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### September 1985

# JOURNAL OF COATINGS TECHNOLOGY

Annual Meeting & Paint Show Issue



Cervantes Convention Center St. Louis, Missouri October 7, 8, 9

Fiftieth Anniversary of the Paint Industries' Show

1985



# WE'RE PLEASED TO ANNOUNCE WE'VE FINALLY COME TO THE COMPETITION'S ATTENTION.

You'd think we just jumped up behind them and shouted, "Boo!" In fact, we've been out in the bright lights for over ten years showing paint manufacturers how UCAR Acrylics can help increase profits without sacrificing performance.

But for those ten long years, the competition didn't blink an eye. Now, with over 140 paint makers using UCAR Acrylics, they've finally decided to respond to our success.

Well, better late than never, we say. Because healthy competition can only benefit the paint maker.

Just remember when you're evaluating those benefits, the cost/performance advantages of UCAR Acrylics are based on lower raw material costs. That means the savings we bring to you are real and permanent.

To find out how you can make quality paints at reasonable costs, contact your local UCAR Emulsion Systems Representative. Or write to us at Dept. K3442, 39 Old Ridgebury Road, Danbury, CT 06817.

We think you'll find it will take more than a bad fright before anyone can do as much for your product line as we can.



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Solution to August Puzzle

# **♥RPA**

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#### Thank You, Exhibitors

The biggest list of exhibitors ever in the biggest Paint Industries' Show ever appears in the special convention section in this issue.

There are 240 exhibitors in 56,300 net square feet of space—a brilliant pair of statistics that adds even more glow to the Golden Anniversary of the Paint Show which the Federation celebrates this year.

And here's some more glowing numbers:

- Three exhibitors have been in the Show all 50 years (Columbian Chemicals Co., Rohm and Haas Co., Union Carbide Corp.).
- Another 43 companies have been in the Show from 25-49 years.
- Fifty-two exhibitors are new in 1985.

The first Paint Show in Washington, D.C. featured 25 exhibitors in 1,000 sf. With that kind of growth in both size and popularity, it is easily understood why the Paint Show has become the biggest and best exhibit serving the coatings manufacturing industry.

And who have been primarily responsible for the great success of the Paint Show? THE EXHIBITORS—the hundreds of suppliers of not only raw materials, equipment, and services, but dispensers of knowledge and assistance as well.

The Federation will recognize their partnership role at the opening session of the Annual Meeting and Paint Show on Monday, October 7, at the Convention Center in St. Louis.

On behalf of the Federation, the industry, and the thousands of people who have walked the aisles of the Paint Show, thank you, exhibitors, for your long friendship, support, and contributions to the advancing technology of the coatings manufacturing industry.

Searl & Danech

Frank J. Borrelle, Executive Vice-President

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HEA and HPA are chemical intermediates useful in the synthesis of coatings resins, latexes, adhesives, radiation-cure oligomers, and other resins. These monomers are not recommended for use as reactive diluents in radiation coatings or as simple additives to other formulations, unless appropriate measures are taken to control vapors and minimize personnel exposure within recommended guidelines. For complete information, write: The Dow Chemical Company, Coatings and Resins Department, Inquiry Services, 1703 S. Saginaw Road, Midland, Michigan 48674.



See us in Booth 617, St. Louis-1985 Paint Show



#### **Product Type**

#### ADHESIVES

Pressure-sensitive Adhesives

Contact Adhesives

Vinyl Film Lamination

INDUSTRIAL COATINGS-WATER-BORNE

Latex Latex

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#### INDUSTRIAL COATINGS-SOLVENT

Urethane Lacquer

Low-molecular-weight Acrylate Resins Automotive Enamels

INDUSTRIAL COATINGS-HIGH SOLIDS

High-Solids Enamel—Automotive Finishes

High-Solids Paint Composition

Dual-Layer Aluminum Flake Coating Automotive Finishes

INDUSTRIAL COATINGS-POWDER

Thermoset Acrylic Polymer Blends

PAPER AND/OR FILM COATING

Olefin Latex

S/B Latex

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Glass Containers

Radiation-Curable Resins Laminate Binder for Printing Plates Resist Lacquer for Electron-Beam Cure Ultraviolet Curable Coatings

