation of the curing reaction, the polymeric vehicle was replaced by highboiling hydroxyl-functional model compounds of known structure and molecular weight. Gel times were measured and related to aminoplast/model compound ratio, as well as to total available hydroxyl groups at fixed aminoplast/model compound ratios.

Equations have been developed and are presented which permit the calculation of equivalent weights and

Presented by Dr. Santer at the 57th Annual Meeting of the Federation of Societies for Coatings Technology in St. Louis, October 4, 1979. \*Technology Dept., Resin Products Div., 730 Worcester St., Indian Orchard, MA 01151.

			(1111 11 105 0)			
Catalyst	Amount	Resin A Combining Ratio M/F/MeOH 1/4.8/3.0	Resin B Combining Ratio M/F/MeOH 1/5.5/3.0	Resin C Combining Ratio M/F/MeOH 1/4.8/4.0	Resin D Combining Ratio M/F/MeOH 1/5.7/5.0	Resin E Combining Ratio M/F/MeOH 1/5.7/5.5
_	_	45+	45+	45+	45+	45+
PTSA	0.24% (Solids on solids)	8	8	9	32	40
All resins	are at 77% solids in isopro	panol.				

Table 1-Gel Times of Various Etherified Melamine Resins Cured Without Vehicle (Min at 135°C)

All results based on duplicate runs.

functionalities of methoxymethylated melamine resins in their reaction under carefully defined conditions with compounds having primary hydroxyl functionality.

#### **GEL TIME TEST RESULTS AND DISCUSSION**

#### Test Method

Measurements of gel time were made with a gel time meter.\* A motor-driven rotating rod is suspended in the sample which is maintained at the test temperature. At the gel point, the very rapid rise in viscosity of the sample greatly increases the torque on the rotating rod thereby closing an electrical timer switch.

A five-gram sample of freshly prepared formulation in an  $18 \times 150$  mm test tube was used in all experiments. The rod was of 6 mm diameter glass immersed to a depth of about 25 mm.

#### Gel Times in Absence of Vehicle

High solids methoxymethylated melamine resins may be synthesized in a wide range of compositions which include almost pure hexamethoxymethyl melamine (HMMM) as well as structures with less combined formaldehvde and methanol.

In the absence of catalyst and vehicle, these resins are all extremely slow curing (Table 1) and do not gel in 45 min at 135°C. This result is expected and simply indicates that self-condensation of etherified melamines is very slow at the basic pH at which these resins are supplied.

\*Sunshine Scientific Instrument Co., Philadelphia, PA 19115.

Table	2—Effect of Tolerance on Gel Times
	Of Etherified Melamine Resins

Experimental Resin (No.)	Modified ASTM Tolerance	Gel Times (min) at 135° C With 1% PTSA			
1	1.7	11			
2	3.3	15			
3	4.3				
4	5.5				
5	6.3	40			

However, cure rate, i.e., self-condensation of these resins, is significantly affected by addition of PTSA catalyst. In the presence of catalyst, the resins can be grouped into three categories of cure:

Fast	•		•	•	•	•	•	•	•	•	•	•	•	•	•	Resins A, B, C <sup>†</sup>
Mode	era	t	e		•	•	•	•		•	•	•	•		•	Resin D
Slow	•	• •	•	•	•	•	•	•	•	•	•	•	•	•	•	Resin E

Resin E, which most closely approximates HMMM, is slowest curing by self-condensation while the slightly less etherified analog, Resin D, is moderate. This indicates the importance of -NH-, -NH<sub>2</sub>, or -CH<sub>2</sub>OH groups in the self-condensation reaction. Those resins containing free -NH-,  $-NH_2$ , and  $-CH_2OH$ groups are fastest curing but within the series show little effect relative to the absolute concentration of these species.

For etherified amino resins, the degree of etherification is normally indicated in part by the tolerance of the resin for hydrocarbon solvents (the higher the degree of etherification, the higher the tolerance). The effect of etherification on cure rate is shown in Table 2 for an experimental methylated resin reacted to increasing tolerance levels. For a 3.7-fold increase in tolerance for this resin, there is almost a four-fold decrease in cure rate, thus indicating the effect of methylol groups on condensation. However, this difference in cure rate narrows upon formulation with vehicle, although the effect is still noted.

#### **Gel Times of Aminoplast/Vehicle Formulations**

Aminoplast resins are generally used in the coatings industry not by themselves but rather as crosslinkers for various vehicles. When cured, the combination of functional vehicle and aminoplast crosslinker forms a hard. protective coating. The cure rate and coating properties are influenced by: (1) Reactivity of the crosslinker; (2) Type and level of functionality in the vehicle; (3) Catalysis; (4) Relative concentration of reactants; and (5) Temperature.

The apparent cure rates of various unpigmented aqueous amino resin/acrylic vehicle formulations are given in Table 3. The two acrylic vehicles used differ by

The compositions of Resins A-E are given in Table 1.

Acrylic/Aminoplast	Temp. °C	Resin A Combining Ratio M/F/MeOH 1/4.8/3.0	Resin B Combining Ratio M/F/MeOH 1/5.5/3.0	Resin C Combining Ratio M/F/MeOH 1/4.8/4.0	Resin D Combining Ratio M/F/MeOH 1/5.7/5.0	Resin E Combining Ratio M/F/MeOH 1/5.7/5.5
70/30 Acrylic/Aminoplast (COOH-functional)	135	24	23	25	48	126
70/30 Acrylic/Aminoplast (OH-functional)	135	15	16	17	27	61

### Table 3—Gel Times (Min at 135°C) of Acrylic/Etherified Aminoplast Formulations (Uncatalyzed)

having predominantly carboxyl versus predominantly hydroxyl functionality while the amino resins again range from almost pure HMMM (Resin E) to the lesser reacted models. When gel times were measured for combinations of vehicle and aminoplast without catalyst, three categories of cure were again obtained:

Fast	 Resins A, B, C
Moderate	 Resin D
Slow	 Resin E

These results are the same as those obtained in the selfcondensation of the amino resins and might indicate that this reaction is still predominant in the acrylic/aminoplast formulations, with the COOH group of the acrylic functioning as a catalyst, albeit of questionable efficiency. However, some coreaction of the amino resin with the vehicle is also apparent, since the predominantly hydroxyl-functional vehicle gives much faster cure than the predominantly carboxyl-functional vehicle.

We then investigated the effect of catalyst on coreaction of aminoplast with vehicle. The results of this study are shown in *Table* 4.

As noted before, without externally added catalyst, the reactive amino crosslinker containing -NH- and  $-CH_2OH$  produces a faster curing formulation than does the resin approaching the hexamethoxymethyl structure. However, only a small amount of PTSA is required to equalize or even reverse the apparent cure rates of the two systems. Since, as will be shown, aminoplast resins approximating the HMMM structure do not

readily self-condense even under strong acid catalysis, the inference must be that rapid coreaction of aminoplast with vehicle takes place in the presence of PTSA.

Table 4 also lists cure rate ratios R (R = [Gel Time at  $135^{\circ}$ C]/[Gel Time at  $150^{\circ}$ C]) for the various formulations. For those which contain catalyst, values of R generally decrease as the catalyst level is increased.

The acid-catalyzed transetherification reaction is generally considered to involve an initial, rapid protonation of ether oxygen, followed by a slow (rate determining) exchange reaction.

$$\sim$$
NCH<sub>2</sub>-OCH<sub>3</sub> + H<sup>+</sup>  $\xleftarrow{\text{fast}} \sim$ NCH<sub>2</sub>- $\circ$ 

$$\sim \text{NCH}_2 \xrightarrow{\Phi} (H_1 + \text{ROH} \xrightarrow{\text{slow}} \sim \text{NCH}_2\text{OR} + \text{CH}_3\text{OH} + \text{H}^*$$

The reaction is, thus, second order in  $\sim NCH_2 - \mathring{O}_{CH_2}$ 

and ROH, or pseudo-second order in  $\sim NCH_2OCH_3$  and ROH. The pseudo-order constant (and hence the reaction rate) will depend on the hydrogen ion concentration; the rate ratio R, however, should not be dependent on hydrogen ion concentration.

One possible explanation for the observed decrease in R is that, at the higher catalyst levels, other reactions (e.g., those leading to self-condensation of the aminoplast, as well as esterification between aminoplast and carboxylic acid groups on the vehicle) become increas-

### Table 4—Gel Times of Acrylic<sup>a</sup>/Aminoplast Formulations

			2/1 Acrylic/Resin	D¢		2/1 Acrylic/R esin	Bď
Catalyst <sup>b</sup>	Amount	135°C	150°C	Cure Rate Ratio R	135° C	150° C	Cure Rate Ratio R
PTSA	0 0.5%	31 min. 11 min.	14 min. 6 min.	2.2	20 min. 11 min.	9 min. 6 min.	2.2 1.8
	1.0%	7 min.	5 min.	1.4	11 min.	5 min.	2.2

(a) OH/COOH functional.

(b) Catalyst is % by weight (solids on solids) on aminoplast resin.

(c) Resin D: Melamine/CH2O/CH1OH = 1/5.7/5.0.

(d) Resin B: Melamine/CH2O/CH3OH = 1/5.5/3.0.

Table 5—Gel Times (Min/135°C) of Solvent Vs. Water-Based Acrylic/Aminoplast Formulations<sup>a</sup>

ingly important, especially with more highly etherified resins such as Resin D.

Assuming that, at the lowest catalyst level (0.5%), the 2/1 Acrylic/Resin D formulation cures predominantly via transetherification, the measured rate ratio (1.8) corresponds to an activation energy of 13.4 kcal/mole for this reaction. For the acid-catalyzed self-condensation of HMMM, which also involves an initial protonation step, followed by elimination of methanol, Saxon and Lestienne<sup>1</sup> have calculated an activation energy of 14.5 kcal/mole.

When relating apparent cure speed, i.e., gel time, of acrylic formulations to actual application properties, a good correlation is obtained in most key properties. However, there are some exceptions. When aqueous acrylic formulations containing the HMMM type of crosslinker are adjusted in cure speed to equal that of the more reactive resins, film properties such as hardness, solvent resistance, and percent nonextractables of the two formulations are essentially equal, but humidity whitening is greater with the HMMM-containing formulation.

Some indication of the importance of investigating factors influencing cure rate is given in *Table* 5 which

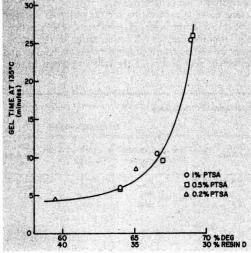


Figure 1-Gel times at 135°C for Resin D/DEG formulations

compares the gel times of solvent and water-based formulations utilizing the same basic acrylic vehicles. The differences in gel times are presumably due to the increased energy necessary to vaporize water and the time required to volatilize the neutralizing amine needed to solubilize the vehicle.

### Gel Times of Aminoplast/Model Compound Formulations

In order to facilitate a more fundamental study of the curing reaction the polymeric vehicle was replaced by hydroxyl functional 'model compounds of known structure and equivalent weight. It is necessary that these model "vehicles" be high boiling and be compatible with each other and with the aminoplast and catalyst. Diethylene glycol (DEG) and trimethylol propane (TMP) were selected to satisfy these requirements. Figure 1 shows the effect on gel time of both aminoplast/polyol ratio and catalyst level (PTSA) on a Resin D/DEG formulation (the composition of Resin D is given in Table 1). The principal conclusion drawn is that the gel time is very much dependent on the aminoplast/DEG ratio, and essentially independent of catalyst level (in the range of 0.2-1%).

When DEG is replaced by a combination of DEG and TMP, it becomes possible by varying the ratio of DEG (difunctional) to TMP (trifunctional) to vary the OHcontent of the formulation while holding the aminoplast/polvol ratio constant. Figure 2 shows the effect of OH-content on gel times at aminoplast/polyol ratios of 30/70 and 33/67. It will be seen that gel times are significantly shortened by either (a) increasing the TMP/DEG ratio at constant aminoplast/polyol ratio, which increases both the total OH-content and the probability that crosslinking will occur, or (b) increasing the aminoplast/polyol ratio. For example, the graph shows that gel times can be reduced by about one-half at the lowest OH-concentration simply by increasing the aminoplast/ polyol ratio from 30/70 to 33/67. A similar reduction in gel time can be brought about by increasing the hydroxyl content from about 19 meq/gm to about 20 meq/gm, at either of the aminoplast/polyol ratios.

These results are far more dramatic than those seen with actual coatings formulations which are generally much lower in total hydroxyl-content. As will be shown, wide variations in gel times are likely at very high hydroxyl levels (see *Figure 3* and related discussion). Nevertheless, the principal conclusions to be drawn will still apply. In particular, significant increases in cure speed should be possible by increasing the hydroxylfunctionality of the vehicle and by increases in aminoplast levels with the higher OH functionality vehicles.

### AMINOPLAST FUNCTIONALITY

### Theory

When compound A having a functionality<sup>2</sup> greater than two reacts with difunctional compound B, whether or not the formulation gels will depend on both the ratio of the two compounds and the functionality of A. Flory<sup>3</sup> has discussed this aspect of polyfunctional condensation polymerization in terms of a branching probability  $\alpha$ , where  $\alpha$  is the probability that a polymer chain begins and ends in a branch unit. For this reaction:

$$\alpha = P_{B}^{2}/r \qquad = P_{A}^{2}r \tag{1}$$

Where  $P_A$ ,  $P_B$  = fractions of functional groups on A, B which have reacted.

r = ratio of total number of A functional groups to total number of B functional groups present at start of reaction.

Further, if compound A has functionality f, then gelation will occur when  $\alpha(f-1)$  exceeds unity. That is:

$$\alpha c = 1/(f-1) \tag{2}$$

where  $\alpha c$  is the critical branching probability.

Now, let A represent an aminoplast resin having an average of f alkoxymethyl groups per molecule (f > 2) and let B represent a difunctional primary hydroxyl compound. Assume that reaction between A and B is exclusively one of transetherification.

We may choose a suitable ratio  $r = r_c$  such that gelation does not occur even when reaction goes to completion. Then:

$$\alpha c = P_A^2 r_c; P_A = 1 \tag{3}$$

Hence, from (2)

$$\alpha c = r_c = 1/(f-1) \tag{4}$$

We now express r and  $r_c$  in terms of the weight ratio of A to B. Thus,

$$r = \frac{\text{total number of alkoxymethyl groups initially present}}{\text{total number of primary OH groups initially present}}$$

That is,

 $\mathbf{r} = [\mathbf{W}_{A}\mathbf{f}/\mathbf{M}_{A}]/[2\mathbf{W}_{B}/\mathbf{M}_{B}]$ (5)

Where  $W_A, W_B$  = weights of A, B initially present  $M_A, M_B$  = molecular weights of A, B.

Rearranging

 $r = [W_A/W_B][M_Bf/2M_A]$ (6)

and 
$$r_c = [1/W_c][M_B f/2M_A]$$
 (7)

Where  $W_c =$  the critical weight ratio of B to A above which gelation will not occur.

Combining equations (4) and (7)

$$1/(f-1) = [1/W_c][M_B f/2M_A]$$
(8)

This expression rearranges to a quadratic in f, from which, rejecting the negative value, we obtain:

$$f = 1/2 \{1 + [(M_B + 8M_AW_C)/M_B]^{1/2}\}$$
(9)

In general,  $M_A$  and  $M_B$  are known, and  $W_c$  can be determined experimentally using the gel meter.

Although equation (9) was derived with the assumption that reaction was exclusively one of transetherification between A and B, in principle it is still valid if selfcondensation of the aminoplast also takes place. In that event, the value of  $W_c$  obtained experimentally will be higher, reflecting the increase in f as aminoplast coupling occurs. However, calculation of f ought also to take into

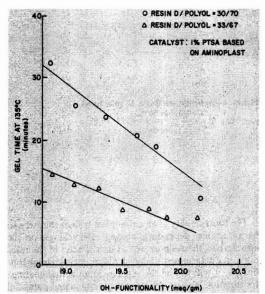


Figure 2—Gel times at 135°C. Effect of OH-Content at fixed aminoplast/polyol ratios

account the new, higher value of  $M_A$  which results from the self-condensation. Unfortunately this is not readily ascertainable. For this reason, application of equation (9) is limited to those systems where self-condensation is less likely to take place, so that  $M_A$  remains essentially constant.

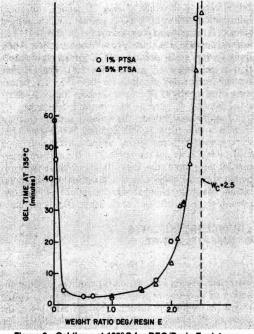


Figure 3—Gel times at 135°C for DEG/Resin E mixtures

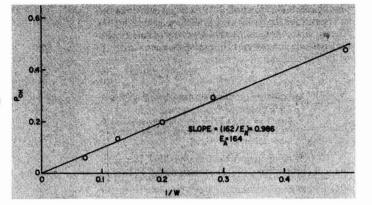


Figure 4—Reaction of Resin E with butyl Carbitol at 50°C

The functionality of an aminoplast resin is thus seen to be a variable, depending, among other things, on the structure of the resin as well as on the ratio of the reactants. As determined under idealized experimental conditions, it will not be the same as in an actual coatings formulation; at best we can expect only to make a useful correlation between them.

### **Measurement of Functionality**

As a first step in the application of equation (9), acidcatalyzed formulations of Resin E, which approximates the HMMM structure, were prepared with DEG and gel times measured at 135°C (*Figure* 3). From the graph,  $W_c = 2.5$ . For DEG,  $M_B = 106$ , while Resin E has a number average molecular weight = 450. Substituting in equation (9) we obtain f = 5.1.

Besides providing a means for estimating  $W_c$  for the DEG/Resin E mixture, Figure 3 carries additional information. When no DEG is present (i.e., the weight ratio = zero) gelation is very slow (~58 min at 135°C) and is, of course, due entirely to self-condensation. Addition of a very small amount of DEG gives a rapid reduction in gel time (~3 min at W = 0.4). The gel time remains reasonably constant for W = 0.5-1.5, before increasing rapidly as  $W_c$  is approached. Catalyst level has little effect on gel time, at least in the 1-5% range.

Additional insight into the actual reactivity in commercial coating formulations is gained from Figure 3. At least 6 meq OH per gram, which corresponds to a DEG/Resin E weight ratio of 0.32, of Resin E are necessary for most rapid cure. Below this level, there are insufficient sites for most effective crosslinking, i.e., coreaction. Many commercial vehicles suffer from this disadvantage, i.e., a deficiency of available OH functionality.

### EQUIVALENT WEIGHTS OF AMINOPLAST RESINS

### Theory

Implementation of equation (9) requires the determination of only one experimental quantity ( $W_c$ ). To the extent that self-condensation reactions take place, the value of  $W_c$  obtained will lead to inaccuracies in the determination of f, as previously described. In order to obtain more information on the reactivity of aminoplast resins with model OH-compounds at varying aminoplast/model compound ratios, an alternative approach was developed which avoids some of the problems inherent in equation (9).

When methylated melamine resins are reacted at moderately low temperatures (~ 50°C) with a compound having primary OH functionality, using PTSA catalyst, the principal reactions which take place are (a) etherification and (b) transetherification. There is little tendency toward self-condensation, particularly if the primary OH-containing compound is present in excess and the melamine has a high level of combined formaldehyde. The by-products of the two principal reactions are (a) water and (b) methanol, both of which can be removed from the reaction medium by volatilization under appropriate conditions. Provided that the primary OH compound is nonvolatile, the extent to which reactions (a) and (b) occur can be determined by measuring the extent of reaction of the primary hydroxyl compound when reaction is complete, and this information is used to determine an equivalent weight for the melamine resin under the reaction conditions.

Thus, consider the reaction of compound B, having primary OH functionality, with an etherified aminoplast Resin A.

Assume B to be present in excess, and assume that the reaction is exclusively transetherification. Compound B may be either mono- or polyfunctional.

Let $W$ = weight ratio of B to A.
Let $E_B =$ equivalent weight of B.
Let $E_{\Lambda}$ = equivalent weight of A (that is, the weight of A containing one reactive

Then, in one part by weight of the mixture of B with A, we have:

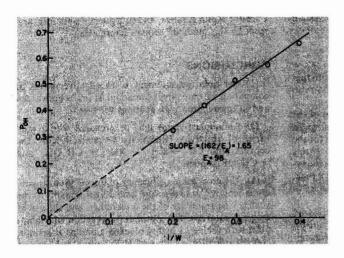
and  $I/E_A(W + 1)$  equivalents of A.  $W/E_B(W + 1)$  equivalents of B.

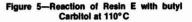
Allow reaction of B with A to proceed to completion.

Then the fraction  $P_{\rm OH}$  of primary OH groups reacted is given by:

$$P_{OH} = [1/E_{A}(W+1)]/[W/E_{B}(W+1)]$$
(10)

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### or rearranging

$$\mathbf{P}_{\rm OH} = \left[\mathbf{E}_{\rm B}/\mathbf{E}_{\rm A}\right] \left[1/\mathbf{W}\right] \tag{11}$$

Equation (11) is linear in  $P_{OH}$  and 1/W. If  $E_B$  is known, the value of  $E_A$  can be calculated from the slope of a plot of  $P_{OH}$  versus 1/W.

### **Measurement of Equivalent Weight**

Diethylene glycol monobutyl ether (butyl Carbitol®) is a useful compound for testing the validity of equation (11), since it is monofunctional and, therefore, cannot cause gelation, is compatible with methoxymethylated melamine resins, and has been shown neither to volatilize (bp 231°C) nor to self-condense under the reaction conditions.

Acid catalyzed (1% PTSA, on aminoplast) formulations of butyl Carbitol (equivalent weight = 162) with

Butyl Carbitol is a registered trademark of Union Carbide Corp.

Resin E in varying ratios were prepared and conditioned at 50°C until no further consumption of OH was seen. Analysis for OH was made by esterification with 3-nitrophthalic anhydride, followed by back-titration with benzyltrimethyl-ammonium hydroxide. This is a modified version of the procedure described by Floria, et. al.<sup>4</sup>

A graph of  $P_{OH}$  versus 1/W (*Figure* 4) shows an excellent straight line relationship. From the slope of the line, the equivalent weight of Resin E is 164.

When the acid-catalyzed Resin E/butyl Carbitol reaction is run to completion at 110° C, and the results plotted graphically, a straight line relationship is again obtained (*Figure 5*). From the slope, the equivalent weight of Resin E is 98 at this temperature.

The measured equivalent weight of Resin E thus depends strongly on the test temperature. The lower value obtained at 110°C implies the reaction at this temperature of methoxymethyl groups which were unreactive at

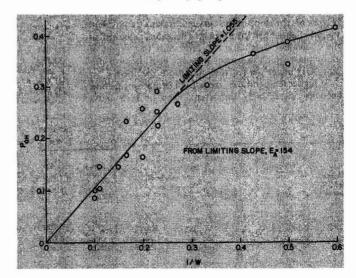


Figure 6—Reaction of Resin A with butyl Carbitol at 50°C 50°C. Calculation of the average functionality of Resin E  $(M_n = 450)$  at these two temperatures gives:

$$\begin{array}{l} f_{50}=450/164=2.7\\ f_{110}=450/98=4.6 \end{array}$$

The functionality at  $110^{\circ}C(4.6)$  is consistent with that obtained at  $135^{\circ}C(5.1)$  using the critical weight ratio method.

These results suggest that the rate of reaction of the first methoxymethyl group on a given nonring nitrogen atom is very rapid compared to the reaction rate for the second methoxymethyl group; as a practical matter this means that at lower temperatures only three methoxymethyl groups per triazine ring will react in a given time, while at higher temperatures it is possible for all six to react.

A similar experiment was run with a PTSA catalyzed Resin A/butyl Carbitol formulation at 50°C (*Figure* 6). Resin A is substantially lower in both combined formaldehyde and methanol than Resin E, and has, therefore, a greater tendency to self-condense. Additionally, because of its higher ratio of  $-CH_2OH/-CH_2OCH_3$ groups, reaction with butyl Carbitol generates a higher ratio of  $H_2O/CH_3OH$ . The high proportion of  $H_2O$  can lead to hydrolysis and, hence, to further self-condensation besides being a potential source of interference in the analysis for unreacted butyl Carbitol. Nevertheless, for high ratios of butyl Carbitol/Resin A (i.e., low values of 1/W) *Figure* 6 shows a good straight line relationship, from which an equivalent weight of 154 was calculated.

At low ratios of butyl Carbitol/Resin A (i.e., high values of 1/W), Figure 6 indicates a deviation from linearity. This is attributed to an increase in self-condensation which leads to an increase in equivalent weight.

By measuring the slope of the tangent to the nonlinear portion of the curve, an equivalent weight determination can be made for low butyl Carbitol/Resin A ratios, and at least in principle, this figure could be used as a guideline in estimating the equivalent weight of Resin A in any given coatings formulation of known OH functionality. For example, for values of 1/W in the range of 0.5-0.6 (*Figure* 6), the slope of the curve indicates an equivalent weight of about 630 for Resin A. This value, as with all of the equivalent weights given, represents the weight of aminoplast which will react with one equivalent of primary OH under the reaction conditions specified.

At higher temperatures, the self-condensation reaction becomes increasingly important, even at high butyl Carbitol/Resin A ratios, and equivalent weight measurement by this method becomes impractical.

### CONCLUSIONS

(1) Gel time measurement is an effective means of comparing the cure rates of aminoplast resins both alone and in formulation with coatings vehicles.

(2) Aminoplast resins may be prepared with a wide range of reactivity. Reactivity is controlled by the concentration of -NH,  $-NH_2$ ,  $-CH_2OH$ , and  $-CH_2OR$  species.

(3) The crosslinking efficiency of formulations is governed mainly by the type and level of functionality of the vehicle, the reactivity of the crosslinker, and the use of external catalysis. For optimum coreaction of aminoplast with vehicle, a highly blocked aminoplast resin in conjunction with a high — OH functional vehicle and strong acid catalyst is preferred.

(4) Data obtained from the use of model "vehicles" strongly indicates that high levels of hydroxyl functionality are necessary to minimize the required cure temperature and/or time.

(5) The effective functionality of an amino resin is a variable parameter and depends on:

Temperature of cure. Level and type of functionality in the vehicle. Ratio of vehicle to aminoplast crosslinker. The combining ratio of melamine/formaldehyde/ alcohol in the crosslinker. Catalysis.

### ACKNOWLEDGMENT

The authors are indebted to G. R. Bowers and C. B. McGinn for much of the experimental work reported herein and to Dr. A. N. MacDonald for his advice on the mathematics of the functionality concept.

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### Nomograms for Solubility Parameter

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A simple equation which defines the effect of temperature on solubility parameter ( $\delta$ ) has been derived using the equation of Fedors and Watson. This equation has also been represented by a nomogram for finding  $\delta$  values at various temperatures provided the critical temperature and one value of  $\delta$ of a substance are known. The basic equation showing the correlation of  $\delta$  with molar volume, internal molar energy, and heat of vaporization has also been transformed into two nomograms for finding  $\delta$  at 25°C.  $\delta$  values obtained from these nomograms have been found comparable with those reported elsewhere.

### INTRODUCTION

Solubility parameter ( $\delta$ ), an expression of square root of cohesive energy density, is often used for determining the compatibility characteristics of polymers and solvents.<sup>1-6</sup> It is directly proportional to internal molar energy ( $\Delta E$ ) or heat of vaporization  $(\Delta H_v)$  and inversely proportional to molar volume (V):

$$\delta = \left(\frac{\Delta E}{V}\right)^{0.5} = \left(\frac{\Delta H_V - RT}{V}\right)^{0.5} \tag{1}$$

The value of  $\delta$  at a desired temperature can be obtained from this equation provided other variables are known at that temperature. Mathematical expressions<sup>7,8</sup> exist which define effect of temperature on  $\delta$ , but they involve more than one variable. Therefore, it is felt to be worthwhile to derive an expression which correlates  $\delta$  directly to temperature.

The heat of vaporization, molar volume, and density  $(\rho)$  of a substance vary with temperature. Consequently,

δ also varies. Fedors<sup>9</sup> reports temperature dependance of  $\delta$  by the following two equations:

$$\frac{\Delta H_{V_{T_2}}}{\Delta H_{V_{T_1}}} = \left(\frac{\rho_{T_1}}{\rho_{T_1}}\right)^{1.27}$$

$$\frac{\delta_{T_2}}{\delta_{T_1}} = \left(\frac{\rho_{T_2}}{\rho_{T_1}}\right)^{1.13}$$
(2)
(3)

(3)

and.

The subscripts  $T_1$  and  $T_2$  represent the variables at two different temperatures. The grouping of equations (2) and (3), thus, results in:

$$\frac{\delta_{T_2}}{\delta_{T_1}} = \left(\frac{\Delta H_{V_{T_2}}}{\Delta H_{V_{T_1}}}\right)^{0.89}$$
(4)

Earlier,<sup>10</sup> it had been found that the effect of temperature on  $\Delta H_v$  was perhaps better defined by Watson's equation than by others' equations:

$$\frac{\Delta H_{V_{T_2}}}{\Delta H_{V_{T_1}}} = \left(\frac{1 - T_{r_2}}{1 - T_{r_1}}\right)^{-0.38}$$
(5)

Here,  $T_{r_1} = T_1/T_C$  and  $T_{r_2} = T_2/T_C$ . T<sub>C</sub> is the critical temperature.

The following equation, obtained by equating equation (4) with equation (5), depends on a single variable (T):

$$\delta_{2/}\delta_1 = \left(\frac{1 - T_{r_2}}{1 - T_{r_1}}\right)^{0.34}$$
(6)

### Table 1-Data Used to Construct Nomogram (Figure 1)

	Mod	ulus			Length of Scale	
Axis m		cm	Range	Scale	in cm	
ΔE	25/0.5	50	2 to 14 Kcal	25 log 14/2	21.13	
v	20/0.5	40	30 to 300 cc	20 log 300/30	20.00	
δ	22.22	22.22	4 to 15 (cal/cc) <sup>1/2</sup>	22.22 log 15/4	12.76	

<sup>\*</sup>Surface Coatings Div., Hyderabad-500 009, India.

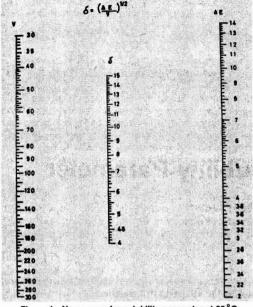


Figure 1-Nomogram for solubility parameter at 25°C

To find  $\delta$  at the desired temperature, the input data required for this equation are T<sub>C</sub> and the  $\delta$  value at a known temperature. The results obtained by transforming equations (1) and (6) into nomograms are discussed here.

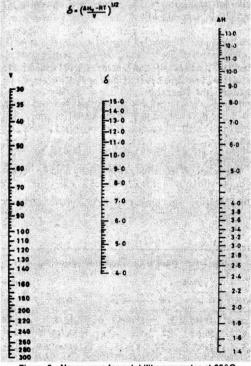


Figure 2-Nomogram for solubility parameter at 25°C

Table 2-Data Used to Construct Nomogram (Figure 3)

Modulus Axis m cm		ulus			Length
		cm	Range	Scale	of Scale in cm
(1-T <sub>r1</sub> )	40/0.34	117.64	0.99 to 0.30	$40\log\frac{0.99}{0.30}$	20.74
(1-T <sub>r2</sub> )	40/0.34	117.64	0.99 to 0.30	40 $\log \frac{0.99}{0.30}$	20.74
$\delta_1/\delta_2$	58.82	58.82	1.50 to 0.75	$58.80 \log \frac{1.5}{0.75}$	17.71

Nomography is the simplest means of presenting an equation or data to find the value of a variable under a given set of conditions. Construction of a nomogram requires the limiting values or ranges of the variables and the relationship by which they are correlated. The moduli or units are so selected that the variables could be represented by three different scales of convenient lengths.<sup>11,12</sup> These principles have been used for constructing nomograms for  $\delta$  at 25°C and at different temperatures.

### CONSTRUCTION OF NOMOGRAMS

Equation (1) is rewritten as:

 $\log \delta = 0.5 \log \Delta E - 0.5 \log V \tag{7}$ 

The arbitrary values of the moduli for  $\Delta E$  and V are so chosen that the lengths of the scales which represent these

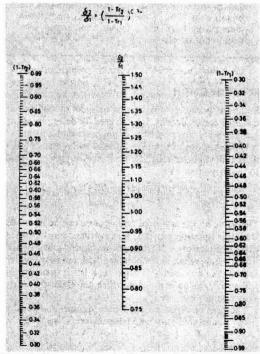


Figure 3-Nomogram for solubility parameter at any temperature

		Molar			Nomo	arem	
		Volume	ΔH25°C	∆E25°C			
Com	pound	V25°C	K cal.	Kcal.	1	2	Literature <sup>13</sup>
Hyd	rocarbons						
1.	Propane	. 75.33	3.61	3.01	6.40	6.70	6.23
2.	Butane	. 96.87	5.05	4.44	6.70	6.95	6.79
3.	Pentane	. 115.26	6.32	5.72	7.15	7.20	7.09
4.	Hexane	130.50	7.54	6.95	7.40	7.35	7.33
	Heptane		8.74	8.14	7.50	7.40	7.46
	Octane		9.92	9.32	7.60	7.50	7.54
	Nonane		11.10	10.51	7.80	7.55	7.58
	Decane		12.28	11.68	7.90	7.60	7.60
	hols Methanol	. 40.41	9.11	8.52	14.60	14.50	14.46
	Ethanol		10.47	9.88			13.00
	Propanol				13.20	13.00	
	Butanol		11.33	10.74 11.74	12.10 11.50	12.00	12.05
Este		62 10	6 94	6.25	10.20	10.20	10.23
	Methyl formate		6.84	6.25	10.20	10.20	
	Ethyl formate		7.62	7.03	9.40	9.40	9.38
3.	Propyl formate	. 97.82	8.60	8.00	9.15	9.05	9.12
4.	Methyl acetate	. 79.88	7.74	7.15	9.50	9.60	9.49
5.	Ethyl acetate	. 97.79	8.52	7.93	9.05	9.00	9.00
	Propyl acetate		9.41	8.82	8.80	8.70	8.77
	Methyl propionate		8.58	7.98	9.20	9.10	9.10
	Ethyl propionate		9.37	8.78	8.80	8.75	8.76
	Isobutyl formate		9.13	8.54	8.60	8.60	8.63
	soamyl formate		10.08	9.49	8.45	8.40	8.50
	Isopropyl acetate		8.94	8.35	8.50	8.45	8.47
12.	Isoamyl acetate	. 149.65	10.77	10.17	8.30	8.00	8.34
Ket	ones						
1.	Acetone	. 73.33	7.66	7.07	9.90	9.95	9.82
2.	Ethyl methyl ketone	. 89.57	8.51	7.92	9.60	9.50	9.40
3.	Methyl isobutyl ketone	. 124.94	10.00	9.41	8.65	8.65	8.68
	Methyl isopropyl ketone		8.94	8.35	8.80	8.85	8.89
	Methyl propyl ketone		9.31	8.71	8.95	9.00	\$ 9.06
	Diethyl ketone		9.32	8.72	9.00	9.05	9.04
							8.79
	Methylbutyl ketone		10.45	9.86	9.10	9.00	
	Methyl amyl ketone		11.33	10.74	8.80	8.70	8.75
	Ethyl butyl ketone Dipropyl ketone		11.27 11.09	10.68 10.50	8.80 8.80	8.60 8.55	8.67 8.70
10.		. 139.09	11.09	10.50	0.00	0.55	0.70
Eth							
	Diethyl ether		6.45	5.86	7.60	7.60	7.51
2.	Methyl isopropyl ether	. 100.88	6.39	5.79	7.60	7.65	7.58
3.	Ethyl propyl ether	. 118.01	7.60	7.01	7.80	7.80	7.71
4.	n-butyl ether	. 166.09	10.73	10.14	7.80	7.75	7.81
5.	Ethyl butyl ether	. 135.86	8.70	8.10	7.85	7.70	7.72
	Methyl ethyl ether		4.86	4.27	7.30	7.40	_
	Isopropyl ether		7.73	7.14	9.20	9.10	
Hal	ogenated Solvents						
	Carbon tetrachloride	. 96.45	8.16	7.57	8.90	8.90	8.86
	Chlorobenzene		9.84	9.24	9.60	9.45	9.53
	Ethyl chloride		5.79	5.19	8.65	8.70	8.61
	Chloroform		7.45	6.85	9.40	9.40	9.27
	Propyl chloride		6.95	6.36	8.60	8.60	8.49
	1-1-dichloro ethane		7.39	6.80	9.00	9.00	8.98
7.	1-2-dichloro ethane	. 78.74	8.34	7.75	10.00	10.00	9.92
Nit	ogenous Solvents						
	Acetonitrile	. 52.43	8.39	7.79	12.40	12.30	12.19
	Propionitrile		9.08	8.49	11.10	11.00	10.98
	Butyronitrile						10.37
			9.93	9.34	10.50	10.30	
	Benzonitrile		12.85	12.26	10.80	10.80	10.90
	Methyl amine		5.66	5.06	11.40	11.60	11.20
	Propyl amine		7.65	7.05	9.40	9.40	9.26
6.				0 30	0.35	0.10	9.10
6. 7.	Butyl amine		8.79	8.20	9.25	9.10	
6. 7.			8.79 5.83	5.24	9.25	9.10	8.89
6. 7. 8.	Butyl amine	. 66.30					

Table 3— $\delta$  Values at 25°C Obtained from Nomograms 1 and 2

δ Values from

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	δ Values at						Input Data	
Compound	0°C	0°C 20°C	40°C	60°C	80°C	100°C	тс	δ25°C
1. Hexane	7.58	7.37	7.19	6.85	6.60	_	507.3	7.26
2. Heptane	7.69	7.46	7.29	7.10	6.80	6.53	540.3	7.38
3. Propanol	12.59	12.21	11.95	11.51	11.14	10.75	536.7	12.09
4. Ethyl ether	7.83	7.59	7.29	-	_	-	465.8	7.44
5. Chloroform	9.48	9.19	8.99	8.67	8.39	8.09	536.6	9.10
6. Acetone	10.08	9.83	9.57	9.13	8.84		509.1	9.68
7. Aniline	10.74	10.53	10.37	10.10	9.97		698.8	10.42
8. Benzene	9.52	9.19	9.05	8.79	8.50	8.20	562.1	9.14
9. Methyl formate	10.30	9.99	9.65	9.28	_	-	487.2	9.84
10. Ethyl formate	9.60	9.33	9.08	8.70	8.38	-	508.5	9.22
11. Methyl acetate	9.76	9.46	9.18	8.79	8.47	_	506.9	9.32
12. Ethyl propionate	8.96	8.73	8.50	8.27	7.95	7.68	546.1	8.60
13. Toluene	9.20	9.01	8.81	8.62	8.38	8.12	592.0	8.92
14. Propionitrile	11.12	10.89	10.67	10.32	10.03	9.69	564.0	10.78
15. Diethyl ketone		9.09	8.91	8.61	8.37	8.09	561.0	9.00
16. Butanol		11.62	11.39	11.01	10.70	10.34	563.0	11.50

Table 4- $\delta$  Values at Different Temperatures Obtained from Nomogram 3

functions are within workable limits and, also, do not differ much from each other. Therefore, rational numbers 25 and 20 are taken as moduli for  $\Delta E$  and V, respectively. The modulus for  $\delta$  is calculated from the moduli of functions  $\Delta E$  and V:

$$m_{\delta} = \frac{m_{\Delta E} \times m_{V}}{m_{\Delta E} + m_{V}} = \frac{25 \times 20}{25 + 20} = 22.22 \text{ cm}$$
(8)

The moduli, ranges, and lengths of scales described in *Table* 1 are used for constructing the nomogram (*Figure* 1). The scales for  $\Delta E$  and V are drawn apart at an arbitrary distance of 16.5 cm and the distance of scale of  $\delta$  from the two is calculated.

$$\frac{m_{\Delta E}}{m_{\Delta E} + m_V} \times 16.5 = 9.17 \text{ cm}$$

$$\frac{m_V}{m_{\Delta E} + m_V} \times 16.5 = 7.33 \text{ cm}$$
(9)

and

Scales,  $\Delta E$  and V, are drawn from the same base line and the starting point for  $\delta$  scale is the point of intersection of lines joining  $\Delta E$  and V scales when  $\delta$  is 4. The nomogram in Figure 2, represents the relationship:

$$\delta = \left(\frac{\Delta H_{v} - RT}{V}\right)^{0.5}$$
(1)

and the procedure of its construction is the same as previously described. Equation (6), which defines the relationship between  $\delta$  and T, is expressed below in its logarithmic form:

$$\log \delta_2 / \delta_1 = 0.34 \log (1 - T_{r_1}) - 0.34 \log (1 - T_{r_1})$$
(10)

The moduli, ranges, and lengths of scales used for constructing the nomogram in *Figure 3* are shown in *Table 2*. Scales, (1-T<sub>r2</sub>) and 1-T<sub>r1</sub>), are drawn at a distance of 16.5 cm and  $\delta_1/\delta_2$  scale is drawn in the middle, as per calculations described for the first nomogram.

### **RESULTS AND DISCUSSION**

The values of  $\delta$  for some hydrocarbons, alcohols, esters, ketones, ethers, halogenated hydrocarbons, and nitrogenous solvents obtained from nomograms (*Figures*)

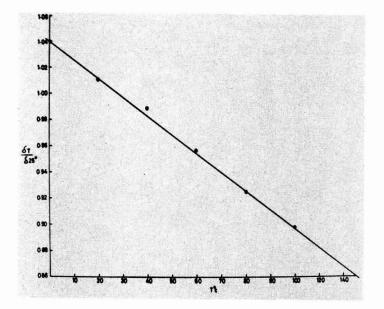
Table 5— $\delta$ Values for	Toluene and Ethy	Acetate at Different	Temperatures
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	δ Values at								
Compound	5°C	10°C	15°C	20 °C	30°C	35°C	40 °C	45°C	
Toluene <sup>a</sup>	9.08	9.05	8.99	8.95	8.86	8.80	8.73	8.68	
$\delta 25 ^{\circ} C = 8.90^{b}$	9.12	9.17	9.06	8.99	8.87	8.71	8.75	8.68	
$T_c = 592^{\circ}$	9.15	9.10	9.02	8.98	8.86	8.81	8.75	8.69	
Ethyl acetate <sup>a</sup>	8.84	8.77	8.72	8.66	8.52	8.46	8.38	8.30	
$\delta 25^{\circ} C = 8.59^{\circ}$	8.87	8.85	8.75	8.68	8.56	8.50	8.44	8.38	
$T_{C} = 523.3^{\circ}$	8.89	8.81	8.74	8.67	8.51	8.44	8.36	8.29	

(a) Values calculated from Nomogram 3.

(b) Values calculated from Figure 4.

(c) Values reported in literature.14



### Figure 4- $\delta T/\delta 25$ vs temperature

1 and 2) and those reported in the literature<sup>13</sup> are listed in *Table* 3 for comparison. *Table* 4 contains  $\delta$  values at 0, 20, 40, 60, 80, and 100°C, obtained from the nomogram in *Figure* 3 for a few selected compounds. In *Table* 5, the reported  $\delta$  values for toluene and ethyl acetate<sup>14</sup> at different temperatures are listed along with those obtained from the nomogram (*Figure* 3) and the plot (*Figure* 4).

A glance at the  $\delta$  values in *Table* 3 indicates that, in most cases, the  $\delta$  values obtained from the nomograms (*Figures* 1 and 2) are almost equal to the reported values. Usually  $\Delta H_V$  values for most of the compounds are reported in literature; they can also be obtained from nomograms published by Othmer.<sup>15</sup> A line joining the  $\Delta H_V$  scale with the V scale produces the desired  $\delta$  value without involving any calculations. This shows that  $\delta$ at 25°C for organic compounds belonging to different homologous series can easily be found from these nomograms.

The nomogram in Figure 3 is a simple representation of temperature dependance of  $\delta$ . To find  $\delta$  at a desired temperature from this nomogram,  $\delta$  at any temperature or at 25°C from any one of the nomograms (Figures 1 and 2) can be used. The line joining  $(1-T_{r_i})$  and  $(1-T_{r_i})$ scales gives  $\delta_2/\delta_1$  and, thus, the desired value of  $\delta$ . The results reported in Table 4 show the convenience of finding  $\delta$  at any temperature.

The comparison of results in Table 5 shows that, at temperatures from 0 to 45°C,  $\delta$  values for toluene and ethyl acetate reported elsewhere<sup>14</sup> are almost equal to those obtained from this nomogram and from Figure 4, even when the change in temperature is as little as 5°C.

The analysis of data reported in *Table* 4 indicates that  $\delta$  decreases linearly with an increase in temperature and the variation observed in  $\delta_T/\delta_{25}$  from solvent to solvent is virtually constant over the range 0 to 100 °C. The values of  $\delta_T/\delta_{25}$  have been calculated and the mean average values are plotted versus temperature (*Figure* 4). The plot is linear and useful for finding  $\delta$  for a solvent at any temperature, provided that the  $\delta$  value at one temperature is known.

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**Coatings Materials** 

# Condensation Polymer Formulation In the Age of Oligomers

Lawrence H. Brown Dow Corning Corporation\*

A review of the theoretical concepts of Carothers and Flory, applied by Kilb, Bernardo and Bruins, Patton, Finney, Earhart and others, shows that condensation polymers for solution coatings can be formulated according to incipient gelation criteria with reasonable assurance of success.

Today, with emphasis on oligomeric polymers for high solids, water-dispersible and powder coatings, the former concepts have less value.

For polyesters, Patton's constant (K = Mo/Ea) becomes less meaningful than theoretical  $\overline{M}n$  and  $EW_{OH}$  (weight of polymer per excess carbinol group). Reactions of the base polyesters with alkoxy-functional melamines, siloxanes or other modifiers during cure have changed the precepts on which formulations are based. This is true especially for the current rapid cures required for prefinished metal.

Application of the new criteria is facilitated by equations which can be handled by programmable calculators or computers. Modification by silicone oligomers has been reduced to a mathematical treatment, and examples show the utility of the method.

### INTRODUCTION

As late as 1936, Dr. Carothers and Professor Staudinger were still disputing<sup>1</sup> whether macromolecules from condensation reactions should be classified as polymers. It is not surprising that, by 1950, when the isomeric phthalic acids and esters became available, most resin chemists had an imperfect picture of condensation polymerization on the molecular level; alkyd chemists considered "percent phthalic" and "oil length" as adequate definitions of their formulae.

Much of the theoretical discussion also was based upon definition of the polymers at the point of incipient gelation. This had practical application for solution alkyds, which must be at high molecular weight when the acid number is less than ten, so that the solution viscosity will be adequate and the vehicle will not react with pigments and extenders.<sup>17</sup>

Today, many condensation polymers are employed in high-solids, powder, and water-reducible coatings. The intermediate polymers at the point of application are oligomeric and react further during cure, sometimes with added reactants. Many of the former principles of formulation, perfectly acceptable for long-oil alkyds, now are found to be insufficiently rigorous to define these low molecular weight coatings. Others simply are not helpful for formulation purposes where the ultimate polymers are far from the point of gelation when they are removed from the pot for pigmentation.

Formulation of addition polymers, e.g., reaction of a diisocyanate with a monoglyceride, is relatively straightforward for oligomeric vehicles. When a molecule is eliminated, e.g., water from polyesters, rigorous mathematical treatment is less obvious, and is the subject of this paper.

### THEORETICAL FORMULATION METHODS

Work of Carothers during the mid-thirties<sup>1</sup> defined the fractional extent of reaction (p) in terms of the average functionality (f) at the critical gel point by the equation:

$$f = 2/f$$
 (1)

Thus, a polyester from equimolar glycol and dibasic acid can react to p = 1, or 100%. When equivalent amounts (OH = COOH) of triol and dibasic acid react, average f = 12/5 = 2.4, and at gelation, p = 2/2.4 = 0.833 $\equiv 83.3\%$ . It is, in fact, impossible to recover 83.3% of the water from this reaction mixture before gelation occurs, as Carothers himself recognized.

It is clear that equation (1) is based upon several assumptions that do not apply to real life: (1) that all polymerization reactions are intermolecular; (2) that all functions are equally reactive; and (3) that molecular size

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can be determined from the number average molecular weight ( $\overline{Mn}$  = the total weight divided by the number of molecules). In his early work, this theory did not treat reaction mixtures in which one functional group was in excess.

The greatest source of discrepancy between actual practice and Carothers' theory lies in his concept of  $\overline{Mn}$  as the determinant molecular weight. The simplified view that each molecule doubles in MW with each reaction is contrary to observation near the gel point, where the frequent result is high viscosity at less than the theoretical p.

Theoretical treatment of polymer formulation was greatly advanced by Flory, whose works<sup>2,3</sup> are still the standard references for chemists today. He was the first to recognize that infinite networks can form at relatively low p values, and he treated mathematically<sup>4</sup> the critical probability of gelation ( $\alpha_c$ ) in terms of the average functionality of the reactants (f) according to:

$$\alpha_{\rm c} = 1/(f-1) \tag{2}$$

A small proportion of infinite network can get a mixture which still contains many molecules, and this verifies the concept that weight average molecular weight  $(\overline{M}w)$ corresponds more closely to viscosity than does  $\overline{M}n$ . Even at the gel point, most of the material is present in polymers of rather low degree of polymerization, and the polydispersity  $(\overline{M}w/\overline{M}n)$  becomes greater as the gel point is approached. Larger species are formed at the expense of smaller species, and the  $\overline{M}n$  of the sol actually decreases after  $\alpha_c$  due to the preferential conversion of large species to gel.<sup>2</sup>

The greatest contribution of Kilb<sup>5</sup> lay in his application of volume concepts to the Flory gel prediction, using the Gaussian expression to determine the distribution of end-to-end distances in a randomly-coiled chain, accounting also for intramolecular condensation. Perhaps the simplest form of his unwieldy polyester equation is:

$$(\mathbf{P}_{\mathbf{a}} \operatorname{gel})^2 = \epsilon / (\mathbf{f} - 1) (1 - \mathbf{k} \mathbf{D} \phi)$$
(3)

where  $\epsilon = \text{equivalent of OH}/\text{equivalent of COOH}$ 

 $P_a = extent of COOH reaction = \epsilon P_b$ 

D = volume of system/volume of polymer

k = calculated from volume and constants (including  $\phi$ ) related to reaction conditions.

The importance of the volume effect (variation in system volume per functional group and increase in cyclization due to dilution) was clearly demonstrated by Bernardo and Bruins,<sup>6</sup> who also verified the Flory equations and cautioned against continued use of Carothers' equation.