These are just a few of the ways

# for all these applications.

#### **Typical Comonomer** Method of Polymerization **Contribution of HEA or HPA** Methyl Acrylate, 2-Ethylhexyl Acrylate Emulsion Adhesion Methacrylic Acid Methyl Acrylate, 2-Ethylhexyl Acrylate, Solution Adhesion Methacrylic Acid Methyl Methacrylate Solution, Ethyl Acetate Reactivity with Blocked Isocyanate Styrene, Butyl Acrylate Emulsion Reaction with Aminoplasts Vinylidene Chloride. Emulsion Adhesion, Stabilization Propylene, Alkyl Acrylate Acrylic Acid Aqueous Solution or Dispersion **Crosslinking Site** Ethyl Acrylate, Methyl Methacrylate Solution, t-butanol Reactivity with Melamine Resin Methacrylic Acid N-Alkoxymethyl Urethanes Solvent **Crosslink Site** Glycidyl Acrylate, Alkyl Acrylate Solvent Crosslinking with Isocyanate Branched Polyester Monomers Crosslink Site for Melamine Resins Solvent Branched-Chain Glycol Polyester, Low Solvent Crosslink Site Alkyl Acrylate, Other Vinyl Monomers Other Vinyl Monomers Low Solvent **Crosslink Site** Other Acrylates Solvent **Crosslink Site** Methyl Methacrylate, Butyl Acrylate Emulsion Reaction with Blocked Isocyanate Ethylene, Vinyl Chloride Emulsion Formulation Compatibility Styrene, Butadiene Emulsion Stability, Compatibility Styrene, 2-Ethylhexyl Acrylate, Acrylic Acid Emulsion or Solution Crosslinking with Cyclic Acetals Isocyanate, Ethyl Methacrylate Solution/Neat Reaction Site for Olefinic Isocvanate Diacrylates, Diisocyanate Reaction Site for Isocyanate Solution Alkyl Acrylate, Acryl Amides Solution/Neat Rad-cure Site All Systems and Monomers (survey) All Types Reaction Site/Rad-cure Site

HEA and HPA contribute to resin performance. Contact Dow to find out how they can perform in your application.



# CIBA-GEIGY epoxy hardeners and the VOC solution

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

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

Ever since VOC became a concern, formulators have been looking to their suppliers

to develop high solids substitutes for existing standard epoxy resin/ poly (amidoamine) hardener formulations — substitutes that could provide the high performance characteristics necessary in coatings for bridges, containers and tanks, pipes, ships, and railroad cars.

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

#### More than just a replacement.

A look at its properties should be sufficient to convince you that XU 283 is more than just a replacement for conventional poly (amidoamine) hardeners to achieve high solids coatings. Because its viscosity ranges between 3000-4500 cP. at



nulation had dramatically demonstrated improved corrosion resistance over control

25°C, XU 283 requires significantly less solvent than standard poly (amidoamine) hardeners to reach a workable stage. Because XU 283 is compatible with solvent-free liquid epoxy resins such as our Araldite® 6010 epoxy resin, no induction period is necessary and, consequently, coatings can be applied immediately after mixing. In addition, XU 283 also resists blushing and exudation.

But what about end results? In the final analysis, how does XU 283 compare to conventional products? The fact is, crosslinked with Araldite 6010 resin, XU 283 actually improves the performance and application of coatings formulations. Compared to formulations using unacceptably high solvent levels, XU 283 produces harder film; exhibits

#### **Chemical resistance**

Immer 12-16 mils; Cure	sion tests d 7-10 days (	@ R.T.
Formulation	1 Parts b	2 y weight
Araldite® 6010 XU 283	100 70	-
"1" Type Epoxy Resi Conventional Polyan	n — nide —	100 54
Weeks	s to failure (1	Year Test)
Tap water	>52	8
Deionized water	>52	8
Sea water	>52	>52
50% NaOH	>52	>52
10% NH4OH	>52	1
10% H₂SO₄	>52	2
10% HCL	>52	1
Unleaded gas	>52	>52
Diesel fuel	>52	>52
Skydrol® 500B*	>52	8
Heptane	>52	>52
Xylene	>52	16
MEK	12	1
Isopropanol	>52	26
50% Ethanol/H2O	>52	8

Note >52=Unaffected after 1 year Substrate: Sandblasted hot rolled steel \*Registered trademark of Monsanto Industrial Chemicals Co. better chemical (see chart), corrosion and abrasion resistance; and can provide excellent mechanical properties, especially adhesion. Add all this to the high solids content of XU 283 and its elimination of sweat-in time, and you can understand why initial response from formulators has been overwhelmingly enthusiastic.

#### Technology and service.

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

# **CIBA-GEIGY**

#### DSC AND DMA ANALYSIS OF THE CURING OF A LIQ-UID EPOXY RESIN WITH DIAMINES—A. Toussaint, P. Cuypers, and L. D'Hont

Journal of Coatings Technology, 57, No. 728, 71 (Sept. 1985)

The curing of a liquid epoxy resin with primary diamines is followed by Differential Scanning Calorimetry (DSC) and by Dynamic Mechanical Analysis (DMA).

The DSC experiments show that the kinetic parameters change with curing temperature, extent of conversion of the reactive groups, and the presence of catalysts.

The DMA experiments point out that the curing process seems to proceed through five steps: microgel formation, macrogel formation, macroscopic gel point, (macroscopic crosslinking), and vitrification. But, depending upon the curing conditions, some of these steps may occur simultaneously and hence may not be observed separately.

#### PREPARATION OF ACRYLIC OLIGOMERS FOR HIGH SOLIDS COATINGS USING HYDROXY-FUNCTIONAL MERCAPTAN CHAIN TRANSFER AGENTS—R.A. Gray

Journal of Coatings Technology, 57, No. 728, 83 (Sept. 1985)

Acrylic oligomers intended for application in high solids acrylic coating formulations were prepared by free radical solution polymerization using hydroxy-functional mercaptan chain transfer agents. Hydroxy-functional mercaptans that are commercially available or that could easily be made commercially available are emphasized.

Properties of the oligomers produced were investigated. These included molecular weight, polydispersity, solution viscosity, and residual odor. Properties of cured coatings resulting from high solids formulations of the oligomers and methylated melamine resins were also investigated.

#### EFFECT OF SOLVENT AND SOLVENT CONCENTRA-TION ON THE INTERNAL STRESS OF EPOXIDE RESIN COATINGS—M. Shimbo, M. Ochi, and K. Arai

Journal of Coatings Technology, 57, No. 730, 93 (Sept. 1985)

The magnitude and the mechanism for occurrence of internal stress were investigated for the solvent-based bisphenol-A type epoxide resin coatings cured with triethylenetetramine. For these coating systems, internal stresses were not observed when the system was in the rubbery region. Rather, they were generated during cooling from the glass transition temperature of each system to room temperature. The magnitude of the internal stress depends on the concentration of the residual solvents, regardless of solvent type. Furthermore, the measured internal stress was in good agreement with the calculated value based on the strain and the modulus of coatings.

#### JCT December 1985 Post-Convention Issue

On October 7-9, the Annual Meeting and Paint Industries' Show of the Federation of Societies for Coatings Technology will take place at the Cervantes Convention Center in St. Louis, Missouri. Complete coverage of this event will be published in the December 1985 issue of the JOURNAL OF COATINGS TECHNOLOGY. Follow-up features will include awards, technical proceedings, exhibitor booth descriptions, and Convention and Annual Meeting news.

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Proven in many diverse applications, Polyvinyl's special-purpose acrylic copolymers, such as NeoCryl<sup>®</sup> A-622, have outstanding moisture resistance built right into the molecule. They dry quickly to high-gloss clear or pigmented finishes that resist water, humidity, salt spray and marring.

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# Program



# CERVANTES CONVENTION CENTER ST. LOUIS, MISSOURI OCTOBER 7-9

# Program

#### MONDAY, OCTOBER 7

#### **OPENING SESSION**

#### (10:00)

Sixty-Third Annual Meeting of the Federation of Societies for Coatings Technology opened by President Joseph A. Bauer

Invocation: The Most Reverend George Gottwald, Auxiliary Bishop of St. Louis

Welcome: Charles L. Grubbs, President of St. Louis Society for Coatings Technology

Howard Jerome, Chairman of the Host Committee

Joseph A. Vasta, Chairman of the Program Committee

John C. Ballard, Chairman of the Paint Industries' Show Committee

Introduction of Distinguished Guests

A salute to raw material and equipment suppliers to the coatings industry.

#### E.W. FASIG KEYNOTE ADDRESS

#### (10:30-11:30)

TUNG OIL TO GROUP TRANSFER POLYMERIZATION— John P. McAndrews, Group Vice-President, Finishes and Fabricated Products Dept., E. I. du Pont de Nemours & Co., Inc., Wilmington, DE

#### **R & D MANAGEMENT SYMPOSIUM**

#### (2:00-5:00)

Often overlooked in industrial research is the importance of attendant factors which strongly influence the success or failure of the technical effort. This session examines some of these factors, ranging from techniques to start the creative juices flowing to the motivation and rewarding of technical personnel.