None of the above-mentioned theorists have had the impact on polyester formulation that resulted from the publication of Patton's book in 1962.<sup>7</sup> His numerous examples are easily understood and his primary equation is easily remembered:

$$\mathbf{K} = \mathbf{M}_{o} / \mathbf{E}_{a} \tag{4}$$

where  $M_o = \text{total moles of reactants}$  $E_a = \text{total COOH equivalents}$  For successful formulation of solution alkyds,  $K \approx 1$ : that is, the necessary high molecular weight at acid numbers less than 10 will be achieved only when K is close to unity.

A second basic equation used by Patton and others is:

$$\mathbf{R} = \mathbf{E}_{\mathbf{b}} / \mathbf{E}_{\mathbf{a}} \tag{5}$$

where  $E_b = \text{total OH equivalents}$ 

E<sub>a</sub> = total COOH equivalents

The first worker to publish the computerization of polyester formulation was Finney.<sup>8</sup> Beginning with equation (4) and equation (5) plus the unit formula concepts of Earhart,<sup>9</sup> he developed a program which calculates all of the alkyd parameters, including cost.

The prime application for Finney's program is in saturated oil-free alkyds, or polyesters, which later are cured with melamines. These are formulated with excess hydroxyl and with  $\overline{Mn} = 1,500$  to 2,500 from triols, glycols, and dibasic acids. Patton's constant, K, is adjusted by any of these parameters during program execution, and becomes unimportant when the coating resin is far from the gel point, which is usual for currently-used oligomers. The equations which determine the starting point for moles of triol and glycol are:

$$Mp = K (E_a) - Ma$$
 (6)

$$At = R (E_a) - 2(Mp)$$
 (7)

where Mp = Moles polyol Ma = Moles acid

Mt = Moles triol

Mg = Moles glycolE<sub>a</sub>, K, R = (See above)

Mg =

These are rigorous solutions for the values, but the independent variables K, R,  $E_a$  are overridden during execution to satisfy other requirements. This computer program has been used by many polyester chemists, especially in the coil-coating area.

All of the methods discussed above, except the last, attempt to discover a formulation that is just short of gelation. All calculations must be based on  $\overline{M}n$  because  $\overline{M}w$  cannot be determined except by experiment and, moreover, varies considerably as the functionality and reactivity of the ingredients vary.

An example of reaction variation due to ingredients is glycerol plus phthalic anhydride (PA) compared with glycerol plus isophthalic acid (IPA). When PA is heated with glycerol, all of the PA molecules react to the extent of p = 0.5 as soon as the PA fuses and before the second COOH on any PA molecule reacts, regardless of excess OH. The resultant homogeneous mixture may be expected to react in a perfectly statistical manner. Contrast this with IPA, where greater than 83% of the water of esterification is recovered before the last of the IPA dissolves.<sup>10</sup> Of the IPA molecules, 83% have reacted completely before the remaining 17% are even available for the first esterification. IPA does not form cyclic intramolecular esters as PA does, and we would expect the Mw and polydispersity of IPA polyesters to be even greater if this natural "withholding" of IPA molecules did not exist.

Chemists who have been involved with the silicone modification of alkyds and polyesters have always pictured polymer formulation on the molecular level. Silicone intermediates are polyfunctional and are used in amounts greater than 25 weight percent so that the resulting vehicle will manifest weatherability or thermal resistance. Their equivalent weights are greater than 190. Because the silicones are initially incompatible with polyesters, they usually must be copolymerized thermally and they contribute crosslinking to the vehicle. Therefore, the polyesters are formulated at  $\overline{Mn} = 900$  to 1,500 for 50% silicone and at 1,250 to 2,000 for 30% silicone: they are oligomeric when compared with long-oil solution alkyds.<sup>11</sup>

Melamine compounds such as hexamethoxymethyl melamine, with theoretical equivalent weights of 65-115, are initially compatible with polyesters and are employed mainly as curing agents for polyesters of calculated  $\overline{Mn} = 2,000$  to 3,500. According to coatings experts, however, high-solids vehicles may be the preponderant form of the future, and their  $\overline{Mn}$  is obviously low in comparison.

### COMPUTATION BASIS

With the realization that the ratio  $K = M_o/E_a$  is no longer critical for oligomeric polyester coating vehicles, a system of formulation was developed based on calculated  $\overline{Mn}$  and  $EW_{OH}$  (equivalent weight/excess OH).

### Number Average Molecular Weight (Mn)

Patton developed three expressions<sup>12</sup> for computing an average molecular weight based upon:

$$\overline{\mathbf{M}}_{av} = \mathbf{W} / \mathbf{M}_{AN} \tag{9}$$

where  $M_{av} = Calculated \overline{M}n$ 

W = Weight

AN = mgm. KOH required to neutralize one gram of sample  $M_{AN} = Polymer$  moles at acid number AN

In no case, however, is W defined rigorously; it is stated to be the "total weight of the alkyd composition" but this weight changes as the reaction proceeds from starting weight (Wo) through weight at a given acid number  $(W_{AN})$  to yield weight (Y) at AN = 0.

Another expression is

$$\mathbf{b} = (\mathbf{A}\mathbf{N}_{o} - \mathbf{A}\mathbf{N}) / \mathbf{A}\mathbf{N}_{o}$$
(10)

where p = fractional extent of reaction AN<sub>o</sub> = acid number at start AN = acid number at reaction p

I

This expression is also inexact, since the sample on which AN is measured represents a larger proportion of the changing weight as the reaction proceeds. For longoil alkyds, where the ratio  $Y/W_o$  is greater than 0.93 (i.e., less than 7% of the initial weight is removed as H<sub>2</sub>O during reaction), the error is minimal, as shown in *Figure* 1. For a zero-oil baking polyester or for an unsaturated polyester, this error is increased to 12-15% at p = 0.9. Consequently, rigorous expressions were derived for use with oligomeric polyesters, and especially those that are used at acid numbers greater than 30:

Let E<sub>a</sub> = total COOH equivalents charged

AN	= acid number at reaction p
$\mathbf{E}_{\mathbf{A}\mathbf{N}}$	= number of COOH equivalents unreacted at acid

$$W = batch weight$$

$$W_{AN} = polymer weight at AN$$

= fractional extent of reaction (OH  $\geq$  COOH).

p is defined as:

$$p = (E_a - E_{AN})/E_a$$
 (11)

The neutral equivalent (NE) at reaction p (acid number = AN) is the weight of polymer per unreacted  $E_{AN}$ and is further defined in terms of AN:

$$NE = W_{AN} / E_{AN} = 56109 / AN$$
 (12)

The number of unreacted COOH times the polymer weight per unreacted COOH equals the weight at a certain p (or certain AN), which also equals the batch weight minus the water recovered:

$$E_{AN} (NE) = W_o - 18 (E_a - M_{ANH} - E_{AN})$$
 (13)

where the total  $H_2O(18E_a)$  is reduced by the number of unreacted COOH ( $E_{AN}$ ) and the moles of anhydride charged ( $M_{ANH}$ ). Expanding and rearranging,

$$E_{AN} (NE) - 18 E_{AN} = W_o - 18 E_a + 18 M_{ANH}$$
 (14)

Now the right-hand side of equation (14) is equal to the yield (Y) at zero acid number.

$$E_{AN} (NE) - 18 E_{AN} = Y$$
  
 $E_{AN} = Y/(NE - 18)$  (15)

Equation (15) is a simple yet rigorous determination of the unreacted COOH. Yield Y is easily determined by assuming complete reaction, and NE is calculated from AN by equation (12). Expressed in terms of AN by use of equation (12), equation (15) becomes:

$$E_{AN} = Y (AN)/(56109 - 18[AN])$$
 (16)

and the weight of polymer at reaction p equals Y plus the water not yet produced:

$$W_{AN} = Y + 18 (E_{AN})$$
 (17)

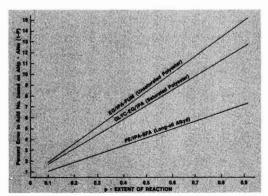


Figure 1—Variation in error for various polyesters through the use of equation (10)

The weight is now defined for any stage of reaction. Substituting equation (16) for  $E_{AN}$  in equation (17), the expression for weight at any stage in terms of AN is obtained:

$$W_{AN} = 56109(Y)/(56109 - 18[AN])$$
 (18)

which may then be used to calculate the Mn at any acid number, according to equation (9).

While the *ratio* of  $M_o/E_a$  is familiar in Patton's alkyd constant K, the *difference* between these values has not

been emphasized sufficiently. In the use of K for formulating solution alkyds,  $M_o - E_a \cong 0$ . For oligomers, though,  $M_o - E_a \ge 1$ . The expression  $M_o - E_a$  is equal to the number of molecules in the polyester when the reaction is complete, assuming intermolecular reactions only. Since each residual COOH increases the number of molecules by one, we need to adjust the result by the unreacted COOH:

$$\overline{M}n = (Y + 18 E_{AN})/(M_o - E_a + E_{AN})$$
 (19)

POLYESTER N	10. 42			ang dia sa sa s Tabu (tener		
INGREDIENT	MOLES	MOL.WT.	WEIGHT	WT. 2	EQUIV.	TOT.EQ.
TRIMETHYLOL PROPANE	8.032 X	136.00=	1092.3	44.310	24.095	24.095
NEOPENTYL GLYCOL	1.319 X	104.15=	AND A DESCRIPTION OF A	5.572	2.638	24.093
ESTERDIOL-204	0.659 X	204.26=	134.7	5.464	1.319	3.957
PHTHALIC ANHYDRIDE	5.734 X	148.12=		34.455	11.469	Call Conte
ADIPIC ACTD	1.720 X	146.14=	251.4	10.198	3.441	14,909
					al Coloria	saut for s
INGREDIENT (\$)	COST/L8.	COST/YIEL	Contraction of the second s		/100# SOL	THE REPORT OF A DESCRIPTION OF A DESCRIP
TRIMETHYLOL PROPANE		0.662		306	47.4921	
NEOPENTYL GLYCOL	0.2500	0.075		149	5.9724	
ESTERDIOL-204	0.3500	0.103	The second second second second	205	5.8565	
PHTHALIC ANHYDRIDE	0.1200	0.224	7 0.0	443	36.9287	
ADIPIC ACID	0.1800	0.099	8 0.0	197	10.9305	
TOTAL =		1.166	4 0.2	300	107.1803	
BATCH WEIGHT ( GM. )	= 24	65.148	NO.POLY.M	01.6.7.81		= 2.550
an John Provident Provident		HAR P	an stars	the second	The sugar	Contraction of the
WATER WEIGHT ( GM. )		.65.147	AVG.INGRE	D.MOLS./	POLY.MOL.	= 6.834
YIELD ( GM. )	= 23	00.001	NO. POLYAC	ID MOLE/	POLY.MOL.	= 2.91
NO.AVG.MCL.WT. @ A#=0	) = 9	000.000	TOTAL MOL	ES TRIOL	an Paya Ratio	= 8.032
NT. POLYESTER / OH		75.000	TOTAL MOL	ES DIOL	inter gitter Inter distant	= 1.978
NO. XS OH / POLYESTER	MOL.=	5.143	TOTAL MOL	ES TRIAC	ID	= 0.000
ND. XS OH / 100 GM. F	PDT. =	0.571	TOTAL MOL	ES DIACI	D	= 7.45
NO. XS OH / YIELD		13.143	TOTAL MOL	ES MONO-	ACID	= 0.000
CH NO. (MGM.KOH/GM.S/	AMP.) = 3	20.617	PATTON'S	CONSTANT	MO/EQ.A	= 1.17
PCT.XS OH		88.153	AVG. EQ.W	T. ACIDS		= 73.832
AVG. EQ.WT. TRICLS	=	45.333	AVG. EQ.W	T. DIOLS		= 68,760
GLYCOL RATIO =66.67/3	3.33 M.*		ACID RATI	0 =76.92	/23.08 M.	2
						A STATE OF A CASE OF A CAS

Figure 2—Printout for IBM 370 computer polyester formulation program

### CONDENSATION POLYMER FORMULATION

000         76 LBI           001         10 E*           002         03           003         01           004         42 STI           005         00           006         01           007         09           010         04           001         10           007         09           008         42 STI           009         08           010         04           011         01           012         32 XII           013         73 RC           014         00           015         69 DP           016         04           021         04           021         04           022         10           021         04           022         04           023         98 ADV           024         69 DP           027         02           028         98 ADV           031         13           032         22 INV           033         35 FI           034         25 CLF
081         69 DP           082         05         05           083         69 DP           084         00         00           085         25         CERN           086         02         RTN           087         00         0           0889         07         7           090         42         STD           091         03         3           092         01         1           093         03         3           095         00         00           095         00         00           096         92         RTN           097         76         LBL           100         10         10           101         10         10           102         61         GTD           103         01         11           104         10         10           105         76         LBL           106         13         43           107         55         #           108         43         RCL           109         12         12
140       12       12         161       43       RCL         162       05       05         163       42       STD         164       11       11         165       43       RCL         166       07       07         165       43       RCL         166       07       07         165       44       RCL         165       06       06         169       25       CLR         171       92       RTN         172       76       LRL         173       11       75         174       32       XIT         175       04       4         177       32       XIT         178       77       GE         179       01       10         180       82       81         181       22       INY         182       86       STD         183       01       01         184       42       STD         185       07       07         186       02       02         187 </td
240       32 XIT         241       30         242       32 XIT         243       67       E0         244       62       22 XIT         243       67       E0         244       62       22 XIT         248       64       64         248       64       64         248       64       04         249       42       STD         251       43       RCL         252       11       11         253       42       STD         254       05       05         255       43       RCL         256       03       03         257       42       STD         258       03       03         263       43       RCL         264       26       26         265       75       -         276       43       RCL         267       95       =         271       95       =         272       35       1/X         273       65          274       53       (
$\begin{array}{cccccccccccccccccccccccccccccccccccc$
12 12 12 12 12 12 12 12 12 12 12 12 12 1
480 86 STF 481 04 0HV 483 58 FI2 486 17 17 487 475 CHS 486 17 CHS 486 17 CHS 489 69 DP 486 17 CHS 489 69 DP 491 71 SBR 492 00 90 493 92 92 92 494 43 RCLL 495 69 D 00 495 69 D 00 495 20 20 495 69 D 00 495 20 20 495 69 D 00 500 79 RADV 500 98 RTL 500 98 RTL 500 79 RADV 500 98 RTL 500 99 RTL 500 98 RTL 500 90 P 500 98 RTL 500 98 RTL 500 80 R

Figure 3—TI-59 program ESTR

In terms of acid number [combining equation (18) and equation (19)]:

$$\overline{Mn} - \frac{56109 (Y)}{(M_o - E_e)[56109 - 18(AN)] + Y(AN)}$$
(20)

### Equivalent Weight of Polyester Per OH (EW OH )

Earhart makes an eloquent plea<sup>9,13</sup> for the use of the unit formula concept, in which all starting and residual functional groups (e.g., COOH and OH) are expressed in terms of equivalents per 100 pbw of completed polymer. Surely, this is a matter of personal preference. This writer can picture a change in  $EW_{OH}$  from 700 to 900 more easily than a small (and inexact) value of 0.143 to 0.111 carbinol per 100 parts of esterified polyester. In addition, formula-

ESTR-INGREDIENT	REGISTERS
331700.13624 373033.13413	31 32
373017.12053	33
222745.09203	34
172200.06202	35 36
332200.07602 313322.10402	30
152316.14422	38
171603.20432	39
373016.14622	40
362113.280411	41
271341.200011	
143146.122111	
243313.166122 331300.148121	44 45
131633.146122	46
134627.188722	47
301300.098021	48
373013.192032	49
PRINT REGIS	TERS
7530322740.	20
2020202020.	21
6430404340. 7515232240.	22 23
6417433223.	23
6420230332.	25
56109.	26
7513403140.	27 28
7544363223.	28 29
7515323223.	30

Figure 4—Program ESTR: Contents of storage registers

tion by the unit method involves simultaneous equations which change in number of terms for each additional ingredient, and this makes the reduction of calculations to computer programs more difficult.

As with  $W_{AN}$ , the equivalent weight per excess OH (EW<sub>OH</sub>) also changes as esterification proceeds. Many water-reducible polyesters are formulated to have infinite EW<sub>OH</sub> and  $\overline{M}n$  at zero acid number. However, they are never allowed to react this far, and the finished polyester has  $\overline{M}n$  and EW<sub>OH</sub> values that are comparable to those values in finished solution alkyds and polyesters. It is necessary to be assured that a given formula will have adequate excess OH for reaction with melamine coreactants or silicone intermediates, adequate COOH to assure water-dispersibility as the amine salt, and  $\overline{M}n$  of optimum value for film-forming and curing rate. The hydroxyl equivalent weight at any stage of reaction may be determined by the quotient of weight at reaction p divided by the number of unreacted carbinol groups:

$$\mathbf{E}\mathbf{W}_{\mathrm{OH}} = \mathbf{W}_{\mathrm{AN}} / (\mathbf{E}_{\mathrm{b}} - \mathbf{E}_{\mathrm{a}} + \mathbf{E}_{\mathrm{AN}}) \tag{21}$$

 $E_b$  = equivalents of OH charged initially (other variables defined above). Although equation (21) is more easily evaluated, EW<sub>OH</sub> can also be expressed in terms of AN [from equations (16) and (21)].

$$EW_{OH} = \frac{56109(Y)}{(E_b - E_a)[56109 - 18(AN)] + Y(AN)}$$
(22)

Equations (15), (16), (18), (20), and (22) are considered by the author as essential to the accurate formulation of water-reducible alkyds and polyesters. At acid numbers in the range of 35-60, the yield,  $\overline{Mn}$  and  $EW_{OH}$  vary significantly from the calculated values available heretofore. Now we have the tools necessary to develop a rigorous method of formulation without recourse to K and R values. The only input need be the ingredient parameters plus desired  $\overline{Mn}$ ,  $EW_{OH}$ , and Y.

### POLYESTER FORMULATION BASIS

An iterative solution to formulation was written for the IBM-370 Computer using PL-I language. The following is only indicative of the method used, and the complete program is available from the writer.

Y = calculated yield
NUMXSOH = total excess carbinol equivalents (OH)
NUMMOLES = number of molecules at AN = 0
TOTOH = number of OH charged
TMB = total moles of polylol
MOLTRIOL = total moles of triol
MOLDIOL = total moles of diol
TEQA = total equivalents of COOH charged,
(Input)
EWOH = weight of polyester per OH (Input)
MOLWT = Mn desired (Input)
YIELD = yield desired (Input)
TMDI = total moles of dibasic acid charged
(Input)
TMTRI = total moles of tribasic acid charged

(Input) = total moles of tribasic acid charged

The starting equations for formulation are presented without derivation:

```
NUMXSOH = YIELD/EWOH
NUMMOLES = YIELD/MOLWT
TMB = TMDI + 2(TMTRI) + NUMMOLES
TOTOH = NUMXSOH + TEQA
MOLTRIOL = TOTOH - 2(TMB)
MOLDIOL = TMB - MOLTRIOL
```

The ingredient input has already defined a starting point for TEQA and the molar ratio of acids. The first results must always be modified. First the yield is adjusted by scaling each acid by the factor YIELD/Y. When this ratio  $\approx 1$ , the program next varies MOLTRIOL and MOLDIOL by similar means to achieve the EW<sub>OH</sub> and Mn wanted and then recycles to recalculate Y. Fewer than 20 iterations are required at each stage for most formulations. If a solution is impossible, the program varies Mn arbitrarily to calculate the correct solution for EW<sub>OH</sub> and YIELD.

As is true with Finney's prior work, this program does not accommodate tetrols. A typical output is displayed as *Figure* 2, where the only inputs were: ingredient parameters, molar ratios for acids and for diols, YIELD = 2,300, EW<sub>OH</sub> = 175, and  $\overline{Mn}$  = 900.

The hand-held programmable calculator has placed such formulation aids at the disposal of every resin chemist. Using the principles from the section on computation basis, a program (ESTR, Figure 3) has been written for the Texas Instruments TI-59 calculator with printer. The program quickly calculates p, R, K, Mn, EWOH, ingredient weights, and true yield for large numbers of possible formulations at any given AN. This calculation accepts input of weights, moles, or equivalents of up to seven ingredients for each resin. Nineteen ingredients and their parameters are permanently stored on a magnetic card for input to the calculator memory registers, and are called out by a code number corresponding to the storage register (Figure 4). Note that each ingredient register contains four values: (1) ingredient name (integer part), (2) formula weight to one decimal place, (3) functional groups per mole, and (4) moles of water produced per mole of acid or anhydride. The values (2), (3), and (4) are concatenated to form the fractional part of the storage register value and are extracted sequentially by the program. Registers 31-49 are loaded once (other ingredients may be substituted) by storing the new ingredient name, followed by summing the fractional part. A list of ingredients may be obtained by pressing E'.

One use of the program is illustrated as Figure 5. We want to make a water-dispersible polyester from trimethylolethane, cyclohexane dimethanol, isophthalic acid, and adipic acid. After trying several other formulae, the first-stage resin in Figure 5-1 is postulated and the calculations are completed to show the water and yield. Then adipic acid is added for a second-stage cook as shown in Figure 5-2. Note that if cooked to AN = 0, the Mn and EW<sub>OH</sub> are both infinite. If the reaction is stopped at AN = 60 (Figure 5-3), the Mn and EW<sub>OH</sub> values are both tractable and the resin can be tried. Recalculation of yield (Figure 5-4) and batch weight (Figure 5-5) are shown.

Use of the keys is shown in Figure 6.

### SILICONE CALCULATIONS

### **Principles**

The use of silicone intermediates with carbinolfunctional organic resins has been a source of frustration for some formulators. Patton states: "The discrepancy

"MOL.	
33. 2.	TME
2.	MOL.
38.	CHD
5.	MOL.
44.	IPA
4.	MQL.
46.	ADP
2.	MOL.
"ĊHG.	
241.00 721.00	TME
664.40	IPA
292.20	ADP
1918.60	
-216.00	-H20
1702.60	esti Yangal
1702.60	M. W.
425.65	EWOH
4.00	VOUL
1.3333	R K

Figure 5-1—Program ESTR first-stage ingredient input and initial printout

Figure 5-2—Program	n ESTR second-	Figure 5-3—Program	ESTR second-	Figure 5-4—Program	n ESTR recal-
1.0000 0.9375	R K	1.1508 1.0789	R K	1.1508 1.0789	r K
		0.8690	P	0.8690	p in
INF. INF. 0.00	=M.W. =EWDH XSDH	10.6935 10.6935	XSDH CODH	2.0965 2.0965	XSOH COOH
1922.80	- Y	935.15 60.00	EWOH A. N.	935.15 60.00	EWDH A. N.
2210.80	CHG. -H20	1788.00	M. W.	1788.00	M. W.
292.20	AIP	10000.00	Y	1960.54	Ťγ
292.20	ADP	-1276.50	-H20	2210.80 -250.26	Section of the sectio
721.00	CHD IPA	11276.50	CHG.		
241.00	TME	1490.41 1490.41	ADP ADP	292.20 292.20	ADP
		3677.56 3388.87	CHD IPA	721.00	CHD
2.	MOL.	1229.26	TME	241.00	TME
46.	RDP				lin eines

Figure 5-3—Program ESTR secondstage results at acid number=60 (Note that all parameters change)

Figure 5-4—Program ESTR recalculation for yield = 10,000 parts

between the theoretical and effective functionality of the silicone intermediates is so extreme that application of conventional alkyd formulating principles to them appears impossible" (Reference (7), p. 27). This is an accurate statement which also applies to most other polymeric and polyfunctional resin modifiers. A polyester chemist would never try to apply theoretical formulation principles to hexamethoxymethylmelamine (HMMM) curing agents or oligomeric butylated melamine-formaldehyde resins; instead, he assures that there is sufficient excess carbinol in the polyester to react with some of the functional groups (HMMM reacts to about 3 to 3.5 methylol groups of its theoretical 6 groups).

stage formula at acid number = 0

Silicones should be treated similarly; the intermediates should be copolymerized to the stage of compatibility with the organic resin plus additional cooking to the desired viscosity, but there should be no attempt to treat the silicones as tribasic or tetrabasic acids in alkyd and polyester formulations.

The reasons for this philosophy can be understood by reference to the nature of the silicones. To date, all commercial intermediates are silanol-functional or methoxy-functional oligomers with  $\overline{Mn} = 800 - 1,200$ . The methoxy silicones react by alcoholysis with excess carbinol in the polyester/alkyd/polyacrylate/polyurethane.

≡Si-OMe + HO-CH<sub>2</sub>-RESIN -

 $\equiv$  Si-O-CH<sub>2</sub>-RESIN + MeOH

(Reaction 1)

Although some recent intermediates contribute rigidity to the vehicles, most are soft, flexible silicones which impart flexibility to the copolymers. These usually are used as weatherable coating vehicles for pre-finished metal in the building industry. The product of Reaction 1 contains only Si-O-C linkages between the silicone oligomer and the organic resin. This linkage is subject to hydrolysis and a few of the Si-O-C moieties undoubtedly hydrolyze with time.

Degradation is minimal in the well-cured coatings, as evidenced by outdoor durability.<sup>14</sup> It is believed that hydrolysis is reversible due to the effect of energy from sunlight (Reference (14), p. 561). Nevertheless, until such reversible reactions can restore the original crosslink density of the coating, it is necessary that the organic portion be able to stand on its own feet; the Mn of the organic portion should be great enough that erosion does not occur during periods of damp, dark weather. Then, the silicone can re-establish its protection of the vehicle at as little as 25 weight percent of the vehicle solids.

If the silicones are treated as reactants, on the other hand, the organic vehicle will be formulated to a low  $\overline{Mn}$ to forestall gelation during processing. In this event, the organic portion may be unable to withstand weathering and the coating is more subject to change (Reference (14), p. 557).

Rigid silanol-functional intermediates usually are employed in long-oil alkyd-silicone maintenance coatings.

"CHG.	
327.03 978.38 901.57 396.51 396.51	IPA
3000.00	снс.
-339.60	-н2D
2660.40	-ч
1788,00	M.W.
935,15	Ewoh
60,00	A.N,
2.8449	XSOH
2.8449	CODH
0.8690	P.
1.1508	R
1.0789	K

Figure 5-5—Program ESTR recalculation for batch = 3,000 parts

While the alkyd must be formulated to moderate  $\overline{M}n$  in order to provide sufficient excess carbinol for reaction with the silicone, the overall vehicle crosslink density is restored during copolymerization.

In general, silanol-functional intermediates provide additional rigidity and viscosity to the silicone-alkyd vehicle. They combine via the reaction:

$$= SiO-CH_2 - RESIN \longrightarrow$$
  
= SiO-CH\_2 - RESIN + H<sub>2</sub>O (Reaction 2)

which is in competition with the reaction:

$$\equiv \text{Si-OH} + \text{HO-Si} \equiv \longrightarrow$$
  
$$\equiv \text{Si-O-Si} \equiv + \text{H}_2 \text{O} \qquad (Reaction 3)$$

Some of the silicone functionality inevitably will condense according to Reaction 3, and the occurrence of Reaction 3 is the basis for Mr. Patton's remark cited previously (Reference (7), p. 27). The quantitative ratio Reaction 2/Reaction 3 is discussed more thoroughly in Reference (14).

### **Weight Calculations**

The silicones can vary according to the type of functionality, e.g., methoxy, silanol, ethoxy, etc., and the weight percent of silicone functionality. The entire range of silicone functionality is accommodated by the following equation, which establishes the amount of silicone needed to meet the U.S. Specifications TTE-490B, TTE-001593, and MIL-E-46141. (Although alcoholysis reactions are allowed, fatty acid esterification is preferred because this process was investigated more thoroughly by the silicone manufacturers who (largely) established the specifications.)

Wt. Sil. =

	(%	6 sil) (Wt. Org. Resin)
100 - %	sil. + (%F/EW	F)[%Sil(0.01)(EWF + 1) - EWF + 8] (23)
Where:	Wt. Sil.	= Weight of silicone intermediate which will provide a certain % silicone in the finished copolymer vehicle.
	% Sil.	= Weight percent polysiloxane specified for the copolymer solids.
	Wt. Org. Resin	= Weight of organic resin solids.
	%F	= Weight percent of functional group in sili- cone intermediate.
	EWF	= Equivalent weight of each silicone func- tional group.
		= 17 for silanol.
		= 31 for methoxysilane, etc.

Equation (23) has been translated to a TI-59<sup>®</sup> program (SILESTR; see *Figure* 7 for listing, *Figure* 8 for

Equation (23) is derived from	Reference (14) n 550	Equation 13-1 and 13-2
Equation (25) is derived from	Reference (14), p. 550	, Equation 13-1 and 13-2.

	ialization for each run. Clears ingredient lis n previous run.
A = Ent	er Ingredient Code Number (Notes 1, 4)
C = Ent	er Weight er Equivalents er Moles One only (Note 1)
cal A' = Ent B' = Ent C' = En (Nc	it stage calculation or copy of last previous culation er Acid Number and Calculate (Note 2) er New Yield and Calculate (Note 3) ter New charge Weight and Calculate te 3)
	t of Stored Ingredients and their Code nbers
Note 1:	After an ingredient code number is entered and (A) is pressed, wait for the printout and then enter the ingredient weight, equiva- lents or moles and press (B), (C) or (D) respectively. All inputs are converted to moles for printing.
Note 2:	Acid number may be entered and (A' pressed, immediately after ingredients have been entered or after (D') calculations
Note 3:	(D') or (A') must have been pressed pre- viously. Used mostly for final charge o yield adjustment, with acid number at any level.
Note 4:	New ingredients may be entered (as in a two-stage cook) or additional amounts of a previous ingredient may be entered (to adjust M.W. or $EW_{OH}$ ), up to seven tota entries. Before adding ingredients, restore acid number to zero by pressing (A') with zero in the display.

Figure 6—Use of keys for program ESTR

0000 76 LBL 002 03 3 003 01 10 E' 002 03 3 003 01 1 005 07 07 006 01 7 007 09 9 008 42 STD 009 004 4 011 01 1 013 69 DP 014 00 04 4 011 01 22 X:T 013 69 DP 014 00 4 015 73 RC* 016 07 07 017 69 JP 018 04 019 43 RCL 022 67 E9 023 06 06 06 025 98 ADV 022 67 E9 023 02 67 E9 023 00 00 033 15 15 034 22 INV 035 55 CLR 037 98 ADV 041 95 15 036 55 21 ENV 039 98 ADV 041 97 DSZ 031 00 00 033 15 15 034 25 CLR 037 98 ADV 042 67 E9 044 67 E9 044 67 E9 044 67 E9 044 53 53 045 952 INV 047 777 GE 048 00 54 54 048 05 69 DP 051 06 06 052 92 STLR 048 05 69 DP 051 06 07 7 048 05 57 INV 048 05 07 F 053 055 29 CRN 054 43 RCL 055 05 S INV 056 57 INV 057 061 057 + 066 57 S INV 058 43 RCL 059 95 S NTT 057 061 01 0 077 43 RCL 068 07 5 = 074 92 RTN 075 69 DP
080         02         02           081         69         0P           082         03         03           083         69         0P           084         05         05           085         69         0P           088         76         LBL           090         55         +           091         43         RCL           092         11         11           093         61         GTD           094         01         01           095         76         LBL           097         12         B           098         63         RCL           1097         12         B           098         55         +           099         43         RCL           100         12         12           104         42         STD           105         07         07           106         44         SUN           107         04         04           108         49         PRD           109         10         11           111
160         32 X:T           161         77 GE           162         01 01           163         65 65           164         22 INV           165         86 STF           166         01 01           167         42 STD           168         07 07           169         02 2:           170         00 0           171         32 X:T           173         00 00           174         67 E9           175         02 23           176         23 23           177         43 RCL           178         07 07           183         69 DP           184         69 DP           185         04 04           186         95 ×           188         06 6 N           199         52 EE           191         52 EE           192         22 ENV           193         52 EE           194         95 \$7           195         59 59           201         10 10           202         71 SBR           203         00 90
240 60 60 241 42 ST0 242 12 12 243 73 RC# 244 12 12 245 69 DF 246 04 247 43 RCL 249 22 INV 250 59 INT 251 35 17X 252 69 DF 255 02 02 256 97 DSZ 259 36 36 264 69 DF 255 02 02 256 97 DSZ 259 36 36 261 75 RC 263 43 RCL 263 43 RCL 263 69 DF 270 85 RCL 263 69 DF 270 85 RCL 263 43 RCL 263 06 06 269 DF 270 85 RCL 263 43 RCL 263 06 06 269 DF 270 85 RCL 263 43 RCL 263 06 06 269 DF 270 85 RCL 277 85 CC 277 85 CC 278 85 CC 277 85 CC 278 85 CC 277 85 CC 278 85 CC 277 85 CC 278 85 CC 277 85 CC 278 85 CC 277 85 CC 278 85 CC 278 85 CC 277 85 CC 278 85 CC 277 85 CC 278 85 CC
$\begin{array}{cccccccccccccccccccccccccccccccccccc$
480         06         06           481         61         CTO           482         00         00           483         34         34           485         16         CTO           483         34         34           485         16         CTO           487         10         NCL           487         10         NCL           487         10         NCL           487         10         OC           489         27         27           490         69         OP           491         04         04           492         43         RCL           493         10         10           493         10         10           493         10         11           501         06         6           502         01         1           503         02         2           504         01         1           505         69         04           506         04         04           507         43         RCL           508 <t< td=""></t<>

Figure 7—TI-59 program SILESTR

storage registers and use of keys) which uses the same inputs (excepting AN) as the ESTR-program, plus EWF, %F, and Wt. % silicone to calculate rigorously the amount of silicone intermediate needed to meet the above Federal Specifications. There are 19 organic ingredients stored by this program, and seven of these may be used in a single formation.

An example of the execution of SILESTR is in Figure 9. We want to make a 30% silicone-alkyd from pentaerythritol, phthalic anhydride, sova fatty acids, and a flaked, silanol-functional silicone intermediate which has 5.6 weight percent OH (=%F). The equivalent weight of the functional group (= EWF) is 17 in this case. For long-oil alkyd copolymers, excess carbinol just equivalent to the silanol EW should be provided. The first attempt (Figure 9-1) was insufficient in this respect (see the OH/F (carbinol/silicone functionality ratio) near the bottom of the printout). Adjustment of the organic resin (Figure 9-2) provided OH/F = 0.5095, a sufficient value. (Note: an equivalent weight of 600 (see section on stoichiometry) requires only an OH/F ratio of 0.506.) The second CHG notation is the weight of polyester plus silicone solids before reaction and the VOLS notation is the weight of volatiles, water in this case, liberated by the copolymerization.

SILESTR can be used for any other functional modifier or curing agent, such as HMMM. This monomeric methyl ether of melamine-formaldehyde has MW = 390. If 3.5 methylol groups per mole typically are available for reaction, the equivalent weight is 111.43 parts/MeO; the functional equivalent weight (EWF) = 31 (methoxy); the weight percent of function becomes (100)(31)/ 111.43 = 27.82% MeO. The high-solids polyester oligomer illustrated in *Figure* 9-3 was shown to have sufficient excess carbinol to accommodate at least 20 weight percent of HMMM (weight listed as SIL; TMD = trimethylpentanediol; AZL = azelaic acid; 16.9 residue percent (listed of HMMM is equal to 20 weight percent of monomer).

### **Polyester/Silicone Stoichiometry**

If the silicone intermediates are not to appear in the polyester calculations, the question remains as to the optimum COH/SiOX ratio to give the best responses, e.g., weatherability. It has been stated above that the COH/SiOH need not exceed unity for long-oil alkyds cooked with silanol-functional intermediates. Although these intermediates as supplied contain approximately six weight percent OH as SiOH (combining weight = 283), a practical combining weight of 600 may be used when the solubility parameters of the organic resin and silicone are similar, as in long-oil alkyds. For other resins, such as epoxy, an equivalent weight of 400 should be employed. Enough excess carbinol must be provided to accommodate these silicone EW-one excess COH in 1400 parts of long-oil alkyd or 900 parts of less compatible resin at 30% silicone, when copolymerized in solution.

For methoxy-functional intermediates of the flexible, low crosslink density type used in coil-coating vehicles, the COH/Si-OMe ratio will vary according to percent

	1.1	PROGRAM SILE	STR
		PRINT STORAGE	REGISTERS
		6436242740. 2020202020. 6430404340. 7515232240. 6417433223. 64432236321. 6400174321. 6461362427. 2431214000. 7530322740.	20 21 22 23 24 25 26 27 28 29 30
USE	<u>OF</u> K	<u>EYS</u> (CF. EXHIBIT 6)	
E	=	INITIALIZATION	
E A	1 4 2 2 2 1 2 2 2 1 2 2 2 1 2 2 2 2 2 2	INITIALIZATION ENTER INGREDIENT CO	de Number
			de number
A B C	н н н	ENTER INGREDIENT CO ENTER WEIGHT ENTER EQUIVALENTS ENTER MOLES	DE NUMBER WT. OF FUNCTIONAL GROUP
A B C D		ENTER INGREDIENT CO ENTER WEIGHT ENTER EQUIVALENTS ENTER MOLES ENTER SILICONE EQ. 17 FOR OH 31 FOR MEO etc. ENTER WT.% OF FUNCT	WT. OF FUNCTIONAL GROUP

Figure 8—Program SILESTR: Storage register and keys

silicone. Earhart<sup>13</sup> suggested ratios that appear to be lower than optimum for coil-coating resins, where the cure must be accomplished in less than 90 seconds. Undercured silicone-polyesters suffer from lack of flexibility and have a tendency to check. A certain excess carbinol, beyond the stoichiometric amount required for reaction with silicone and melamine, provides durability and improves flexibility.

Some workers have shown that excess carbinol does not bear the deleterious effect on durability<sup>15,16</sup> that the alkyd tradition implies. A polyester varnish (for bonding glass fiber insulation to wire) made from glycerol/ isophthalic acid in molar ratio 16/13 (84.6% excess OH), when modified with 10 weight percent of a monophenyl

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### L.H. BROWN

		«MOL.
"MOL.	«MDL.	32. TMP 0.75 MDL.
31. PE 10. MOL.	31. PE 10. MOL.	40. TMD 3.25 MDL.
45. PA 9. MDL.	45. PA 9. MDL.	44. IPA 1.85 MDL.
41. SFA 18. MDL.	41. SFR 16.9 MOL.	47. AZL 1.15 MDL.
17. EWF 5.6 %F 30. %SIL	17. EWF 5.6 %F 30. %SIL	31. EWF 27.82 %F 16.9 %SIL
	¢HG.	℃HG <b>.</b>
1372.00 PE 1332.00 PA 5040.00 SFA	1372.00 PE 1332.00 PA 4732.00 SFA	102.15 TMP 475.15 TMD 307.10 IPA 217.01 AZL
7744.00 CHG. -486.00 VOLS	7436.00 CHG. -466.20 VDLS	1101.41 CHG.
7258.00 Y 3164.18 SIL.	6969.80 Y 3038.53 SIL.	-108.00 YDLS 993.41 Y
10422,18 CHG. -187.62 VDLS	10008.33 CHG. -180.17 VDLS	249.41 SIL. 1242.82 CHG. -71.62 VOLS
10234.56 Y	9828.17 Y	-71.62 VOLS
7258.00 M.W. INF. =EWDH	6969.80 M.W. INF. ≃EWDH	993.41 M.W. 1941.26 EWDH
0.3838 DH/F 1.1111 R 1.0278 K	0.5095 DH/F 1.1461 R 1.0287 K	1.2286 DH/F 1.4583 R 1.1667 K
Figure 9-1—Program SILESTR un- successful formula for 30% silicone- alkyd	Figure 9-2—Program SILESTR modified alkyd formula for 30% silicone	Figure 9-3—Program SILESTR cal- culation of batch weights and stoichiometry for 20 wt % curing agent (HMMM)

Figure 9-1—Program SILESTR un-successful formula for 30% siliconealkyd

siloxane intermediate, produced the highest helical-coil bond strength (a measure of adhesion and intermolecular forces) ever observed in these laboratories on aluminum wire (90.2 lb at RT after 6 hr/200°C cure; 85.2 lb at RT after 6 hr/250°C cure, and greater than 50 lb measured at 100°C after the same cure). The initial dielectric strength of 2115 V/mil (where 1 mil = 0.001 in. = 0.254 mm.) decreased only slightly to 1858 V/mil after 24 hr in H<sub>2</sub>O and to 1901 V/mil after 24 hr at 96% relative humidity, showing that the large excess of OH had little, if any, effect on the performance of this varnish.

A list of starting polyester formulae may be found in Reference (14). At 30% silicone, these polyester oligomers have  $\overline{Mn} = 1,250$ . For 50% silicone,  $\overline{Mn} = 925$ . Within the range of silicone content from 25 to 50 weight percent, the COH/SiOMe ratio derived by experimentation can be determined from the expression:

$$COH/SiOMe = (69.6 - \% Sil)/15.7$$
 (24)

These data may be summarized: The lower weight percent silicone requires a greater polyester  $\overline{M}n$  and a greater COH/SiOMe ratio.

Incidentally, Thames, Patel, and Bufkin<sup>18</sup> have learned that the mere presence of a silicon atom in an alkyd ingredient can enhance weatherability. Improved performance was demonstrated when bis-(p-carboxyphenyl) dimethylsilane replaced 15% of the phthalic anhydride in an alkyd paint vehicle. The conclusion is that the Si atom can improve coating properties without the need for large proportions of polysiloxane Si-O-Si structures. This tentative result will be subject to verification.<sup>19</sup>

### SUMMARY

Polyester formulation methods traditionally use equations that are derived from critical gel point considerations. Although probably adequate for the formulation of long-oil alkyds, these become less applicable for oligomeric condensation polymers in use today. In particular, new methods were needed for water-reducible alkyds and polyesters, which are used at elevated acid numbers. Rigorous expressions for unreacted acid groups and weight of polymer at a given acid number were developed and applied to a computer program which uses Mn,  $EW_{OH}$  and yield as independent variables. Two programs for hand-held calculators rapidly assess the effect of formulation changes on these parameters. The equations may be applied to other condensation polymers by substituting the MW of the eliminated molecule for 18 (which represents  $H_2O$ ) in the new expressions. Equally rigorous expressions were developed for modification of polyesters by silicone intermediates.

Finally, it should be said that all formulation methods based on  $\overline{Mn}$  calculations must be verified by experimentation, since practical considerations (such as vehicle viscosity) depend on  $\overline{Mw}$ , which varies greatly with ingredient type and reaction conditions. Once the chemist can correlate calculated  $\overline{Mn}$ , etc., with actual performance, he will be able to use these calculation methods for everyday vehicle formulation. The use of oligomeric vehicles increases the necessity for accurate calculations, to achieve balance between polymer parameters in the current effort to meet stringent environmental regulations.

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## Nondestructive Drying Of Amino Resin Solutions

S.M. Kambanis and J. Rybicky Reichhold Limited\*

Techniques for the removal of solvent from unmodified and etherified urea-formaldehyde resins were investigated. Vacuum drying of 50  $\mu$  thick films at temperatures of approximately 50°C and pressures of about 0.2 kPa for the duration of 1 hr was found to provide good and practical drying conditions. The drying was shown to have no observable effect on the structure of resin. The free formaldehyde content in dried resin was found to be less than 0.006%.

### INTRODUCTION

Due to the unstable nature of amino resins, the solvent removal from a resin sample requires delicate conditions and a prior investigation of the effect of drying on the resin structure. For practical purposes of the manufacturer and the consumer, techniques have been developed which determine the nonvolatile content of amino resins under specified conditions.<sup>1,2</sup> It is generally understood that the nonvolatile content is more or less cured resin, rather than the true solids of a liquid sample.

Several investigations have been carried out with the aim of finding drying conditions which are effective enough to provide, essentially, a free-of-solvent resin and yet mild enough to have no effect on the resin structure by initiating the curing process. These methods were recently reviewed by Christensen.<sup>3</sup> The review indicated that temperature between 45 and 60°C is optimal when heat drying is employed.<sup>1,4-6</sup> The thickness of the original film recommended ranged from 12  $\mu^6$  through 45  $\mu^1$  to a few hundred  $\mu^4$  Both the ambient and reduced pressures were investigated with times of drying ranging from

hours to months.<sup>1,4-8</sup> For water-based amino resins, freeze drying has been investigated.<sup>1,9-11</sup>

Recently, the Supported Polymer Films Working Party of the Macromolecular Division of I.U.P.A.C. launched a project for the investigation of analytical techniques for the determination of functional groups in amino resins.3 A part of the project was the removal of solvent from liquid amino resins. The authors undertook the role of investigating drving techniques for ureaformaldehyde (UF) resins. Forced-air drying was compared with vacuum drying. The latter was preferred on the basis of reproducibility of results and more definite end-point of drying. The nondestructive character of the method was investigated by the determination of the methylol groups content before and after drying, the formaldehyde content in dried resins, and by analysis of the removed volatile matter. Eight different laboratories employed the method and assessed the repeatability of results.

### EXPERIMENTAL

### Preparation of Samples for Drying

All samples had a pH of 7.0 to 7.5. Glass plates with dimensions of  $15 \times 10$  cm were used. A sample was deposited near the edge of one of the plates by means of a syringe. Immediately afterwards, the area of the plate with the wet sample was covered by the marginal area of another glass plate and then the two plates were slid against each other to completely overlap. Just before drying, the two plates were separated by sliding them apart. In this manner, the films were smeared on the glass plates. The syringe containing resin was weighed before and after sample deposition. The pair of glass plates was weighed before sample deposition and again after drying to determine the progress of drying.

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This work is a part of an interlaboratory project launched by the Working Party on Supported Polymer Films of the Macromolecular Division of 1.U.P.A.C.

Table 1-Effect of	Eilm	Thickness	00	Vacuum	Drving
Table I-Effect of	гиш	I IIICKIIC33	011	* acuum	Dijing

Amount of Resin (g)	Average Solids (%)	Standard Deviation (%)
Nonetherified UF Resin		100110
Around 0.1	63.9	0.3
Around 0.5	64.3	0.1
Around 1.0	64.5	0.1
Methoxylated UF Resin		
Around 0.1	86.6	0.8
Around 0.5	89.0	0.2
Around 1.0	90.0	0.2
Butoxylated UF Resin		
Around 0.1	68.6	0.5
Around 0.5	69.2	0.4
Around 1.0	69.6	0.3

Vacuum Drying

The separated glass plates with deposited wet films were set vertically on a rack and placed in a vacuum oven for 1 hr.

VACUUM OVEN:	Thelco	o Precis	ion Sci	. Model	10,
	wit	h 6.6 litr	es of spa	ce, $50^{\circ} \pm 3$	°C.
VACUUM PUMP:	Two	stages;	Welch	DUO-SE	AL

Model 1402, free air displacement 160 litres/min; ultimate pressure 0.13 kPa. PRESSURE: 0.13 - 0.20 kPa.

### **Tunnel Drying**

The separated plates with samples were placed inside a glass cylinder 13 cm in diameter and 120 cm long. Heated air was forced through the cylinder by means of a heated air-fan at a fixed position. The plates were at least 60 cm away from the tip of the blower, in a horizontal position. More than one sample could be dried at the same time, provided none of the plates were obstructing the flow of air.

### **Collection of Dried Samples**

After drying, the two matching plates were sandwiched again, placed in a dessicator to cool to room temperature, and weighed. When the sample weight was investigated as a function of time of drying, the plates were weighed separately in order to avoid the destruction of the film surface. The dried resin was removed from the glass plates by means of a paint scraper (a razor blade in a holder) and kept in a closed vial for further work, if required. (Static electricity problems, encountered when scraping and collecting the resin, can be eliminated by employing the so-called static gun for grammophone records.)

### Calculation of Solids

Solids (%) = 
$$\frac{W(d)}{W(1)}$$
 100

where W(1) is the weight of liquid sample measured as a difference between two syringe weighings; and W(d) is the weight of dry sample measured as a difference between the weight of plates with dried film and clean plates.

### RESULTS

### **Drying Techniques**

The time dependence studies of the weight loss of wet films (100 $\mu$  or less) indicated that the optimum time and temperature for both vacuum and air drying was I hr and 50°C, respectively. Below this temperature, the time of drying was impractically long and/or the resin was not dried properly.

When the vacuum drying and the air drying techniques were compared, the former was found to yield better reproducibility of results, judging on the basis of standard deviation.

For vacuum drying at  $50^{\circ}$ C and 0.13-0.20 kPa, the time-dependence curve assumed an almost constant slope after a half an hour. The value of the slope was small with only a slight tendency to become zero. It was estimated that 10 hr or more could lead to an almost zerovalue slope. The nuclear magnetic resonance spectroscopy and the carbon/nitrogen analysis were applied to

### Table 2—Solids Found in Different Laboratories (%) 15ª

	Laboratories									
Resin	1	2	3	4	5	6	7	8		
i-But-UF in i-butanol	76.8	77.2	76.6	77.2	77.5	77.8	76.5	76.9		
But-MF in butanol + xylene	61.2	61.5	60.9	62.5	62.3	61.9	61.5	63.6		
Met-MF in water	92.9	92.9	94.0	95.0	90.2	93.4	93.1	93.6		

(a) 59°C; 0.13 - 0.20 kPa; 1 hr. Results were provided by the following laboratories: BAM Bundesanstalt für Materialprüfung, Germany; BASF, Germany; DKI, Deutsches Kunststoff-Institute, Germany; Montedison, Italy; NIF, Scandinavian Paint and Printing Ink Research Institute, Denmark; PPG Industries, Inc., U.S.A.; Reichhold Ltd., Canada; Sadolin & Holmblad Ltd., Denmark. samples that had been dried for 1 hr and less. No detectable differences were found in samples dried for about a half an hour and longer. Drying at lower temperatures resulted in poor reproducibilities when compared with those obtained at 50°C.

Based on these preliminary results, the vacuum drving carried at 50°C for 1 hr was chosen as optimum with respect to practicality and performance. This method of drying was investigated further.

### Film Thickness Investigation

For the vacuum drying, the effect of variation of the film thickness on the found solids and on the reproducibility of findings were evaluated. Four or more experiments were performed for each average value. For the sake of simplicity, it was the weight of sample that was controlled rather than the film thickness. Since the area of the film was approximately the same for each experiment (two-thirds of the surface of a pair of glass plates, i.e. 200 cm<sup>2</sup>), the film thickness was proportional to the sample weight and could be calculated, when required. The results are summarized in Table 1.

### Destructiveness of Drying

In order to assess the effect of the vacuum drying method on the possibility of structural changes of the dried resin, the content of methylol groups in a nonetherified resin before and after drying was determined by the hypoiodite method.<sup>12</sup> The content of methylol groups was found to be 24.1 - 24.3% and 24.2 - 24.4% before and after drying, respectively, when expressed per solids.