Moderator—Percy Pierce, Manager of Physical/Analytical Research, PPG Industries, Inc., R & D Center, Allison Park, PA

COATINGS TECHNOLOGY—HIGH TECH, LOW RE-SPECT—Marco Wismer, Vice-President of Science and Technology, PPG Industries, Inc., Pittsburgh, PA

THE PIONEERING RESEARCH EXPERIENCE AT ROHM AND HAAS—William D. Emmons, Director of Pioneering and Specialty Chemicals Research, Rohm and Haas Co., Spring House, PA

CREATIVE THINKING AND PROBLEM SOLVING—THEIR APPLICATION TO BUSINESS AND MANAGEMENT—John S. Ostrowski, Research Director, The Valspar Corp., Minneapolis, MN

MAKING THE DUAL LADDER WORK IN MONSANTO'S TECHNICAL COMMUNITY—William E. Koerner, Director, Physical and Environmental Sciences Centers, Central Research Laboratories, Monsanto Co., St. Louis, MO

PUTTING THE "R" INTO PRODUCT AND PROFIT—Peter V. Robinson, Associate Director, Polymer and Coatings Research, Glidden Coatings & Resins, Div. of SCM Corp., Strongsville, OH

#### MANUFACTURING THE NEXT GENERATION OF COATINGS

(2:00-5:00)

Panel of speakers will discuss various aspects of the manufacturing process, including automating the paint plant, trade sales and industrial water-bornes, and high-solids vs. conventional.

### **Registration Hours**

 Sunday, October 6
 1:00 pm-5:00 pm

 Monday, October 7
 8:00 am-5:30 pm

 Tuesday, October 8
 8:00 am-5:30 pm

 Wednesday, October 9
 8:00 am-3:00 pm

### **COLORSHIP** is **DISTRIBUTION**

RONDB

"CREATIVE C

The Yankee Clippership was the mainstay of America's world trade network in the 19th century. Clipperships best fulfilled the essentials of successful distribution – delivering the right material to the right place, at the right time, in the right condition.

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#### PHYSICAL CHEMISTRY OF COATINGS (9:00-11:00)

MECHANICS OF ELECTROSTATIC ATOMIZATION, TRANSPORT, AND DEPOSITION OF COATINGS—Guy C. Bell, Jr., Senior Research Associate, E. I. du Pont de Nemours & Co., Inc., Finishes and Fabricated Products Dept., Marshall Laboratory, Philadelphia, PA

THE INFLUENCE OF FINE-PARTICLE SIZE EXTENDERS ON THE OPTICAL PROPERTIES OF LATEX PAINTS—Luigi Cutrone, Supervisor, Application Development, Tioxide Canada Inc., Sorel, Quebec, Canada (A Roon Awards competition paper)

PHOTODEGRADATION OF MELAMINE-ACRYLIC COAT-INGS—Richard C. Wilson, Technical Leader, Fundamental Sciences Section, Specialty Resins R & D, Monsanto Polymer Products Co., Springfield, MA

DYNAMIC PROPERTIES AND PERFORMANCE OF THER-MOSET FILMS—Loren W. Hill, Science Fellow, Monsanto Polymer Products Co., Springfield, MA

#### **CORROSION AND ADDITIVES**

#### (9:30-11:30)

ACCELERATED NATURAL CORROSION TESTING—Civilingenior Helge Meyer, NIFAB, Malmo, Sweden, and Civilingenior Ago Saarnak and Klaus Lampe, Scandinavian Paint and Printing Ink Research Institute, Horsholm, Denmark (Presented on behalf of SLF: Federation of Scandinavian Paint and Varnish Technologists)

CATIONIC ELECTRODEPOSITION PRIMERS OVER ZINC COATED STEELS (PART I): EFFECT OF VOLTAGE AND SUBSTRATE ON COATING QUALITY—Clifford K. Schoff, Scientist, Coatings and Resins, PPG Industries, Inc., R & D Center, Allison Park, PA

AN INVESTIGATION OF MICROENCAPSULATED FUNGI-CIDES FOR USE IN EXTERIOR TRADE SALES PAINTS— G. K. Noren, M. F. Clifton, and A. H. Migdal, DeSoto, Inc., Administrative and Research Center, Des Plaines, IL (A Roon Awards competition paper)

URETHANE-FUNCTIONAL ALKALI-SOLUBLE ASSOCI-ATIVE LATEX THICKENERS—G. D. Shay and A. F. Rich, DeSoto, Inc., Administrative and Research Center, Des Plaines, IL (A Roon Awards competition paper)

#### RHEOLOGY

#### (9:00-12:00)

PRINCIPLES AND RHEOLOGY OF ADHESION—A. N. Gent, The University of Akron, Akron, OH