The nondestructive character of the drying conditions was also supported by the absence of any resin constituents in the volatiles, as determined by the TLC analysis of condensate collected in a liquid nitrogen trap.<sup>13</sup>

### Free Formaldehyde in Dried Resin

The effectiveness of the vacuum drying for the removal of free formaldehyde may become important for various purposes, such as the determination of F/U or F/Mratio in dry solids by elemental analysis.<sup>14</sup> Also. it indicates a destructiveness of the drying when the free formaldehyde content cannot be suppressed to a value close to zero. It was found that nonetherified vacuumdried UF resin contained less than 0.006% free formaldehyde. This value was arrived at by finding that a solution of 1 g of dried resin was overtitrated by one drop (0.02 ml) of deci-normal acid after the addition of neutralized sodium sulphite solution at 0°C. (The content of free formaldehyde in the original solution was cca. 1%.)

### Repeatability and Reproducibility of Results

The vacuum drying technique was used by eight different laboratories on three different resin solutions. Table 2 summarizes the results which represent averages of one to five experiments. The standard deviation calculated from all the single experiments (n = 51) was found to be  $\pm 0.42\%$ . When calculated for separate resins, the standard deviations differed insignificantly (from 0.36 to 0.48%.)<sup>15</sup>

### Table 3-Vacuum-Dried Solids vs **Nonvolatile Content and Loaded Solids**

UF Resin I	Conventional I.V. Found (%) <sup>a</sup>	Solids Found (%)	Total Load (%) <sup>c</sup>
Butoxylated	48-50	64-67	73
Methoxylated	74-78	87-89	100
Nonmodified	60	64	72

(a) 105°C, 2 hr. (b) 50°C, 1 hr, 0.13 - 0.20 kPa.

(c) Calculated as total reactants before condensation

### SUMMARY AND CONCLUSIONS

On the basis of the foregoing findings, it is concluded that the vacuum drying of amino resins at temperature 50°C, pressure 0.13 - 0.20 kPa, for the duration of 1 hr provides nondestructive conditions for the solvent removal from films of resin solutions. The solvent removal is complete after 1 hr for film thickness of 10 to 50  $\mu$ ; this corresponds to 0.1 -0.5 g of resin solution spread on an area of 100 cm<sup>2</sup>.

Smaller quantities of resin might still provide lower solids. It seems impractical, however, to dry smaller amounts of resin, since the objective of drying is usually the isolation of a reasonable amount of resin for further analytical work. Even when the only objective of drving is the determination of the solids content, it is not beneficial to use less sample since the relative error of weighing and, consequently, the standard deviation increase.

Destructiveness of the vacuum drying was investigated by means of the methylol groups determination. It was assumed that the first structural change resulting from a drying that was not mild enough would be the condensation of methylol groups.

$$\sim$$
CONHCH<sub>2</sub>OH + H<sub>2</sub>NCO $\sim$  –

$$\sim$$
CONHCH<sub>2</sub>NHCO $\sim$  + H<sub>2</sub>O

~CONHCH2OH + HOCH2NHCO~ --- $\sim$ CONHCH<sub>2</sub>NHCO $\sim$  + H<sub>2</sub>O + CH<sub>2</sub>O

 $\sim$ CONHCH<sub>2</sub>OH + ROCH<sub>2</sub>NHCO $\sim \rightarrow$ ~CONHCH2NHCO~ + ROH + CH2O

This approach should be far more sensitive to structural changes than the infrared spectroscopy employed in some previous investigations.<sup>4,5</sup> The content of methylol groups before and after vacuum drying at 50°C for 1 hr was found the same within the accuracy of the method. Although the test was done on a nonetherified UF resin, the stability of alkoxy groups relative to that of methylol groups (the former being more stable than the latter under the conditions of drying) allows the application of the result to etherified resins as well.

In many cases, the solids are isolated for analytical purposes and the knowledge of the free formaldehyde content may be necessary. The conditions of the vacuum drying discussed are such that one would expect free formaldehyde to be removed with solvent. This expectation was confirmed when the free formaldehyde content in a vacuum-dried UF resin was found to be below the detection limit of the method employed, i.e., below 0.006%, compared with 1% in the original solution.

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Finally, the vacuum drying as described was applied to unmodified, methoxylated and butoxylated UF resins. The solids content found this way was compared with the conventional nonvolatile content determined at 105°C by the PMMA method<sup>2</sup> and with the total load in the reactor. As indicated in Table 3, the solids found by the vacuum-drying method relate to the conventional nonvolatiles and to the total load according to the expectation. The difference between the total load and the solids found by vacuum drying is due to the water of condensation produced during the manufacture of the resin. The difference between the conventional nonvolatiles and the vacuum-dried solids is the result of the water of condensation generated by the curing process which takes place with the PMMA method,<sup>2</sup> and also by the loss of low molecular weight species which may take place at 105°C.

It may be worth mentioning that, to be dried, a resin solution should be nonacidic. Unmodified resins should have a pH of 7.0 - 7.5. Neutral or slightly alkaline conditions are necessary for prevention of condensation which is otherwise possible under acidic conditions.

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### United Kingdom Orders of FSCT Educational Literature

Mr. Ray Tennant, of the Birmingham Society, will act as a source of Federation educational literature (Color-matching Aptitude Test Set; Infrared Spectroscopy; Paint/Coatings Dictionary) for United Kingdom customers. Anyone interested in receiving these items is urged to contact Mr. Tennant. His address is: Carrs Paints Limited, Westminster Works, Alvechurch Road, Birmingham B31 3PG, England.

# Multifunctional Epoxy Resins Come of Age

Marianne DiBenedetto CIBA-GEIGY Corporation\*

Multifunctional epoxy resins such as epoxy cresol and epoxy phenol novolacs and triglycidyl paraaminophenol have long found utility in the molding, casting, and electrical areas of application. In combination with anhydride or aromatic amine curing agents, these resins were traditionally heat cured to form networks of extremely high crosslink density. This phenomenon was responsible for the high heat deflection temperatures and the excellent chemical resistance properties for which such systems were and still are known.

Today, however, the use of multifunctional resins need not be restricted to applications which require high gelation and post-cure temperatures. Through careful formulation, systems based on multifunctional resins show promise in room temperature cured coating applications. Since they possess the same high crosslink density as their casting and molding counterparts, these room temperature cured coating systems should afford excellence in the areas of chemical resistance, surface hardness, and thermal stability.

Numerous application possibilities for such systems exist in the areas of maintenance and marine coatings and energy transmission service.

### INTRODUCTION

Since their commercialization in the late 1940's, epoxy resins have proven themselves to be one of the most versatile and useful polymers in their field. Application areas include adhesives, casting, coatings, flooring, laminating, electrical potting, and encapsulating.<sup>1</sup> The per-

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formance and utility of epoxies are, overall, dependent upon the nature of their structure, the chemical nature of the curing agent, and the conditions of cure. With proper formulating, which includes judicious choice of resin and hardener, epoxy systems can be devised to provide many outstanding properties, such as chemical and corrosion resistance, toughness, high adhesive strength, and high heat deflection temperatures. Difunctional epoxy resins will provide these properties in varying degrees of effectiveness. However, these properties can be optimized with the use of multifunctional epoxy resins.

### DEFINITION AND TRADITIONAL USE

By definition, multifunctional epoxy resins are those which contain more than two epoxy groups per molecule. As such, they provide a three-dimensional, densely crosslinked network when combined with the appropriate curing agents. This phenomenon is responsible for the excellent properties for which these systems are noted.

Since their introduction, multifunctional epoxies have traditionally been used in the electrical and structural industries. In combination with anhydride or aromatic amine curing agents, these resins are heat cured to form their networks of extremely high crosslink density. Today, however, the use of multifunctional epoxy resins need not be restricted to applications which require high gelation and post-cure temperatures.

This paper describes systems based on multifunctional epoxy resins which show promise in room temperature or force cured ( $65^{\circ}$ C) coating applications. Since they possess high crosslink densities similar to their adhesive, casting, and molding counterparts, these novel coating systems should afford excellence in the areas of chemical resistance, surface hardness, and thermal stability. Possible uses would include chemical tank linings, main-

Presented by Ms. DiBenedetto at the 57th Annual Meeting of the Federation of Societies for Coatings Technology in St. Louis, October 3, 1979. \*Resins Dept., Ardsley, NY 10502.

		Table 1—Comparative Physical Properties of Epoxy Resins	Epoxy	Resins				
Epoxy Resin	No.	Structure	EEW	Epoxy Value (eq/100g)	"n" Value	Functionality	Viscosity or M.P.	Appearance
EPN	-	o-cH;-cH-CH, o-cH;-cH-CH, o-cH;-cH-CH; CH,-cH,-cH; o-cH;-cH, o-cH;-cH,	175	0.57	0.2	2:2	1,700 cP. @ 52° C	Amber, extremely viscous liquid
ECN	7	$\overbrace{CH_i}^{O-CH_i}-\overbrace{CH_i}^{O-CH_i}, \overbrace{O-CH_i}^{O-CH_i}-\overbrace{O-CH_i}^{O-CH_i}, \overbrace{O-CH_i}^{O-CH_i}-\overbrace{CH_i}^{O-CH_i}, \overbrace{O-CH_i}^{O-CH_i}, [C]_{O-CH_i}, [C]_{O-CH_i},$	200	0.50	0.7	2.7	35° C	Light yellow solid
TGpAP	m	о-сн,-сн,-сн,-сн,-сн, сн,-сн, сн,-сн-сн,	011	06.0	L	0. 6	3,000 cP. @25° C	Amber liquid
DGEBPA	4	$\dot{c}H_{H}^{0}$ , $cH_{H}-cH_{H}-0$ , $cH_{H}$ , $cH_{H}$ , $cH_{H}-cH_{H}-cH_{H}-0$ , $cH_{H}-cH_{H}-cH_{H}$ , 189 $cH_{H}-cH_{H}-cH_{H}-cH_{H}$ , 189	681	0.53	0.2	<u>∞</u> .	@ 25° C	Light yellow, viscous liquid

### M. DIBENEDETTO

Journal of Coatings Technology

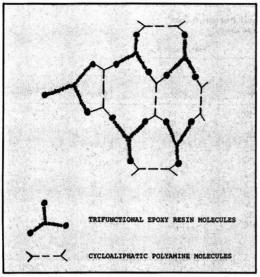


Figure 1—Optimum crosslink density of a trifunctional epoxy resin

tenance and marine coatings,<sup>2</sup> energy transmission, and drilling pipe applications.

### CHEMICAL NATURE AND STRUCTURE

The multifunctional epoxy resins used in this study were an epoxy phenol novolac (EPN), an epoxy cresol novolac (ECN), and triglycidyl para-aminophenol (TGpAP). *Table* 1 lists the general structures and the physical characteristics of these materials.

The EPN shown in Structure 1 of this table is the result of glycidylation of the reaction product of phenol and formaldehyde. In a similar manner, glycidylation of the reaction product of ortho-cresol and formaldehyde results in the ECN shown in Structure 2. TGpAP, pictured in Structure 3, results from the glycidylation of para-

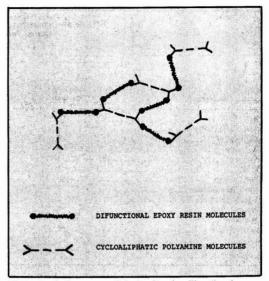


Figure 2—Optimum crosslink density of a difunctional epoxy resin

aminophenol. The presence of a tertiary amine in this structure imparts an "auto-catalytic" effect to systems containing this material.

The control, a commercial grade of diglycidyl ether of bisphenol A (DGEBPA) with a practical functionality of 1.8, is shown in Structure 4.

All of these materials are TSCA listed.

### **CROSSLINKING REACTIONS**

The key to the superior chemical resistance exhibited by systems containing multifunctional epoxies is the density of the crosslink network developed during the cure. The curing agents utilized are cycloaliphatic poly-

	Та	ible 2—Form	ulations of T	est Systems				
				Formula	ion No.			
	1	2	3	pb 4	N 5	6	7	8
Epoxy Resin								
DGEBPA	100					100	_	
EPN	_	100			_		_	_
ECN		-	100	-			-	_
TGpAP			_	100	_	_	100	-
LSU 909		_	_		_			100
LSU 912	—	—	_		100	—	—	-
Curing Agent								
Cycloaliphatic polyamine	50	54	48	86	68		—	_
Aliphatic polyamine adduct	-	-	-	_	_	20	34	24
Solvent								
Xylene	_		18		_			_

			Formulation No.			
	1	2	3	4	5	
Reagents	DGEBPA (Control)	EPN	ECN	TGpAP	Blend I	
HCl (36%)	58	89	14	< 2	< 2	
Acetic acid (10%)	4	12	10	< 2	< 2	
NH4OH (30%)	44	>150	93	93	48	
Cellosolve	< 2	7	7	>150	13	
Acetone	< 2	< 2	4	36	29	
MEK	< 2	< 2	< 2	>150	29	
Butyl acetate	>150	42	13	>150	>150	
Methylene chloride	< 2	< 2	< 2	29	29	
Trichloroethylene	< 2	< 2	< 2	>150	50	
Ethanol (95%)	4	26	7	29	29	
Methanol	< 2	68	16	< 2	< 2	
Skydrol 500B	>150	>150	>150	>150	>150	
(a) Days to failure						

### Table 3—Comparative Chemical Resistance Data<sup>a</sup> (Room Temperature Cured)

amines or aliphatic polyamine adducts. The choice of hardener would depend upon the particular requirements which the coating must meet.

A visual representation of the dense crosslink network which develops when 5 molecules of a multifunctional resin of epoxy functionality of 3.0 are reacted with 5 molecules of a cycloaliphatic polyamine is given in Figure 1. In contrast, reaction of the same number of molecules of a difunctional epoxy resin with the same polyamine molecules produces the less densely crosslinked configuration pictured in Figure 2.

### CHEMICAL RESISTANCE PROPERTIES

In order to demonstrate superior chemical resistance in the shortest amount of time, extremely aggressive chemical reagents were chosen for testing. These included inorganic and organic acids, plus solvents such as esters, ketones, chlorinated hydrocarbons, and lower molecular weight alcohols. Most coating systems based on difunctional epoxy resins are not resistant to such reagents, particularly to the aggressive solvents listed.

The coating systems tested were prepared according to stoichiometric mixing of epoxy resin and amine curing agent. The cycloaliphatic polyamine used in *Table 2* possessed an active hydrogen equivalent weight of 95. The polyamine adduct used in *Table 3* possessed an active

Table 4-Properties	of	Blends	of	DGEBPA	and	TG	AP
--------------------	----	--------	----	--------	-----	----	----

Blend I	Blend II
50/50	75/25
7,000	8,900
6-7	4-5
0.72	0.62
139	161
	2.2
	50/50 7,000 6-7 0.72 139

hydrogen equivalent weight of 38. Formulations are listed in *Table 2*.

The coatings were applied by doctor blade on sandblasted hot rolled steel panels at a film thickness of 16 mils. Curing took place at  $23^{\circ}$ C over a period of 10 days for the systems listed in *Table* 3. The systems listed in *Table* 4 were force cured for 10 hr at 65°C. All systems were clear and unfilled.

Chemical resistance testing was conducted in chemical cells at 23°C. Criteria for failure were both visual and tactile. Blistering, softening to substrate, substrate rusting, swelling, and actual dissolving of the coating were considered failures. The NACE Standard for evaluating protective coatings, ASTM D1308-57, and ASTM D1540-61 served as guides.

The enhanced chemical resistance obtained with multifunctional epoxy resins cured with a cycloaliphatic polyamine is demonstrated in *Table* 3.

Relative to the control, the system containing the EPN resin exhibited a threefold improvement in acetic acid (10%) resistance, at least a threefold improvement in ammonium hydroxide (30%) resistance, a sixfold improvement in ethanol (95%) resistance, and a thirty-fourfold improvement in methanol resistance. The use of the ECN resin was not as effective, but did extend the resistance to include a twofold improvement to acetone. Resistance to HCl (36%), however, was decreased. Possible steric hindrance, due to the presence of the ortho-methyl groups in this material, would prevent the formation of the higher degree of crosslink density theoretically obtainable with this epoxy resin. As a result, the ECN was not as effective an upgrader in practice as would be predicted from theory.

Blend I, a resin mixture described in *Table* 4, exhibited exceptional improvements not only over the control but also over the EPN- and the ECN-containing systems. Notable was improved resistance to ketones and chlorinated solvents.

	Formulation No.							
	1	2	5					
Reagents	DGEBPA (Control)	TGpAP	Blend II					
HCl (36%)	>300	120	240					
Acetic acid (10%)	150	150	150					
NH4OH (30%)	>300	210	210					
Cellosolve	7	>300	>300					
Acetone		>300	< 2					
мек	< 2	>300	>300					
Butyl acetate		> 300	>300					
Methylene chloride		150	< 2					
Frichloroethylene		>300	20					
Ethanol (95%)	9	>300	>300					
Methanol		>300	>300					
Skydrol 500B	>300	Not Tested	>300					

### Table 5—Comparative Chemical Resistance Data<sup>a</sup> (Force Cured)

The most dramatic results were seen with the system containing TGpAP. Although both inorganic and organic acid resistance was lost, startling improvements were obtained in resistance to acetone, methyl ethyl ketone, ammonium hydroxide (30%), butyl acetate, methylene chloride, trichloroethylene, and ethanol (95%). The loss of acid resistance is explained by the presence of the amino nitrogen in the molecule. This site is quite vulnerable to attack by acids.

The excellent results obtained with this trifunctional resin were paralleled in systems cured with an aliphatic polyamine adduct. *Table* 5 lists the results of a study in which coatings were force cured at  $65^{\circ}$ C for 10 hr before testing. Again, the control was based on DGEBPA. Blend II, another multifunctional resin mixture (*Table* 4), was included in this study.

The effects of force curing were immediately apparent on comparison of these three systems. The system containing TGpAP outperformed the control in all the areas except HCl (36%) and ammonium hydroxide (30%), where a decrease in performance was seen, and acetic acid (10%), where a parallel performance was observed.

The solvent resistance of Blend II was fairly comparable to that of TGpAP except in resistance to acetone and the chlorinated solvents. Resistance to trichloroethylene, however, was improved tenfold over that of the control.

Since encouraging results were obtained with these two multifunctional resin blends, the testing of multifunctional epoxies was extended to include the experimental systems listed in *Table* 6.

Systems A, B, and C are combinations of epoxy resins and proprietary curing agents. The curing agents are based on cycloaliphatic polyamines and possess active hydrogen equivalent weights of 66, 76, and 94, respectively. Respective mix ratios for A, B, and C are 35, 40, and 50 phr.

Reagents	System				
	DGEBPA/DETA (Control)	A (7W-12-02)	B (7W-10-01)	C (7W-07-01	
HCl (36%)	53	28	60	120	
Acetic acid (10%)		28	28	7	
NH4OH (30%)	>270	184	>270	>270	
Cellosolve	< 3	>270	>270	>270	
Acetone	7	>270	>270	< 2	
MEK	< 3	>270	65	>270	
Butyl acetate		>270	>270	>270	
Methylene chloride		< 3	< 3	< 2	
Trichloroethylene		163	101	>270	
Ethanol	>270	>270	>270	210	
Methanol	< 3	>270	>270	120	

### Table 6-Comparative Chemical Resistance Data of Proprietary Systems<sup>a</sup>

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	Formulation No.				
	1	2	3 ECN	4 TGpAP	
	DGEBPA (Control)	EPN			
Pot life (23°C), min	32	34	153	31	
Dust Dry Time, hr		2	3	3	
Through cure time, hr	6	4	6	6	
Pencil hardness Impact resistance, inlb	HB	3H	F	3H	
direct/reverse	16/0	24/0	24/2	18/0	
Adhesion	Excellent	Fair -Clear, colorless; no bl	Fair ushing or exudation—	Fair	

### Table 7—Comparative Formulation and Mechanical Properties

Since these systems represent novel approaches to increasing the chemical resistance of coatings, the only available control was one of the industry standards consisting of DGEBPA and diethylenetriamine (DETA). A mix ratio of 11 phr was used.

Noteworthy is the comparative superiority of the proprietary systems in resistance to Cellosolve,<sup>®</sup> acetone, methyl ethyl ketone, and methanol.

### **MECHANICAL PROPERTIES**

Besides the chemical resistance properties of systems containing multifunctional resins, some of the formulation and mechanical properties were also investigated. *Table* 7 lists these properties for systems whose chemical resistance properties were discussed in *Table* 3. These mechanical properties were generated on the formulations listed in *Table* 2 applied to cold rolled steel (Q-Panels®) at a film thickness of 7 mils and cured at 23° C for 10 days. Cold rolled steel was used since the gauge of the hot rolled steel used for chemical resistance testing was too thick for impact testing. The thickness of the substrate had no effect upon the outcome of pencil hardness, adhesion, dust dry, or through cure testing.

Pot lives were determined on a 100 g mass at 23°C using a Techne Gelometer. Dust dry and through cure times were determined according to the Landolt method.

Cellosolve is a registered trademark of Union Carbide Corp. Q-Panel is a registered trademark of Q-Panel Co. Pencil hardness testing followed the method described in the "Paint Testing Manual," 12th edition, 1962. Impact and adhesion tests were conducted according to ASTM D32794 and D3359, respectively. All systems were clear and unfilled.

All pot lives were found to be similar to that of the control with the exception of the system based on the ECN resin. Solvent addition, necessitated by the solid nature of this resin, would account for this observed extension in pot life.

The EPN-based system exhibited a faster dust dry and through cure time response than systems based on the other resins. Also, its pencil hardness (3H) was higher than that of the other systems but comparable to that of TGpAP. This higher degree of hardness is a direct result of the more densely crosslinked network obtained with the multifunctional resins. Steric hindrance, combined with the presence of residual solvent, serve to explain the softer film obtained with the ECN resin.

Impact resistance and film appearance properties were fairly comparable for all systems.

A major difference identified in this experimental work was in adhesion. As *Table* 7 indicates, the control exhibited excellent adhesion, while that of the other systems was only considered as fair. However, laboratory work presently underway to improve the adhesion of multifunctional resin-containing systems indicates promising results. Excellent adhesion has been obtained with the use of silane coupling agents. In addition, a notable increase in pencil hardness was also observed.

Table 8—Comparative Formulation and Mechanical Properties of Experimental Systems	Table 8—Comparative	Formulation and	Mechanical Pro	perties of Ex	perimental Sy	ystems
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	System				
	DGEBPA/DETA (Control)	A (7W-12-02)	B (7W-10-01)	C (7W-07-01)	
Pot life (23° C), min	33	52	51	42	
Dust dry time, hr		2	3	3	
Through cure time, hr		5	7	8	
Pencil hardness		4H	4H	4H	
Impact resistance, inlb	and an address of the second			constant of the second	
direct/reverse	160/0	16/8	16/2	20/12	
Adhesion	Fair	Fair	Good	Fair-Poor	
Film appearance	Blushed, dry	Clear, dry; very high gloss			

Where Table 7 lists the mechanical properties of room temperature cured systems, Table 8 lists a comparison of the mechanical properties of systems cured at  $65^{\circ}$  C for 10 hr. The chemical resistance properties of these systems are discussed in Table 4.

The fact that all of the experimental systems (A, B, C) exhibited shorter dust dry times than the control is significant. The film appearance of these systems was also superior to that of the control. However, through cure times were comparable to that of the control for system A, but longer for systems B and C. Impact resistance values were fairly comparable in all cases.

Improved chemical resistance, then, can be obtained without a great sacrifice of mechanical properties.

#### CURRENT EPOXY SYSTEMS

Commercial epoxy systems for protective coatings are presently based on two main types of systems. The first is DGEBPA (EEW 189) cured with polyamines or polyamine adducts based on DETA or TETA, etc., while the second type is a "1" type epoxy/polyamide system.

The performance of a DGEBPA/DETA system has already been seen in comparison with our experimental systems. For further comparison, the resistance of an epoxy/polyamide system was tested. The epoxy resin was a bisphenol A-based solid material and possessed an EEW of 500. The polyamide had a viscosity of 70,000 cP at 40° C and an amine value of 238. The mix ratio was 50 phr. Due to the high viscosities involved, 37 parts of a mixture of Cellosolve, xylene, and butanol, in equal proportions, were added to the formulation. Tris(dimethylaminomethyl) phenol, 4 phr, served as an accelerator.

This system was cured for 10 hr at  $65^{\circ}$ C before testing. Panels were prepared and tested according to the conditions previously described.

The poor performance observed in *Table* 9 underscores the inability of epoxy/polyamide systems to meet the growing need for excellence in the area of solvent resistance. Table 9—Chemical Resistance of an Epoxy/Polyamide System<sup>a</sup>

Reagents	Epoxy/Polyamide
HCl (36%)	 < 2
Acetic acid (10%)	
NH4OH (30%)	
Cellosolve	
Acetone	 < 2
MEK	
Butyl acetate	 < 2
Methylene chloride	
Trichloroethylene	
Ethanol (95%)	5
Methanol	 5
(1) D	
(a) Days to failure	

CONCLUSIONS

Comparative testing has demonstrated that the use of multifunctional epoxy resins in the coatings area will greatly enhance resistance to aggressive chemical reagents, particularly to solvents. With this finding, multifunctional epoxies face a new dimension of application. Commercially proven success in adhesives and in the electrical and structural industries supports the belief that, with the proper formulation and cure conditions, these resins will also become firmly established in the coatings industry.

Further development and testing of systems containing multifunctional epoxy resins are currently underway to expand the scope of application for these materials.

#### ACKNOWLEDGMENT

The author thanks Ms. Rita Bosco and Mr. Frank Forte for their invaluable assistance in this study.

#### References

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- (2) Banfield, T.A., "The Protective Aspects of Marine Paints," Progress in Organic Coatings, 7, 253-277 (1979).

### **Constituent Societies Report on Technical Programs**

This report is published as part of the Federation's efforts to keep members informed of technical activities, both underway and planned, and to stimulate interest in formulating new programs at the local level.

#### Baltimore

Four projects currently underway: (1) Formulation Data Retrieval—Objective is to develop a low cost system for storage and retrieval of product formulas, primarily for access of current and historical data; programs have been developed, using micro-computer hardware, on formulation, inventory, costing, etc.

(2) Near Infrared Reflectance of Paint Pigments-Compilation of infrared spectra of pigments for use by coatings industry in developing solar selective coatings-studies have shown that dark colored coatings reflect solar energy if pigments selected have high reflectance in the spectral region between 0.6-2.5 microns. To make technology available, the near infrared reflectance characteristics of pigments must be determined. To date, 80 pigment dispersions in an acrylic resin binder have been prepared and applied by drawdown over black and white hiding power charts. Spectra at 2 film thicknesses have been measured on 71 of these from 0.4-2.5 microns. Several sets of duplicate colors are planned, with one of each set using high IR absorbing pigments and the other with low absorbing pigments, and then collecting surface temperature data to demonstrate how such spectra can be used-paper anticipated for 1981 Annual Meeting on this work.

(3) Correlative Testing—Round-robin testing is offered to any member laboratory wishing to correlate questionable test results. Round-robin testing is currently underway on reflectance of a flat white paint to assist member company correlate readings between two of its plants; nine labs are cooperating in this effort. Service is also available to ASTM Committee D-1.

(4) Coatings Library—Attempting to establish coatings library for use by members; list of technical publications currently being assembled.

#### Birmingham

Completed slide/tape presentation on "The Setaflash Tester" and submitted to Federation . . . Developing slide/tape program on "An Introduction to the Paint Industry," which focuses on career opportunities . . . Sponsoring one-day symposium on "Effects of Current Legislation in the U.K. upon Industry"  $\dots$  Have set up operating procedures to act as agents in the U.K. for marketing Federation literature and slide/tape programs.

#### Chicago

Four subcommittees currently active in project work:

(1) High Solids Oligomers—Preliminary work has been initiated on literature search to determine that effort will not intrude into proprietary procedures and proprietary products.

(2) Water-Borne Anti-Corrosion Paints—Literature search being undertaken and contact initiated with appropriate allied organizations to obtain all available information on correlation of salt fog and other corrosion tests with actual field exposures. Ultimately, hope is to be able to recommend specific lab tests to predict actual field performance.

(3) Biocide Polymerization—Organization has been established to evaluate all experimental biocidal polymers as they are produced, including test fences, microbiology labs, biocide chemists, paint formulators, and polymer suppliers; in addition, written program is being prepared to instruct each participant for uniformity in testing.

(4) Renewable Resources—Literature search underway to explore every avenue, particularly among flora, that will produce a resource useful in either paint or petrochemical industry; also being considered is "cracking" of such things as drying oils, inedible lard, turpentine, etc., as a source of new monomers for polymerization. In addition, new fermentation procedures being considered to produce new products from old raw materials, i.e., methacrylic acid from cellulose or polysaccharides. Plan to report on work at 1980 Annual Meeting.

#### Dallas

Current project is on Industrial Water-Borne Vehicle, State of the Art—Selected coatings to be produced for a few comparative physical properties ... Sponsored joint program with Education Committee on "Formulation of Interior Flat Latex Paints"... Chairman made presentation to polymer students at University of Texas at Arlington on subject of "Polymer Application-Research in the Latex Coatings Field," which highlighted career opportunities in the industry... Plan paper for 1981 or 1982 Annual Meeting on Industrial Water-Borne Coatings (Baked/Spray Systems).

#### **Golden Gate**

Currently evaluating exposure performance of commercially available nontoxic, water-borne, corrosion-inhibitive primers that conform to CARB's volatile organic compound regulations for 1984: methodology outlined in Steel Structures Painting Council's PACE (Performance of Alternate Coatings in the Environment) project being followed insofar as practical. Primers from five raw materials manufacturers have been coated at 2 mils on hot rolled steel panels, and are being exposed to salt fog, water immersion, and atmospheric environments. Paper planned on this work for 1980 Annual Meeting.

#### **Kansas City**

Project underway on Evaluation of Performance of Coatings on Exterior Hardboard Surfaces. Goal is to identify cause of discoloration, peeling and generally poor coating performance on exterior hardboards in the midwest, and to develop proper coating recommendations to give optimum performance over this type of substrate. Future considerations would include paint performance on hardboard with varying wax content; evaluation of proprietary coatings on hardboard; and recoatability of hardboards that have exhibited paint failures.

#### Los Angeles

(1) The Angle Function of Color —Long-standing problem is matching deep colors from high gloss to dead flat. It has been determined that each color has its angle or range of angles at which colors should be viewed. Instrument is available which has three light sources and a device for changing the angle of view; some colors (deep black) have very short angle in which colors can be matched, others have very wide angle. Project goal would be to determine cause of color difference at different angles of viewing, primarily with flat to high gloss matches.

(2) Study of Extenders with Varying Surface Treatments Serving as Fillers for Polymers—Object is to determine impact of surface treated extenders on physical properties of polymers in pigmented coatings; will include study of changes of barrier properties relating to water permeability, performance of treated extenders in flash rusting and salt spray resistance with a view to compiling data for comparing treated and untreated extenders for general performance in corrosion atmospheres. Initial work is with styrene acrylic resin pigmented with silane treated talcs. To date, treated talcs have been prepared, base formulations have been worked out, and finished paints for study will be available shortly; water permeability and corrosion resistance tests will then be run on these materials. Ultimate aim is to expand study to other surface treated extenders and to differing polymer systems.

#### Louisville

Project underway on Extender Pigments in Latex Wall Paints-intent is to review various extender pigments currently used in latex wall paints and catalogue their influence on critical paint properties. Seventeen extender pigments were selected for evaluation; work carried out in typical latex flat wall paint formulas at 50 and 60 PVC has been completed and results are now being compiled. Data from this work (which will be reported in paper to be presented at 1980 Annual Meeting in Atlanta) will permit correlations to be drawn concerning effect of extender pigments on important paint properties such as hiding, scrub resistance, stain resistance, and raw material costs ... Waste management control subcommittee working with student at University of Louisville on study of waste samples supplied by Louisville firms to determine best method of treatment.

#### Montreal

#### Two projects currently:

(1) Factors Influencing Freeze-Thaw Stability in Flat Latex Paints—In recent years, resistance to freezing and thawing of flat latex paints has fluctuated in importance, primarily dictated by cost and availability of ethylene glycol and the cost of heated transport. Purpose of this project is to show how formulating parameters other than ethylene glycol influence freeze-thaw stability of flat latex paints. Paper will be presented on this work at the 1980 Annual Meeting.

(2) Adhesion of Latex Paints—Work has been directed to semi-gloss latex paints and their adhesion to alkyd substrates.

#### New England

Project work continuing on various aspects of corrosion control. Current study is on Flash Rust Inhibitors: An Evaluation of Some Amines and Organic Salts in an Aqueous Acrylic Coating —Some amines and organic salts are being evaluated at different concentration levels in an aqueous acrylic coating for flash rust resistance over bare steel. Coatings on bare steel will also be evaluated for humidity resistance and salt spray resistance to evaluate effect of additives on the coating's environmental exposure performance. Paper will be presented at 1980 Annual Meeting.

#### **New York**

Four projects currently being worked on are in various stages of development: Defoamers; Pigment Stability in Aqueous Systems; Low Cure Systems; and Waste Control... Other projects being considered include: Soluble Non-Solution Coalescents for Emulsions; Air Drying Gloss Water Vehicles; Cross-Linking of Emulsions; High-Solids House Paint; and Corrosion.

#### Northwestern

Working on 3-dimensional study of effects of Environment on Curing of Normal House Paint; will study air flow speed, as well as temperature and humidity. Literature search is underway and work will initially involve only long oil alkyd type, flat exterior paint, and acrylic latex; will work with stable substrate —metal panels with baked primers. Must have constant environment chamber for testing; will run variety of tests: wet and dry; tensile; cross-hatch adhesion; plus some outside exposure tests.

#### **Pacific Northwest**

Working on development of data sheet for handling of hazardous materials for use by in-plant floor operators.

#### Philadelphia

Three projects currently being worked on:

 Flash Point Study—Reactivated subcommittee is reviewing data of previous studies and plans to develop new project, possibly involving study of high solids vehicle systems.

(2) Arrhenius Plot Study—Literature search on application of the Arrhenius equation has been completed; data from exposure studies and lab stability tests are being considered for analysis by this technique.

(3) *HLB System*—New subcommittee formed to explore the HLB number of water dispersible/soluble resins.

Other projects include an audio/visual program on "A Microbiological Audit of a Paint Plant" and one on "Color," as well as sponsoring seminar on "Quality Assurance."

#### Pittsburgh

Work getting underway on Waterborne Coatings Flash Point—Object is to determine precision and pinpoint difficulties in measurement of flash points of waterborne coatings. Samples are being prepared and will be sent out soon. Both latex and dispersion-type paints will be included, and both miscible and imiscible solvents... Additional project will be cooperative effort with ASTM subcommittees and working groups that need help on round-robin testing.

#### Southern

No projects currently, but several being considered. One is on Natural Clear Wood Finishes. Others are on: Interpolymerized Biocide Latex Paints; Low Solvent Industrial Coatings; Microvoids; Concrete Coatings; and Paint Properties to Aid Home Insulation.

#### St. Louis

Planning project to develop method for determining resistance properties of coatings.—This would be a simple and accurate method for determining comparative resistance of films to water, chemicals, and solvents. Hope to have paper for 1981 Annual Meeting.

#### Toronto

Four projects currently active:

(1) Binder Index—Paper presented at 1979 Annual Meeting noted anomalous behavior of certain pigments in course of determining their CPVC or in relating the CPVC value to binder index. Subcommittee plans to continue its efforts to elucidate reasons for the unusual observations and furnish the required proof.

(2) Wet Adhesion—Work on this project produced paper at 1979 Annual Meeting which presented conclusions on: value of wet adhesion test methods, successful preparation of a good wet adhesion polymer based on vinyl acetate monomer, and influence of paint formulating parameters on wet adhesion. Work is continuing to clarify significance of parameters of influence of polymer molecular weight and particle size on wet adhesion, with respect to PVC and PVC/CPVC.

(3) Parameters Affecting Wear Rate in Small Media Mills—Objectives are focused on pigments, grinding times, media types, and wear rates. Some interesting conclusions have been generated on media and mill lining types and media size vs. dispersed pigment particle size; paper planned on this work for 1981 Annual Meeting.

(4) Assessment of Flow and Sagging by a Novel Instrumental Technique—Still in early stages, this project will evaluate fluid flow in the ultra-low shear rate range. Apparatus has been assembled and several trials have been run to demonstrate feasibility. Literature search is underway.

### Society Meetings

#### Cleveland

May 20

Honored during the meeting were the following Society Past-Presidents: Charles Beck (1978-79); Helen Skowronska (1977-78); Fred G. Schwab (1976-77); Tom Keene (1975-76); Paul Sleeman (1971-72); Vic Sandorf (1967-68); Ken Waldo (1965-66); Robert Taub (1963-64); Fred Hollenburg (1958-60); George Selden (1957-58); Mike Malaga (1956-57); Bill Tomc (1953-54); Sam Huey (1949-50); Gordon Muttersbaugh (1940-41); and Ed Shulte (1938-39).

Federation Honorary Member David Gans and Society Honorary Members Gord Muttersbaugh, Ed Schulte, and John Weaver were also recognized.

The following slate of officers was elected for the year 1980-81: President— Jack S. Malaga, of Body Brothers, Inc.; President-Elect—Thomas Tuckerman, of Body Brothers, Inc.; Secretary—Carl J. Knauss, of Kent State University; and Treasurer—Girish Dubey, of Cambridge Coatings Co.

John C. Weaver was presented with an Award of Merit for his fine service to the coatings industry.

The presentations of 25-year pins were made to Robert S. Taub, Kenneth C. Waldo, and John W. Culler. All are employed by the Sherwin-Williams Co.

In addition, Science Fair Awards were presented by the Society to Gary Grottenthaller (Senior High) and Rafael Omerza (Junior High).

Mrs. Anne Price and Mrs. Linda Fulp, of SCM Corp., gave a slide presentation focusing on "THE AFFECTS AND IMPOR-TANCE OF COLOR IN DECORATING AND FURNISHING."

THOMAS D. TUCKERMAN, Secretary

#### Dallas

May 15

The following officers were elected for 1980-81: President—Richard Williamson, of Trinity Coatings Co.; Vice-President—Ray Marett, of Western Specialty Coatings Co.; Secretary—William Wentworth, of Jones Blair Co.; Treasurer— Leon Everett, of Dan-Tex Paint & Coatings Mfg.; and Society Representative— Carlos Dorris, of Jones Blair Co.

Honored during the meeting were these Past-Presidents in attendance: Fred L. Couch (1952-53); Willis R. Currens (1939-40); Ross D. McLarty (1947-48); C. Gus Anderson (1962-63); Marvin D. Smith (1966-67); F. Wayne Minner (1967-68); John R. Rothermel, Jr. (1969-70); Ollis L. Walker (1970-71); John C. Walter (1971-72); and Tom B. Currens (1972-73).

Ed Antonucci, of Drew Chemical Co., spoke on "FLASH RUST INHIBITION IN WATER-BASED PAINTS."

Defining flash rusting as that which occurs during the first thirty minutes to four hours of drying, Mr. Antonucci stated that there is no single product that will prevent it in all formulas. Although several chemicals were shown to have no effect, barium metaborate has been successfully used and sodium nitrate is beneficial at a pH of  $8\frac{1}{2}$  to 10, he said. To prevent flash rusting the objective is to prevent oxygen formation or to neutralize the acid formed in the corrosion process. Mr. Antonucci noted that some solvent-type coatings will cause flash rusting if moisture is present.

Q. Can an inhibitor hurt salt spray resistance?

A. If the second coating of the system has good adhesion, it is not likely.

Q. Can phosphate treatment in metals help?

A. Yes.

Q. What are manufacturers doing to their products to improve them?

A. They have changed the emulsifiers, raised the pH, and some are incorporating salts in their products.

W. A. WENTWORTH, Secretary

May 19

#### **Golden Gate**

Robert C. Anderson, of Tokheim Corp., spoke on "METERING SYSTEMS FOR PAINTS AND THEIR COMPONENTS."

Mr. Anderson presented a series of slides depicting meters and their various uses, such as increasing efficiency, stopping material loss, and improving safety conditions. Included in his discussion were several types of meters available to the industry, including rotary positive displacement meters, flow meters, and piston positive displacement meters.

When choosing a meter, it is important to determine the products to be metered, as well as the chemical composition, viscosity, and solids of these products, he explained. With this information, the proper selection can be made of an instrument with the correct accuracy, metallurgy, and capacity. Mr. Anderson described a number of options, such as automatic shut-off valves, pulsers, batch controllers, and strainers which can be added to complete the typical installation.

Mr. Anderson pointed out the importance of proving the meter with a known container volume to check accuracy following installation of the system. In order to keep this accuracy, cleanliness is important, he said. A flush with solvent or water may be necessary to keep some materials from settling in the meter components.

Each meter is a delicate instrument and, if treated properly, will last for years and will assist in performing tasks efficiently and accurately, he concluded.

DON L. MAZZONE, Secretary

#### Los Angeles

May 14

This meeting was designated Past-Presidents Night and the following were present: Fred Croad (1978-79); Bob Koperek (1977-78); Gerry West (1976-77); Ken O'Morrow (1975-76); Bob McNeill (1974-75); Duke Cromwell (1963-64); Alan U. Hershey (1961-62); Fred Oberlin (1960-61); John R. Warner (1945-46); R.J. Blackington (1944-45).

In recognition of their contributions to the Federation, 25-year pins were awarded to Arnie Hoffman, Earl Fenstermaker, Tom Nicholson, John Plant, Al Seneker, Wharton Jackson, Bob McNeill, and Bob Koperek.

Robert Anderson, of Tokheim Corp., spoke on "METERING SYSTEMS FOR PAINTS AND THEIR COMPONENTS."

Q. What type of meter is best when a pulsating (diaphragm) pump is used?

A. It really depends on what type of capacity you need. If you are looking for low capacity, a piston meter would probably be best.

Q. Can you use an air pump diaphragm with your meters? Is it hard to calibrate?

A. Yes, you can, if you use a back pressure valve. It is quite easy to calibrate with a rotor meter by taking a volumetric reading. It is a little more difficult to calibrate with a piston meter, but it can be done.

ROMER E. JOHNSON, Secretary

#### Los Angeles June 11

A moment of silence was observed in memory of Jim Gartland, of Miramar

Publishing and Tom McEneany, of PVO International, who died recently.

The following officers were elected for the year 1980-81: President—Donald Jordan, of Cargill, Inc.; Vice-President— Jan Van Zelm, of Byk-Mallinckrodt; Secretary—Lloyd Haanstra, of Ameritone Paint Corp.; and Treasurer— Romer Johnson.

Trev Whittington, instructor of the L.A. Trade Technical Paint Technology Course, announced that the third semester will be held in the fall of this year. A second course, taught by Bill Meadows and George Kashmer, will be added in the fall and will follow the lines of the Pacific Northwest curriculum.

Robert E. Van Heuit, of the Los Angeles County Sanitation District, gave a presentation entitled "LET'S TALK TRASH."

Mr. Van Heuit explained that the 30,000 tons of solid waste materials generated per day by the County of Los Angeles is primarily disposed of in land fill developments. Various land fills and the uses they are put to upon their retirement were described. To combat the expected higher costs of disposing of this trash, transfer stations have been established which take waste from smaller vehicles, store and compact it, then load it into larger vehicles that can make the longer haul more economically, he revealed.

Mr. Van Heuit stated that recovery of resources from solid waste is one of the primary goals of the sanitation districts. The recovery of decomposition gases is one workable method, he said.

Q. What are the chances of a Love Canal situation occurring in the land fill sites due to liquid chemical disposal?

A. Relatively slim. The Love Canal was strictly used for chemical waste whereas ours are mainly dilute wastes as they are mixed with other materials. We employ a much higher technology.

Q. What are the provisions made in a land fill to prevent contamination of the ground water in the surrounding area, especially contamination from solvents and water-soluble chemicals?

A. The Regional Water Quality Control Board (RWQC) investigates thoroughly any proposed site. There are three classes of sites: Class III which can only receive inert materials; Class II which can receive biodegradable materials and Class I which can receive the former plus hazardous and liquid wastes. The geology of the site is important because we must prove to the RWQC that we have excellent control in the protection of surface and ground water. Only three Type I sites exist in the L.A. County.

ROMER E. JOHNSON, Secretary

#### **Constituent Society Meetings and Secretaries**

BALTIMORE (Third Thursday—Eudowood Gardens, Towson). DONALD KEEGAN, Valspar Corp., 1401 Severn St., Baltimore, MD 21230.

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). LAYTON F. KINNEY, Standard T Chemical Co., Inc., 10th & Washington Ave., Chicago Heights, IL 60411.

C-D-I-C (Second Monday-Sept., Nov., Jan., Mar., May in Columbus; Oct., Dec., Feb., Apr., in Cincinnati, Kings Island Inn). NELSON W. BARNHILL, Inland Div., G.M.C., P.O. Box 1224, Dayton, OH 45401.

CLEVELAND (Second Tuesday-meeting sites vary). CARL J. KNAUSS, Kent State University, Kent, OH 44242.

DALLAS (Thursday following second Tuesday). WILLIAM A. WENTWORTH, Jones-Blair Co., P.O. Box 35286, Dallas, TX 75235

DETROIT (Fourth Tuesday—meeting sites vary). JOHN J. GENTILLA. Union Carbide Corp., 26500 Northwestern Hway., 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). KLEBERT JACOBSON, Cron Chemical Corp., P.O. Box 14042, Houston, TX 77021.

KANSAS CITY ( Second Thursday—Cordon Bleu). MIKE BAUER, Tnemec Co., Inc., P.O. Box 1749, Kansas City, MO 64141.

LOS ANGELES (Second Wednesday-Steven's Steak House). LLOYD HAANSTRA, Ameritone Paint Corp., P.O. Box 190, Long Beach, CA 90801.

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). G.L. SIMPSON, Sico, Inc., 3280 Blvd. St. Anne, Quebec, P.Q., Canada G1E 3K9.

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, NJ). TED YOUNG, Jesse S. Young Co., Inc., P.O. Box 275, Hewlett, NY 11557.

NORTHWESTERN (Tuesday after first Monday-Edgewater Inn). DON EMCH, Valspar Corp., 1101 3rd St. S., Minneapolis, MN 55415.

PACIFIC NORTHWEST (Portland Section—Tuesday following second Wednesday; Seattle Section—the day after Portland; British Columbia Section the day after Seattle). ROBERT MILLER, Imperial Paint Co., 2526 N.W. Yeon, Portland, OR 97210.

PHILADELPHIA (Second Thursday-Valle's Steak House). WAYNE KRAUSE, Lawrence-McFadden Co., 7430 State Rd., Philadelphia, PA 19136.

PIEDMONT (Third Wednesday—Howard Johnson's Coliseum, Greensboro, NC). SARA M. ROBINSON, Union Oil Co. of Calif., P.O. Box 7129, Charlotte, NC 28217.

PITTSBURGH (First Monday—Skibo Hall, Carnegie Mellon Univ.). WILLIAM CIBULAS, Mobay Chemical Co., Penn Lincoln Pkwy. W., Pittsburgh, PA 15205.

ROCKY MOUNTAIN (Monday prior to second Wednesday). DONALD R. BAGGE, George C. Brandt, Inc., 5050 Colorado Blvd., Denver, CO 80216.

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; Memphis Section—Second Tuesday). WILLIAM G. EARLY, Piedmont Paint Mfg. Co., P.O. Box 6623; Station B, Greensville, SC 29606.

TORONTO (Second Monday—Town and Country Restaurant). A. SUK, K-G Packaging Ltd., P.O. Box 658, Concord, Ontario, Canada L4K 1C7.

WESTERN NEW YORK (Second Tuesday—Holiday Inn, Cheektowaga, NY). CHARLES S. GLINSKI, Pierce & Stevens Chemical Corp., 710 Ohio St., Buffalo, NY 14240.

#### **New York**

#### May 13

The following slate of officers was elected for 1980-81: President—Marvin Schnall, of Troy Chemical Corp.; Vice-President—Don Brody, of Skeist Laboratories, Inc.; Secretary—Ted Young, of Jesse S. Young Co., Inc.; and Treasurer—Herbert Ellis, Jr., of D. H. Litter Co., Inc. Serving on the Board of Directors until their terms of office expire in 1980 are Walter Engel, Jeffrey C. Kaye, Sanford M. Liedeker, and Arthur Nortman.

Dr. Robert E. Baier, of Calspan Corp., spoke on "BLOOD-COMPATIBLE COAT-INGS FOR ARTIFICIAL ORGANS, THE ULTIMATE CHALLENGE."

DON BRODY, Secretary

#### Northwestern— Winnipeg Section

lips Paint Products, Ltd.

Society.

The following slate of officers was

elected for the year 1980-81: Chair-

man-Ed Deefholts, of Northern Paint

Ltd.; Secretary-Francois Lafortune,

and Treasurer-Bert McWade, of Phil-

Honored guests in attendance included

Frank J. Borrelle, Federation Executive

Vice-President and Lowell Wood, So-

ciety Representative for Northwestern

his discussion, Mr. Borrelle detailed Fed-

eration activities and services.

Incorporating a slide presentation into

ED DEEFHOLTS, Secretary

May 28

#### Piedmont

A moment of silence was observed in memory of H. Harris Ruwe, Society Honorary Member, who died recently.

Alfred Whitton, of NL Chemicals, spoke on "RHEOLOGY."

After defining flow, sheer rate, and yield value, Mr. Whitton discussed the different types of rheological additives available. Viscosity measuring instruments were also described. The cone and plate type viscosimeter is the most accurate of all types, he stated. Mr. Anderson explained the best methods of incorporating rheological additives. He concluded his presentation with a comparison between organo-clay and organictype additives.

JAMES ALBRIGHT, Secretary

#### Piedmont

June 18

Debra Massouda, of Hercules, Inc., spoke on "RELATIONSHIP AMONG CEL-LULOSIC MOLECULAR WEIGHT, POLY-MER SOLUTION RHEOLOGY, AND THICK LATEX INTERACTION."

Ms. Massouda explained how polymer solution relates to high sheer paint properties and how thick latex relates to low sheer paint properties. Under polymer solution rheology, the elasticity of a polymer controls spatter. As paint is applied by a roller, it stretches from the work to the roller. When it breaks, a spatter results.

Ms. Massouda explained the measure of viscosity flow. Using slides, she illustrated steady shearing as pure viscosity, transient shear as viscosity recovery, and dynamic shear as strain rate. In conclusion, Ms. Masssouda noted that most fluids are viscoelastic because most materials have elasticity.

Q. How do you measure elasticity of a polymer?

A. Extensional viscosity is used and measured using a cone and plate viscometer.

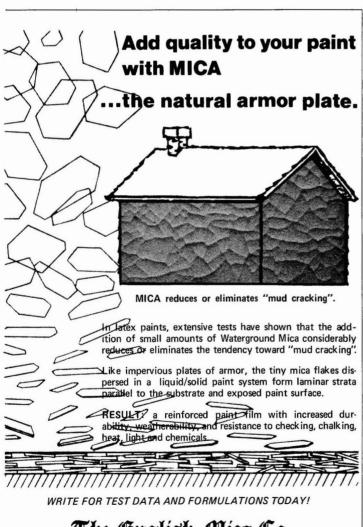
JAMES ALBRIGHT, Secretary

#### Rocky Mountain May 12

The following slate of officers was elected for the year 1980-81: President— Bruce Regenthal, of Komac Paints; Vice-President—Stephen Crouse, of Kwal Paints, Inc.; Secretary—Don Bagge, of George C. Brandt, Inc.; and Treasurer— Don Shillingburg, of Union Chemical.

Robert Anderson, of Tokheim Corp. spoke on "METERING SYSTEMS FOR PAINTS AND THEIR COMPONENTS."

STEPHEN CROUSE, Secretary



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## Elections

#### BALTIMORE

#### Active

BROWN, WAYNE M.—Contact Paint & Chemical Co., Baltimore, MD.

CRISTY, J. LEE – Duron, Inc., Beltsville, MD. PRESTON, HENRY C. – Lenmar Lacquers, Inc., Baltimore.

RATAJCZAK, EDWARD A.—Lenmar Lacquers, Inc., Baltimore.

Associate

BERBERIAN, EDWARD M.—Columbian Chemicals Co., Cranbury, NJ.

#### CHICAGO

#### Active

- ALLARD, DAVID N.-Rust-Oleum Corp., Evanston, IL.
- BALLWANZ, SANDRA J.—DeSoto, Inc., Des Plaines, IL.
- BELONGIA, DAN-Valspar Corp., Rockford, IL.
- BRIGGS, LINDA—DeSoto, Inc., Des Plaines. CLIFTON, MARY FRANCES—DeSoto, Inc.,
- Des Plaines. FETHIERE, JOHN R.-Rust-Oleum Corp.,
- Evanston. FISKE, ROBERT E. – DeSoto, Inc., Des Plaines. GRONET, ARTHUR T. – Armstrong Prods. Co.,
- Warsaw, IN. HANSEN, NESTOR PAUL-DeSoto, Inc., Des
- Plaines.
- HAYDEN, CARL-Dana Corp., Lisle, IL.
- JACKSON, EMMOJEAN-DeSoto, Inc., Des Plaines.
- LAVIN, COLLEEN A.—DeSoto, Inc., Des Plaines.
- PRIEST, BRAD-Mills-Winfield Eng. Sales, Inc., Chicago, IL.

Associate

- AMBERG, EDWARD J. JR.—Promac, Inc., Michigan City, IN.
- JOHNSON, MARK D.—ICI Americas, Inc., Wheeling, IL.
- MAGETT, ROY-BASF Wyandotte Corp., Palatine, IL.
- PARKER, DON W.-Promac, Inc., Michigan City.
- WEINER, RUBIN-BASF Wyandotte Corp., Hoffman Estates, IL.

#### CLEVELAND

#### Active

- KRUEGER, WILLIAM A.—PPG Industries, Inc., Cleveland, OH.
- MALIHI, FARROKH B.-Glidden Coatings & Res., Div. SCM, Strongsville, OH.
- SNIFF, WALTER-Orrville Prod. Inc., Orrville, OH.

SNYDER, JOHN J.—Republic Steel, Independence, OH.

#### Associate

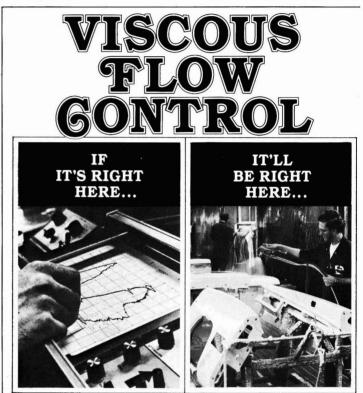
- FRANZEN, WAYNE L.—Rohm and Haas Co., Cleveland, OH.
- HARMAN, B. NEAL—Rohm and Haas Co., Cleveland.
- MORSEK, ROBERT J.—Cabot, Cab-O-Sil Div., Rocky River, OH.

DETROIT

Active

- AFEK, STANLEY Firestonesteel Prod., Riverview. MI.
- BADYRKA, PAUL-Magni Industries, Troy, MI.

DETZLER, DAVID A.—Chrysler Corp., Troy. DRAKE, SHEILA G.—Grow Group, Inc., Troy.



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- FEISEL, ROBERT B.-Wyandotte Paint Products, Troy.
- FLAHERTY, JOHN P.—Argo Paint & Chem. Co., Westland.
- FRANTZ, KENNETH LOUIS-Electro-Cote Chemical, Detroit, MI.
- FRENETTE, DANIEL A.—Fiberglas Canada, Sarnia, Ont., Can.
- GOSSE, J. ROBERT G.—Argo Paint & Chem. Co., Westland.
- GRAHAM, DR. JOHN C.-Seibert Oxidermo, Inc., Romulus, MI.
- HILL, BRUCE H.-Cook Paint & Varnish Co., Detroit.
- KREYSSLER, JOHN-Chrysler Corp., Bloomfield Hills, MI.
- LIEDTKE, ROBERT-Wyandotte Paint Prod. Troy.
- MARUSZCZAK, WALTER-Valspar Corp., Ferndale, MI.
- MCCORMACK, TIMOTHY B.—Titan Finishes Corp., Riverview.
- O'TOOLE, MICHAEL-Seibert Oxidermo, Romulus.
- PASKOFF, PHILIP H. Wyandotte Paint Prod., Troy.
- PELLOSKI, PETER E. Magni Industries, Troy.
- PETTIT, JR., PAUL H.-Grow Group, Inc., Troy.
- TOCHMAN, ANTHONY Argo Paint & Chemical Co., Westland.
- WILLIAMS, CHARLES-Grow Group, Inc., Troy.

#### Associate

- BREN, WILLIAM D.—Spencer Kellogg Div., Textron, Troy, MI.
- KENT, ARTHUR J.—Seegott, Inc., Lathrup Village, MI.
- MARSHALL, RICHARD-R.J. Marshall Co., Farmington Hills, MI.
- OLINGER, DEAN-Clawson Tank Co., Clarkston, MI.
- SAAD, ANNE M.-Union Carbide Corp., Southfield, MI

#### LOS ANGELES

#### Active

- ARCEBAL, BILL G.—Modern Coatings Inc., Cerritos, CA.
- CROAD, FRED-Tnemec Co., Compton, CA. DE LA VEGA, EVE-Cal-Western Paints,
- Santa Fe Springs, CA. KO, BENITA U.-Glidden Div. SCM Corp.,
- Los Angeles, CA.
- OLLERVIDES, ROBERTO E. Quimica Ind. Del Norte, San Ysidro, CA.
- SHAH, SUBHASH V.-MPV Co., Vernon, CA.
- WEST, GERALD L.—Devoe & Raynolds Co., Inc., Riverside, CA.

#### Associate

- BRISLIN, EDWARD J.-R.T. Vanderbilt Co., Los Angeles, CA.
- MULLINS, PETER J.—Miramar Publishing, Los Angeles.

#### Retired

LITTLE, KENT E.-Pico Rivera, CA.

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#### **NEW YORK**

#### Active

- BORN, EDWARD F.—Tenneco Chemicals, Inc., Piscataway, NJ.
- BORROMEO, SEVERA D.-Hoboken Paints, Inc., Lodi, NJ.
- HURLEY, RAYMOND—Tenneco Chemicals, Inc., Piscataway.
- JACKSON, SUZANNE M.—Tenneco Chemicals, Inc., Piscataway.
- KALATHUNKAL, JOSEPH P.-Koppers Co., Inc., Newark, NJ.
- ST. AMOUR, DAVID-Maratmon Industries, Inc., Sayreville, NJ.
- TURNER, CHARLES—Premier Vacuum Proc., Maspeth, NY.

#### NORTHWESTERN

#### Active

- CHOCK, RONALD E.—Midwest Paint Co., Minneapolis, MN.
- NEUBURGER, NEAL A.—Sonneborne Building Products, Edina, Minn.
- REGAN, JOHN F.—Contech Inc., Minneapolis, Minn.
- REUSCH, JANET S.—Whittaker Corp., Minneapolis.

#### Associate

- HAIGHT, THOMAS-Union Chemical, Minneapolis, Minn.
- LILJEGUIST, ROGER L.—Union Chemicals Co., Minneapolis, MN.

#### PITTSBURGH

#### Active

- MARTIN, MICHAEL J.—PPG Industries, Inc., Pittsburgh, PA.
- O'BRIEN, ROBERT-Mobil Chemical Co., Pittsburgh.
- TRAFICANTE MICHAEL A. III, PPG Industries, Inc., Springdale, PA.

#### Associate

POPE, CHARLES A. JR.—Olin Corp., Mc-Murry, PA.

#### PHILADELPHIA

#### Active

- COFFMAN, ROBERT E.—M.A. Bruder & Sons, Philadelphia, Pa.
- KENWORTHY, CRAIG—Clement "Coverall" Co., Camden, N.J.

#### Associate

BEVITZ, LAIRD S.—Cyprus Industrial Min. Co., Belmeade, N.J.

- DOMEN, VICTOR J.—Metro Oil & Chemical Corp., Medford, N.J.
- FISHER III, BARRETT C.—General Electric Co., Trevose, Pa.
- LOWE JR., JACK W.-Eastman Chemical, Kennett Square, Pa.
- MELORE, PAUL J.-Ebonex Corp., Hamilton Square, Pa.
- SMITH, MICHAEL J.—Pioneer Salt & Chemical, Philadelphia, Pa.
- TURNER, JANE M .- ASTM, Philadelphia.

#### Retired

STACKHOUSE, WARD-Boca Raton, Fla.

#### ST. LOUIS

#### Active

- CUNNINGHAM, ROBERT D. Dennis Chemical Co., St. Louis, Mo.
- FOY, WILLIAM L.—LanChem Corp., E. St. Louis, Ill.
- GRUBBS, CHARLES L.—Rockford Chem. Coatings, St. Louis, Mo.
- HINDEN, EDWIN R.—Thomas & English St. Louis.
- LUDWIG, BARBARA E.—Carboline, St. Louis. MACDOUGALL, PAUL—Kyanize Paints, Inc.,
- Springfield, Ill. SCHUERMAN, GEORGE E.-Kyanize Paints,
- Inc., Springfield. SOKOLOWSKI, SAM-Sinnett Lacquer Mfg.,
- St. Louis. WROBEL, JOSEPH J., JR.-CIBA-GEIGY
- Corp., St. Louis.

#### Associate

- ASHEY, EDWARD P.—Tioxide America, Inc., Ballwin, Mo.
- ERNST, ANNE M.—Sun Chemical Corp., Creve Coeur, Mo.
- PHILIPS, HERMAN G.—Hercules, Inc., Kirkwood, Mo.
- REYNOLDS, PHILLIP-Byk-Mallinckrodt, Mehlville, Mo.

#### TORONTO

#### Active

- BOBKER, R.V.—Interprovincial Corrosion Control Co. Ltd., Burlington, Ont. Can.
- BUMANLAG, REYNALDO G. Tremco (Canada) Ltd., Toronto, Ont. Can.
- CURRIE, CATHERINE M.—Home Hardware, Burford, Ont. Can.
- KLAUS, MUELLER-Superex Canada Ltd., Willowdale, Ont. Can.
- NG, SIU H.-Tremco (Canada) Ltd., Toronto. PENNEY, JOSEPH A.-Almatex Coatings,

SHAO, PETER-Tremco (Canada) Ltd.,

STONE, GEORGE L.-G.L. Stone Enter-

WIERSMA, DONALD S.-Celanese Canada

Journal of Coatings Technology

prises, Mississauga, Ont. Can.

London, Ont. Can.

Scarboro, Ont. Can.

Inc., Mississauga.

### **Technical Articles in Other Publications**

#### Compiled by the Technical Information Systems Committee-H. Skowronska, Chairman

#### Deutsche Farben-Zeitschrift (Defazet) (in German)

Published by Edition Lack und Chemie, Elvira Moeller GmbH, Karl-Benz-Strasse 11, Postfach 11 68, 7024 Filderstadt 1.

Vol. 33 No. 12

Dec. 1979

- Stange, R., Klug, L., and Klug, H.—The Problem of the Determination of Stable Compounds in Amino Resins, Reported in the Instance of Melamine Resins"; 426-433.
- Ostertag, W., Ebenhöch, F., Bittler, K., and Wunsch, G.—"Transparent Iron Oxide Reds as a Result of Iron Carbonyl Combustion"; 434-435.
- Müller, W.—"Origin of Building Structural Damage Owing to Failure to Consider Water Vapor Diffusion"; 435-437.
- Kondrachoff, M.W.—"Lead Chromate—Pigment of the Future"; 437-440.

#### Double Liaison (in French)

Published by EREC, 68 rue Jean-Jaures, 92800 Puteaux, France

Vol. 27 No. 293

Jan.-Feb. 1980

- Pichot, C.—"Influence of the Emulsion Polymerization Process on Some Colloidal Properties of Latex Copolymers (Vinyl Acetate (V.A.)—Butyl Acrylate (B.A.))"; 19-28.
- Thol, Miss and Kaeuffer, J.L.—"A New Possibility of Aqueous Media Pigmentation"; 30-34.

#### Farbe und Lack (in German)

Published by Curt R. Vincentz Verlag, Schiffgraben 41-43 3000 Hannover 1, Postfach 6247, Germany

Vol. 86 No. 3

March 1980

- Möhler, H.—"Quantitative Differential Microcalorimetric Investigations into the Curing of Varnish Systems. (1) Basic Principles"; 211-215.
- Koch, W.A.—"Problems in Painting Aluminium Alloys"; 216-218.
- Christensen, G. and Mork Pedersen, C.—"Unintended Chemical Reactions in Varnishes"; 219-223.
- Kirchmayr, R., Berner, A. and Rist, G.—"Photoinitiators for UV Curing of Paints"; 224-230.
- Brushwell, W.—"Contributions by Lacquer Technology to Environmental Protection and Safety at Work"; 230–235. (Literature review)

#### Vol. 86 No. 4 April 1980

- Zorll, U.—"Rheological Terms for Characterizing the Flow Behaviour of Lacquer Materials"; 301-307.
- Buter, R.—"Structure-Dependent Properties of High-Solids Polyester Lacquers"; 307-312.
- Henig, A., Jäth, M., and Möhler, H.—"Quantitative Differential Microcalorimetric Investigations into the Curing of Varnish Systems. (2) Epoxy Resin-Based Powder Paints"; 313-319.
- Brushwell, W.-."Ways of Saving Energy in Paint Technology"; 320-321.
- German Standard Draft DIN 53241 Part 1: Determination of Iodine Value with Solution According to Wijs; 322-324.
- German Standard Draft DIN 55940: Binders for Paints and Varnishes; Dehydrated Castor Oil; Technical Delivery Specifications; 325-328.
- German Standard Draft DIN 55952; Binders for Paints and Varnishes; Cellulose Ether, Methods of Test; 329-333.
- German Standard Draft DIN 55962: Binders for Paints and Varnishes; Distilled Dehydrated Castor Acid, Technical Delivery Specifications; 334-336.

#### Smith, A.—"Review of Lacquering"; 12-14.

Scott, J.— Toes Correlation Exist between Accelerated and Conventional Outdoor Exposures? Part I"; 15-18. (Paper presented at European Coil Coatings Association Conference, Brussels, December 1979)

**Pigment & Resin Technology** 

Published by Sawell Publications Ltd.,

127 Stanstead Road, London SE23 IJE.

#### Progress in Organic Coatings (in French, German or English)

Published by Elsevier Sequoia S.A., P.O. Box 851, 1001 Lausanne, Switzerland

Vol. 8 No. 1

Vol. 9 No. 2

Sato, K .- "Hardness of Coating Films"; p. 1.

Nakamichi, T.—"Behavior of Thermosetting Powder Coatings During the Curing Process"; p. 19.

Zosel, A.- "Mechanical Behaviour of Coating Films"; p. 47.

Kamarchik, P. and Cunningham, G.P.—"Applications of X-ray Techniques to Coatings Analysis"; p. 81.



1980

February 1980

### "Talks Available" Booklet Offers 43 Presentations

The 1980-81 listing of "Talks Available for Constituent Societies" contains a total of 43 presentations which are available for the upcoming meeting season. Divided into seven subject headings, the booklet presents 20 new titles.

The list has been compiled and distributed to the Societies by the Federation's Public Relations Committee, chaired by Harry Poth, of Burk-Hall Co., Memphis, TN. Available from Federation headquarters, the booklet includes for each presentation: (1) Title; (2) Name of speaker; (3) Company affiliation, address, and telephone; (4) Geographic areas where talk is available; (5) Equipment needed; (6) Abstract; (7) Biographical sketch of speaker; and, if applicable, (8) Where and when talk has previously been given.

The following presentations are listed:

#### Additives

"Flash Rust Inhibition in Water-Based Paints"—Drew Chemical Corp.

"New Approaches to Foam Control in Aqueous Industrial Coatings"—Drew Chemical Corp.

"Use of Titanate Coupling Agents in Coatings"—Kenrich Petrochemicals, Inc.

"Driers for Water-Borne Coatings"-Tenneco Chemicals, Inc.

"Latex Paint Spoilage vs. Plant Housekeeping"—Tenneco Chemicals, Inc.

"Microbiological Problems Associated with Water-Reducible Industrial Coatings"—Troy Chemical Corp.

#### **Colors and Pigments**

"Efficient Formulating Techniques for the 80's"—Burgess Pigment Co.

#### Southern Society Schedules Annual Meeting, March 11-13

The 45th Annual Meeting of the Southern Society will be held from Wednesday, March 11, through Friday, March 13, at the Plaza Hotel South in Birmingham, AL. Presentations at the meeting will focus around the theme, "Coatings Technology—The End Users Perspective." Several panel presentations will be featured, as well as short discussions on practical problems in using water-borne coatings and on paint quality as it enhances the salability of products.

The symposium is also expected to include technical presentations covering such topics as high solids, solvent-based coatings, and cosolvents in water-borne "Colorant Formulation: Pigment Selection"—CIBA-GEIGY Corp.

"Development of Computer Selected Blends"—CIBA-GEIGY Corp.

"Chlorite: A New Extender Pigment for the Coating Industry"—Cyprus Industrial Minerals Co.

"Talc-That Crazy White Stuff"-Cyprus Industrial Minerals Co.

"Factors Governing Tinter Performance"—Daniel Products Co.

"Extenders, the Inorganic Backbone of Flats and Primers"—Engelhard Minerals & Chemicals Corp.

"New Formulating Parameters in the Switch from Bag to Slurry Clay"— Engelhard Minerals & Chemicals Corp.

"Polymeric Organic Dispersants for

Pigments—Principles and Practices" —Swedlow, Inc.

#### Production

"The 'Whys' and 'Wherefores' of Cartridge Filtration in the Coatings Industry"—AMF Cuno Div.

"Dispersion and Mixing"-Hockmeyer Equipment Corp.

"Plant Design—Build New—Redesign Old"—Hockmeyer Equipment Corp.

"Slurry-The New Way"-Hockmeyer Equipment Corp.

"Particle Size Reduction Techniques" -Netzsch Corp.

"The Sandpiper"—The Warren Rupp Co.

#### Resins

"High Solids Coatings, A Smorgasbord Approach"—Cargill, Inc.

"Maintenance Paints with Chlorinated Rubber"—ICI Americas, Inc.

-ICI Americas, Inc. -Byk-Ma

systems. Two presentations on trade paint quality and a discussion of trends in metal finishing have been scheduled.

The Planning Committee is chaired by Peter Decker, of Union Carbide Corp. Members include Frank Rector, of Indurall Coatings; Oliver Cline, of SCM Corp., Div. of Glidden; Dan Dixon, of Freeport Kaolin; William Early, of Piedmont Paint; Len Kustra, of Southeastern Steel Container; Bobby Moore, of Interstate Paint; Ron Nelson, of Kelco Corp.; and Pat Perkins, of Ashland Chemical.

For additional information, contact Peter Decker, Union Carbide Corp., Coatings Materials Div., 17 Executive Park Drive, NE, Atlanta, GA. "Design Considerations for High Solids Reactive Coatings"—Monsanto Plastics & Resins Co.

"New Developments in the Formulation of Gloss Latex Paints"—Pacific Scott Bader Inc.

"Conversion Enamels—Energy Savers"—Reichhold Chemicals, Inc.

"Advances in Acrylic Latex Maintenance Paints"-Rohm and Haas Co.

"An Introduction to Formulating Latex Paints (Anatomy of a Latex Paint)" -Rohm and Haas Co.

"Telechelic Polymers—Precursors to High Solids Coatings"—Swedlow, Inc.

#### Solvents

"Economic Recovery of Solvent Vapors"-DCI Corp.

"Recovery of Paint Wash Solvent— An Economic and Environmental Necessity"—DCI Corp.

"Solvent Recovery System Based on a Wiped Film Evaporator"—The Pfaudler Co.

"Computer Prediction of Evaporation of Aqueous Solvent Blends with Any Number of Cosolvents at Any Humidity" —Shell Development Co.

"Computer Selection of Solvent Blends"—Shell Development Co.

#### Testing

"Accelerated Weathering—Past, Present, and Future"—Atlas Electric Devices Co.

"Natural Outdoor Testing"—Atlas Electric Devices Co.

"Sedimentation Analysis as a Method for Early Determination of the Formation Settlements in Coatings Systems" —Byk-Mallinckrodt Chem. Prod. BmbH.

"Exposure Evaluation: Quantification of Changes in Appearance of Pigmented Materials"—CIBA-GEIGY Corp.

"Exposure Evaluation: Part II-Bronzing"—CIBA-GEIGY Corp.

"Know Your Enemy—The Weather" —The Q-Panel Co.

"An Investigation of Abrasion Resistance"—Shamrock Chemicals Corp.

#### Environmental

"Effective Treatment of Paint Waste" — Drew Chemical Corp.

"Toxicity"-H. Kohnstamm & Co., Inc.

"Environmental Update"-Sun Chemical Corp.

#### **Rolla Adds New Courses to Fall Schedule**

The series of Paint Short Courses sponsored by the University of Missouri—Rolla has expanded to include in its Fall schedule two new courses, "Techniques and Mechanics of Selling Specialty Coatings," and "Managing for Innovations in Coatings." The courses are scheduled to be presented in St. Louis September 22-23 and September 24-25, respectively.

Additionally, the workshop, "Women in Coatings: Meeting the Challenges," will be held in Atlanta October 26-28.

The remainder of the Fall schedule is as follows:

(Aug. 18-22) Composition of Paint and Coatings. Fee: \$275.

#### NACE Corrosion Courses Scheduled for September

The National Association of Corrosion Engineers (NACE) has scheduled several continuing education courses for September 1980.

The first seminar will be held at the Tyson's Corner Holiday Inn, McLean, VA, on September 7-12. The NACE Basic Corrosion Course and the Corrosion Prevention by Cathodic Protection Course will be offered.

Chicago will be the site of the second seminar on September 14-19. The Corrosion Prevention Course will be presented.

A third seminar will be held at the University of Alabama, in Tuscaloosa, on September 21-26. The NACE Basic Corrosion Course will be offered.

Four courses will be held concurrently in Houston, September 21–26, at the AstroVillage Hotel. The seminar will include the NACE Basic Corrosion Course, the Corrosion Prevention by Cathodic Protection Course, the Corrosion Prevention by Coatings Course, and the Corrosion Prevention in Oil and Gas Production Course.

Corrosion Prevention by Cathodic Protection will also be offered September 28-October 3, 1980, in Philadelphia at the Marriot Motor Lodge. This course will be held in conjunction with the Liberty Bell Corrosion course. The Liberty Bell seminars will also include: Principles of Corrosion, Marine Conference, Protective Coatings and Linings, and Water Treatment.

Registration fee for each is \$400 for NACE members and \$475 for nonmembers.

For additional information, contact the Education Dept., NACE Headquarters, P.O. Box 218340, Houston, TX 77218. (Aug. 25-29) Basic Quality Control. Fee: \$350.

(Sept. 8-12) Introduction to Formulation. Fee: \$350.

(Sept. 15-19) Advanced Paint Formulation. Fee: \$350.

(Nov. 18-20) Job Estimating Workshop for Contractors. Fee: \$350.

For additional information, or to register, contact Norma Fleming, Extension Div., Univ. of Missouri-Rolla, 501 W. 11th St., Rolla, MO. 65401; or, for technical information, contact the Course Director, John A. Gordon, Jr., Chemistry Dept., Univ. of Missouri-Rolla, Rolla, MO.

#### Western Coatings Societies Update 15th Biennial Symposium

The Western Coatings Societies will present their 15th Biennial Symposium and Show on March 4-6, 1981, at the Disneyland Hotel and Convention Center, Anaheim, CA.

Twenty papers of technical interest, as well as three workshops have been planned. In addition, 35 exhibitors have reserved 50 show booths.

Industry suppliers interested in participating in the show are urged to contact Exhibit Chairman, Earl B. Smith, 11911 S. Woodruff Ave., Downey, CA 90241.



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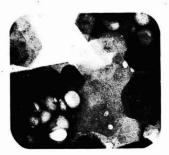
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FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY 1315 Walnut Street Philadelphia, Pennsylvania 19107

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I am a member of the	Society for Coatings Technology.

#### First-Half Contributions Boost 1980 PRI Fund

Contributions for the 1980 research programs of the Paint Research Institute were received at the Federation headquarters during the first six months of this year. These supplement funds committed by the Federation to PRI.

Contributors (to June 30, 1980) include the following:

#### Societies

Golden Gate; Montreal; Pacific Northwest; Southern.

#### Corporations

The M.F. Cachet Co.; Canada Color & Chemicals Ltd.; Canadian General Electric Co. Ltd.; Celanese Canada, Inc.; Chemcentral; CIL Paints, Inc.; Cook Paint & Varnish Co.; Dar-Tech, Inc.; Davies Can Co.; Dexter Midland Div.; Dominion Colour Co. Ltd.; The Dow Chemical Co.; Drew Chemical Corp.; Ferro Corp.; Grund Associates; Harshaw Chemical Corp.; Inmont Canada Ltd.; International Minerals & Chemicals Corp.; Jamestown Paint & Varnish Co.; Kalcor Coatings Co.; David Litter Labs, Inc.; L.V. Lomas Chemical Co. Ltd.; Lord Corp.; Mameco International; Mobil Chemical Canada; Monsanto Co.; Benjamin Moore & Co. Ltd.; Nacan Products Ltd.; Palmer Supplies Co.; Pfizer, Inc.; Q-Panel Co.; Rohm and Haas Co.; Seegot, Inc.; Shell Chemical Co.; Silberline Mfg. Co., Inc.; Donald McKay Smith, Inc.; Tioxide of Canada Ltd.; Union Carbide Corp.

#### **Foundations**

The N/L Industries Foundation; The Sherwin-Williams Foundation.

#### Associations

Ontario Paint Association.

#### Individuals

James W. Broggini; Everett B. Euchner; David M. Gans; Raymond R. Myers; E. William Henschel.

#### **Mildew Consortium**

Troy Chemical Corp.

#### **Pigment Dispersion is Topic of Cleveland October Symposium**

The Manufacturing Committee of the Cleveland Society will present a symposium on "Formulation for and Utilization of Pigment Dispersion Equipment" at the Cleveland Scientific & Engineering Center on October 7.

Scheduled for presentation are the following:

"Pigment Dispersion and Grinding— An Overview"—Michael W. Malaga, of Glidden Coatings & Resins.

"High Speed Dispersers"—James White, of Hockmeyer Equipment Co.

"Titanium Dioxide: a Pigment for High Speed Dispersion"—Calvin C. Tatman, of Glidden Pigments.

#### Call for Papers Issued By Electrochemical Society

A symposium on "Adhesion Aspects of Polymeric Coatings" will be held at the Electrochemical Society Meeting in Minneapolis, MN, on May 10-15, 1981.

The program will comprise both stateof-the-art invited overviews and contributed original research papers. Papers dealing with any of the above or allied topics are solicited.

All inquiries should be directed to Dr. K.L. Mittal, Symposium Chairman, Dept. 49F, Bldg. 300-40E, IBM Corp., Hopewell Junction, NY 12533. "Small Media Mills and Milling"— Leo Dombrowski, of Chicago Boiler Co.

"Organic Pigments for Small Media Mills"—Fred W. Scheidegger, of CIBA-GEIGY Corp.

"Large Media Mills"—William L. Callahan, of Kerr Engineered Sales Co.

"Carbon Blacks: Pigments for Large Media Mills"—Victor Lewis, of Cabot Corp.

#### Southern Society Adds Memphis Section; Nominating Committee Appointed

As a result of a May 6, 1980, meeting of the Southern Society for Coatings Technology, a Memphis Section has been organized. The Society is comprised of four additional Sections: Atlanta, Central Florida, Miami, and Gulf Coast.

Proceedings of the meeting were announced by Society President, A.L. Hendry, of A.L. Hendry & Co. Harry Poth, of Burk-Hall Co., will serve as Temporary Chairman and Brian L. Budzien, of United Paint Co., will act as Temporary Secretary-Treasurer.

A Nominating Committee consisting of Stan Hirshfield, of United Paint Co.; Tully Lynch, of Delta Industries; and Dick Hirt, of Burk-Hall Co., was appointed to select officers for the year.



#### (Continued from page 86)

It should be remembered that PVC and CPVC are not the only factors determining the porosity of a flat latex paint film. The binding power index also plays an important part, as may be illustrated by the following equation from my 1967 Mattiello Lecture (*J. Paint Tech*nology, 39, No. 515, 703):

$$L.P. = 1 - \frac{CPVC (1 - PVC)}{PVC (1 - CPVC)} x$$
  
where L.P. = latex porosity

CPVC = pigmentation CPVC from oil absorption PVC = formula PVC x = binding power index

Given three latex vehicles with binding power indexes of 0.80, 0.70, and 0.60, pigmented with the same pigmentation at the same PVC (above the pigmentation CPVC), three uniformly spaced levels of high dry-hiding will be produced, with the highest dry-hiding associated with the lowest binding power index.

Since the polystyrene bead dispersion might be described as a latex with a binding power index of zero, might not its effect upon high dry-hiding be the result of depressing the average binding power index of the vehicle system? This would eliminate the violence to conventional formulating concepts created by speaking of a low-refractive-index organic polymer as "pigment."

The effective binding power index of a latex/polystyrene-bead blend would probably not be a simple average, due to particle-size disparities, but it should be a simple task to identify the true relationship using my modification of Schaller's contrast-ratio technique (J. Paint Technology, 42, No. 545, 329) to identify the PVC of zero porosity for the same extender dispersed in a series of blends of varying composition.

If Dr. Ramig's "plastic pigment" is indeed providing effects that are unlike those of mineral extenders of similar particle size (there is as yet no published data to that effect), the most likely explanation would seem to be that it is not a pigment, but a nonfilm-forming portion of the vehicle system. This, of course, does not detract from its ability to enhance the optical efficiency of titanium dioxide, which can be a valuable contribution if employed to improve or retain quality, rather than to merely lower cost.

> FRED B. STIEG Pigmentech Consulting Jekyll Island, GA



Leon Kutik has been appointed Director of Research and Technical Operations for the Coatings Group of Dutch Boy, Inc., Baltimore, MD. In his new position, Mr. Kutik will have direct responsibility for all trade, traffic, and industrial laboratory operations within the Coatings Group. He serves on the Federation's Publications Committee Editorial Review Board.

Ernest W. Hughes has been named to the newly created position of Manager, special products, for Ferro Corp., Coatings Div. In this position, Mr. Hughes will be responsible for directing the marketing, technical support, and research and development programs for specialty products.

William J. Hook has been promoted to the position of Senior Project Chemist in the Special Products Laboratory of the Midland Div., Dexter Corp. He formerly served as Technical Service Representative and was assigned to product development projects.

Freeport Kaolin Co., New York, NY, has appointed **Mack L. Williams** Technical Service Manager. In this position, he will be responsible for all technical service activities in the paper, paint, and polymer areas for the company.

Thomas P. Moran, a Director for the O'Brien Corp., has retired after 40 years in the paints and coatings industry. A member of the Baltimore Society, Mr. Moran was Vice-President and General Manager of the company's operation in Baltimore, and Group Vice-President, responsible for the firm's Baltimore and Brunswick, GA operations.

Meanwhile, Robert E. Dermody has joined the O'Brien Corp. as an Area Manager in the company's midwestern region. He will report to Douglas W. Miller, Regional Manager, South Bend.

Charles E. Lowden has been named National Sales Manager of Day-Glo Color Corp., Cleveland, OH. He will assume responsibility for the firm's domestic sales.

In addition, Jim Skelly has been promoted to Regional Sales Manager. In his new position, Mr. Skelly will be responsible for sales, management, and training in an area including Ohio and five surrounding states.



Harry Poth has joined the Memphis

based Burk-Hall Co. as Trade Sales

Director of Technical Services. He will

work with the firm's sales force on

product development, special coatings

problems, and customer technical

paint industry, Mr. Poth most recently

served as Technical Director of Dean &

Barry Co. in Columbus, OH. He is a

Past-President of the C-D-I-C Society.

former Treasurer of the Federation, and

an honorary Trustee of the Paint

Polychrome Corp. has named Steven

Mandel to the position of Technical Sales

Representative for its industrial chemical

subsidiaries, Cellomer Corp., Newark,

NJ, and Cellofilm Corp., Wood-Ridge,

In a series of managerial appoint-

ments, Glidden Coatings & Resins, Div.

of SCM Corp., has named Michael E.

George Plant Manager at its Columbus,

GA plant. Jay F. Gross will succeed Mr.

George as Manager of the Charlotte, NC

Glidden Pigments as a Sales Representa-

tive for the Illinois and Wisconsin areas.

Meanwhile, Ed Croco has joined

Dr. F. Thomas Krotine has joined the

Sherwin-Williams Co. as a Senior Vice-

President of research and development.

He will report directly to John G. Breen,

Chairman, President, and Chief Execu-

Howard S. Tokarsky has been named

Operations Manager, Los Angeles, for

The Harshaw Chemical Co.'s Color

Dept. In this position, he will be

responsible for the manufacturing of the

firm's universal colorants at the Sylmar.

CA, facility. He is a member of the Los

With 31 years of experience in the

L. Kutik

Research Institute.

NJ and Chicago.

powder coatings plant.

tive Officer.

Angeles Society.

service

H. Poth

9



S. Mandel

A. Ingoglia

Universal Color Dispersions has appointed **Augie Ingoglia** to the position of National Sales Manager. A member of the Philadelphia Society, he will be transferring his affiliation to the Chicago Society.

Valspar Corp., Minneapolis, has elected four new corporation officers: Christian P. Bosset—Vice-President, Consumer Brands; Duane R. Syvrud— Corporate Vice-President and President, Colony; Donald Ochs—Vice-President, Farm and Industrial Coatings; and Vincent J. Greci—Vice-President, Product Finishes.

International Minerals & Chemicals Corp. has combined the company's Industrial Minerals and Foundry Products Div. to form the IMCORE Div. within the firm's Industry Group. Lyle A. Holmes will serve as Vice-President and General Manager of the IMCORE Div. Named as IMCORE's Vice-President of Operations is Donald H. Freas.

Meanwhile, **Dr. Morton Blakeman** Ingle has been elected a Vice-President of International Minerals & Chemicals Corp. He will be responsible for all IMC business groups, sponsored research with universities, and research work performed by consultants or by outside commercial facilities under contract.

John Wilkening has been promoted to the newly-created position of Southwest District Sales Manager for the U.S. Paint Div. of Grow Group, Inc., St. Louis, MO. He will represent U.S. Paint in Arkansas, Louisiana, New Mexico, Oklahoma, and Texas.

Richard Van Buren has been appointed Director of Marketing for the J.M. Huber Corp., Clay Div., Macon, GA. Mr. Van Buren joined the company in 1956 and has held various technical service and sales positions.

Journal of Coatings Technology

Dr. Richard Nathan has been named Manager of the newly-created Hazardous Materials Program Office of Battelle Columbus Laboratories. Previously, he led an international study team that analyzed the status of toxic materials control technology and related activities.

Other key personnel in the new office are Dr. Gary L. McKown, Theodore M. Prociv, and Rebecca J. Porter, all of whom have been involved with a wide variety of toxic materials control.

Harold F. Cole has been promoted to Supervisor of Packaging Closure Group of the Midland Div., The Dexter Corp. In addition, David G. Jewell has been promoted to Group Leader in the Packaging Products Developmental Laboratory of the firm. Both men are members of the Chicago Society.

Meanwhile, **Thomas M. Daly** has joined the staff of the Midland Div. as Manager of Analytical Chemistry

Bennett's Glass & Paint, Salt Lake City, has appointed John W. Nielson, Jr. Executive Vice-President. He will supervise and coordinate production, warehousing, and services for glass and paint. Mr. Nielson is a member of the Rocky Mountain Society.

G. Dale Ernst, a member of the Northwestern Society, has been named Principal Scientist of Tennant Co. He will report directly to Don Thomsen, Director of Research. Mr. Thomsen is also a member of the Northwestern Society.

The C.J. Patterson Co., Kansas City, has appointed Alan J. Dankwerth to General Manager of the Specialty Chemical Div. In this capacity, he will be responsible for the coordination, development, and implementation of plans for all chemical operations. He is a member of the Kansas City Society.

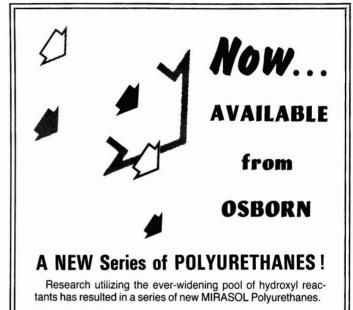
Ed Zahora has been promoted to Research Chemist for DeSoto, Inc., Des Plaines, IL. He is a member of the Chicago Society.

In addition, E.L. Jones has been named Market Manager for Business Products—Toners and Resins for the firm. Mr. Jones will be responsible for marketing resins and toners for the international office copy market.

In a series of appointments, the Petrochemical Group, Union Chemicals Div., Union Oil of California, has named Billy J. Morris—Product Manager, petrochemicals; Frederick S. Bartholomew—General Manager of the midwestern region; and James W. Cawley— National Accounts Manager. In addition, Allen V.A. Moon has been promoted to Sales Manager of the company.

Dr. James F. Roth has joined Air Products and Chemicals, Inc. as Chief Scientist, reporting to J. Robert Lovett, Vice-President of Research. Dr. Roth will direct a Science Center being formed by the company. Robert L. Webb, Vice-President of Union Camp, has been elected President of the Pulp Chemicals Association. He succeeds Stanley Fenelon, of Hercules, Incorporated. Elected to the post of Vice-President was Stuart I. Bergman, of Gilman Paper Co.

Elected members of the Executive Committee are: William R. Laidig, of Great Southern Paper Co; Richard E. Lodmill, of Weyerhaeuser Co.; Peter J. Rinkevich, of Champion International; Peter Rosasco, of Reichhold Chemicals, Inc.; Joseph A. Schmidlein, of Arizona Chemical Co.; and Stanley Fenelon, of Hercules, Incorporated.



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## Letters to the Editor

Are Polystyrene Beads Pigment?

Dr. Ramig's challenge in the April

issue, (J. Coatings Technology, 52, No. 663, 95-96) for me to cite some reference

to establish the ability of a conventional

pigment to produce a semi-gloss lustre in

a high-PVC, porous coating, is one that I

cannot meet in terms of any published

reference. Although academic research

may tend to report any finding, irrespec-

tive of any practical application, this

often is not true of applied research. As

director of technical service activities for

many years with the Titanium Pigment

Corporation, I made a number of obser-

vations that did not find their way into

Among these was the impractical ob-

servation that a high-gloss enamel with

all of the porosity of a blotter could be

produced by pigmenting a normal archi-

my published work.

TO THE EDITOR:

### CLASSIFIED **ADVERTISING**

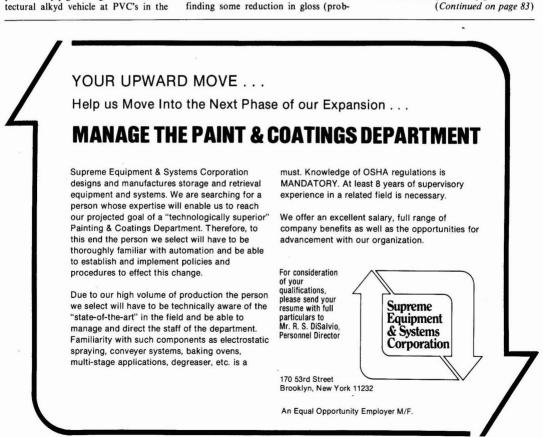
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ably a refractive index effect), but nothing quite as low as a semi-gloss.

There is, however, some question in my mind as to the validity of the high PVC's referred to by Dr. Ramig. While he has attempted to establish the convention of treating his polystyrene beads as "pigment" in PVC calculations, presumably in recognition of their ability to promote film porosity and high dryhiding, their solubility in hydrocarbon solvents and heat-softening characteristics make them obviously more closely related to the latex vehicle polymer particles than to any known pigment.

(Continued on page 83)



range of which Dr. Ramig speaks. This is

not too surprising, since the pigment

contains no particles large enough to

destroy the optical planinity of the sur-

face, and its high refractive index con-

tributes to a higher gloss level than would

be possible for either a mineral or

plastic extender. Gloss actually increases

as the pigmentation is raised high enough

for the resin film over the pigment parti-

cle to become so thin that the optical

effect of a pigment/resin interface is com-

Obviously, such a discovery is imprac-

tical because of the cost of pigmenting

with pure titanium dioxide at so high a

level, as well as the questionable demand

for a porous enamel. Nevertheless, I did

experiment with the possibility of replac-

ing some of the titanium dioxide with an

ultra-fine calcium carbonate extender,

finding some reduction in gloss (prob-

pletely lost.

Journal of Coatings Technology

## **Coming Events**

#### FEDERATION MEETINGS

#### 1980

(Oct. 28)—Federation Board of Directors Meeting. Hyatt Regency Hotel, 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." Executive West, Louisville, KY. 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).

(May 14–15)—Spring Meetings. Society Officers on 14th; Board of Directors on 15th. Hilton Hotel, Denver, CO. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Oct. 28–30)—59th Annual Meeting and 46th Paint Industries' Show. Cobo Hall, Detroit, MI. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

#### 1982

(Apr. 22–23)—Spring Meetings. Society Officers on 22nd, Board of Directors on 23rd. Sheraton Hotel, Boston, MA. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Nov. 3-5)—60th Annual Meeting and 47th Paint Industries' Show. Sheraton Washington Hotel, Washington, D.C. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

#### PAINT RESEARCH INSTITUTE MEETING

(May 4–5)—Paint Research Institute Symposium on "Stability and Stabilization of Coatings Systems," Battelle Memorial Institute, Columbus, Ohio. (Dr. Raymond R. Myers, Chemistry Dept., Kent State University, Kent, Ohio 44242).

#### SPECIAL SOCIETY MEETINGS

#### 1980

(Sept.17-18)—Montreal and Toronto Societies Joint Symposium on "Corrosion." 17th in Montreal; 18th in Toronto.

(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. (Richard C. Sutherland, E.T. Horn, Co., 16141 Heron Ave., La Mirada, CA 90638.)

(Mar. 11-13)—Southern Society. 45th Annual Meeting. Plaza South Hotel, Birmingham, AL (Peter F. Decker, Union Carbide Corp., 17 Executive Park Drive, N.E. Atlanta, GA 30359).

(May 1-2)—Pacific Northwest Society. Annual Symposium. Washington Plaza Hotel, Seattle, WA.

(May 9-Montreal Society. Fiftieth Anniversary dinnerdance. Montreal, Que.

#### 1982

(May 6-8)—Pacific Northwest Society. Annual Symposium. Vancouver, B.C.

#### **OTHER ORGANIZATIONS**

#### 1980

(Sept. 8-Nov. 21)—Paint Short Courses at University of Missouri-Rolla. 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 Felming, 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. 18–19)—Society of Plastics Engineers 4th Annual Decorating Plastic RETEC. Sheraton Center Hotel, Toronto, Ontario, Canada. (Stephen Kennedy, Binks Manufacturing Co., 9201 W. Belmont Ave., Franklin Park, IL 60131.)

(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, III. (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).

(Oct. 5-8)—Society of Plastics Engineers and Fire Retardant Chemicals Association Joint Conference on "Flammability in Building and Construction—Today and Tomorrow." Ponte Bedra Club, Ponte Vedra Beach, FL. (Fire Retardant Chemicals Assoc., 265 Post Road West, Westport, CT 06880).

(Oct. 7-9)—AFP/SME "Liquid Coatings Conference." Conrad Hilton Hotel, Chicago, IL. (Susan Buhr, Society of Manufacturing Engineers, One SME Dr., P.O. Box 930, Dearborn, MI 48128).

(Oct. 13–15)—Society of Plastics Engineers National Technical Conference, "Coloring of Plastics; Update of 'The Law and You'." RETEC sponsored by the Baltimore Section and Color and Appearance Div., Baltimore Hilton Hotel, Baltimore, Md. (Robert C. Foley, Society of Plastics Engineers, 656 W. Putnam Ave., Greenwich, Conn. 06830.)

(Oct. 13-17)—"High Performance Coating Procedures," Monterey, Mexico. (Institute of Applied Technology, Jean L. Kaplan, 1200 – 17th St., N.W., Suite 406, Washington, D.C. 20036.)

(Oct. 14–16)—Seventh Annual Conference on Energy, "Energy Future: Prophets, Profits & Policies!" University of Missouri-Rolla/Department of Natural Resources. (Dr. J. Morgan, Conference Director, 122 Electrical Engineering, University of Missouri-Rolla, Rolla, MO 65401.)

(Oct. 17–19)—University of Waterloo Short Course, "Developments in Polymer Technology." Hotel Toronto, Canada (Prof. A. Rudin, Dept. of Chemistry, University of Waterloo, Waterloo, Ontario, Canada).

(Oct. 27–29)—93rd Annual Meeting, National Paint and Coatings Association, Atlanta Hilton Hotel, Atlanta, Ga. (Karen Bradley, NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005.)

(Nov. 5-6)—Third Resins & Pigments Exhibition. Hotel Nikko, Dusseldorf, Germany.

(Nov. 7-9)—University of Waterloo Short Course, "Engineering Aspects of Polymer Systems." Hotel Toronto, Canada (Prof. A. Rudin, Dept. of Chemistry, University of Waterloo, Waterloo, Ontario, Canada).

(Nov. 10-14)—"High Performance Coating Procedures," Houston, Tex. (Institute of Applied Technology, Jean L. Kaplan, 1200 – 17th St., N.W., Suite 406, Washington, D.C. 20036.)

(Nov. 11-13)—4th International Conference of Paint Research Association, Excelsior Hotel, London Heathrow Airport. (PRA, Waldegrave Rd., Teddington, Middlesex TW11 &LD, England.)

(Nov. 17-Dec. 17)—Short Courses for the Chemical and Process Industries, The Center for Professional Advancement. "Water-Borne Industrial Coatings: Application"—Nov. 17-18; "Water-Borne Industrial Coatings: Chemistry and Formulation" —Nov. 19-20; "Organic Coatings Technology"—Dec. 3-5 and 15-17. Sheraton Motor Inn, East Brunswick, NJ (Rosanne Razzano, Dept. NR, The Center for Professional Advancement, P.O. Box H, East Brunswick, NJ 08816.)

(Nov. 19-21)—3rd Annual Western Plastics Exposition. Anaheim Convention Center, Anaheim, Calif. (Western Plastics Exposition, 1625 17th St., Santa Monica, CA 90404.)

(Nov. 21–23)—Annual National Decorating Products Association Convention and Show, H. Roe Bartle Convention Center, Kansas City, Mo. (NDPA, 9334 Dielman Industrial Dr., St. Louis, Mo. 63132.)

(Dec. 9–11)—Plant Engineering and Maintenance Show and Conference/West. Convention Center, Anaheim, CA. (Clapp & Poliak, Inc., 245 Park Ave., New York)

1981

(Jan. 21)—ASTM Symposium, "Regiments for Predicting Permanence of Decorative and Protective Surfaces." Orlando,

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(Apr. 6–10)—National Association of Corrosion Engineers "Corrosion/81." Sheraton-Centre and Hotel Toronto, Toronto, Ontario, Canada. (Conference Coordinator, NACE, P.O. Box 218340, Houston, TX 77218).

(Apr. 28-30)—OCCA-32. Oil and Colour Chemists' Association 32nd Annual Technical Exhibition. Alexandra Palace, London, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HAO2SF, England.)

(June 17-20)—Oil and Colour Chemists' Association Conference, "Alternative Technologies in Coatings." Stratford Hilton Hotel, Stratford-on-Avon, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HA0 2SF, England.)

(Sept. 20-25)—4th Congress of the Association Internationale de la Colueur, "COLOR 81." International Congress Centre (ICC), Berlin (West), Germany. (Prof. Dr. Heinz Terstiege, (AIC COLOR 81), Bundesanstalt fur Materialprufung (BAM), Unter den Eichen 87, D-1000 Berlin 45, Federal Republic of Germany).

(Oct. 13–15)—Association for Finishing Processes of the Society of Manufacturing Engineers. "Finishing '81" Conference and Exposition, "Economics, Compliance, and Energy." Cobo Hall, Detroit, MI. (William J. Yeates, Executive Director AFP/ SME, One SME Dr., P.O. Box 930, Dearborn, MI 48128.)

1982

(May 9-14)-XVIth Congress of FATIPEC, Brussels, Belgium.

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Color-matching Aptitude Test Set was created by the Inter-Society Color Council and sponsored by the Federation. It is in world-wide use as a means for estimating color-matching skill. The 1978 edition contains minor refinements over the previous editions (1944, 1953, 1964), and these bring it closer to the original ISCC plan, making it a still more successful tool for evaluating color-matching skill.

The basic aim of the Test is to provide an objective measure that will aid in determining an individual's ability for performing color-matching tasks accurately. Changes in a person's color-matching skill may occur over a period of time, improving due to training, experience, and motivation, or lessening when removed from practice, or as a result of health disorders. Retesting at regular intervals will provide an indication of any such changes in an individual's ability.

The Test is not designed to indicate or measure "color-blindness" (anomalous color vision). Special tests designed for this purpose should be used.

The Color-matching Aptitude Test Set consists of a carrying case, an easel on which are mounted 48 color chips, a dispenser which holds 48 matching chips, score sheets, and a scoring key.

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One panel is coated with a popular "solvent" oil modified urethane coating. The other with our new **water reducible** urethane: Spensol F.

The way they look is the way they perform: very much alike. Similar gloss, toughness, and resistance to water and chemicals. But Spensol F gives you **more**. Cleanup is easier, faster, and more convenient. And water is more available and less costly than solvent. And helps you meet government regulations. (By the way, Spensol F is on the left.)

To get our new brochure of WD Urethanes, please call your SK representative, or contact us at Spencer Kellogg Division of Textron Inc., P.O. Box 807, Buffalo, NY 14240.

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