PREDICTING PERFORMANCE OF COATINGS OPER-ATIONS WITH COMPUTER-ASSISTED FINITE ELEMENT MATHEMATICS—L. E. Scriven and C. W. Macosko, Dept. of Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, MN MODELING OF THERMOSET OVEN-CURING INCLUDING FLOW AND EVAPORATION EFFECTS USING FINITE ELE-MENT ANALYSIS—R. L. Mullen, Dept. of Civil Engineering, Case Western Reserve University, Cleveland, OH, and M. J. Braun, Dept. of Mechanical Engineering, The University of Akron, Akron, OH

POLYVINYLACETATE LATEX DESIGN AND RHEOLOGI-CAL PERFORMANCE IN LATEX PAINTS—Alfred Rudin, Chemistry Dept., Institute of Polymer Research, University of Waterloo, Waterloo, Ontario, Canada

EFFECT OF MOISTURE AGING ON DISPERSIBILITY OF CARBON BLACK, KAOLIN, AND TITANIUM DIOXIDE DIS-PERSED IN POLYBUTENE AND 1,2,6 HEXANETRIOL MEA-SURED BY SHEAR SENSITIVITY—Jen-Ten Kuo, Raymond R. Myers, Carl J. Knauss, and Richard J. Ruch, KSU Coatings Laboratory and Chemistry Dept., Kent State University, Kent, OH

THE CONTRIBUTIONS OF DYNAMIC UNIAXIAL EXTEN-SIONAL VISCOSITIES TO TRADE-SALES ROLL AND IN-DUSTRIAL ROLL AND SPRAY APPLICATION PERFOR-MANCE—R. H. Fernando and J. E. Glass, Polymers and Coatings Dept., North Dakota State University, Fargo, ND (A Roon Awards competition paper)

#### SOCIETY PAPERS

#### (2:00-4:00)

THE CORRELATION OF RESULTS FROM "COLOR-MATCHING APTITUDE TEST" SCORES—Birmingham Paint, Varnish & Lacquer Club. Presented by B. J. Addenbrooke, Croda Paints Limited, Birmingham, England

IMPLEMENTATION OF LABORATORY PAINT FORMULA CALCULATIONS ON AN APPLE II MICROCOMPUTER—Los Angeles Society for Coatings Technology. Presented by James D. Hall, Sinclair Paint Co., Los Angeles, CA

URETHANE ACRYLIC INTERPENETRATING POLYMER NETWORKS (IPN's) FOR COATING APPLICATIONS—Detroit Society for Coatings Technology. Presented by P. 1. Kordomenos, Paint, Plastics, and Vinyl Operations, Ford Motor Co., Mt. Clemens, MI

INFLUENCE OF RHEOLOGY MODIFIERS ON PERFOR-MANCE CHARACTERISTICS OF LATEX PAINTS—Montreal Society for Coatings Technology. Presented by John Hall, Tioxide of Canada Ltd., Montreal, Canada

#### COATING TECHNOLOGY

#### (2:00-4:30)

THE NEW PAINT TECHNOLOGY FOR THE 80'S AND BE-YOND—Alan D. McInnes, Managing Director, Vapocure Limited, Sydney, Australia (Presented on behalf of Oil and Colour Chemists' Association—Australia)

EFFECT OF POLYESTER CARBOXYL AND HYDROXYL END GROUP BALANCE IN ISOCYANATE CURING POW-DER COATINGS—John J. McLafferty, Peter A. Figlioti, and Louis T. Camilleri, Ruco Polymer Corp., Hicksville, NY

HIGH SOLIDS HYBRID THERMOSET COATINGS—Ellsworth E. Faust, Senior Applications Chemist Marketing-Technical Development Laboratory, Celanese Chemical Co., Inc., Summit, NJ

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THE EFFECT OF  $\alpha$ -METHYL GROUPS ON ROOM TEM-PERATURE CROSSLINKING IN ACRYLIC POLYMERS CONTAINING MAGME MONOMERS—H. R. Lucas, Research Chemist, American Cyanamid Co., Stamford Research Laboratories, Stamford, CT (A Roon Awards competition paper) THE EFFECT OF COBALT DRIERS ON THE DRYING AND THE POST-CURE EMBRITTLEMENT OF PALE BODIED VARNISH LINSEED OIL IN MINERAL SPIRITS AND 1,1,1-TRICHLOREOTHANE—Esin Gunay, Laboratory Assistant, The Sherwin-Williams Co., Cleveland, OH, and Harvest L. Collier, Assistant Professor, Dept. of Chemistry, University of Missouri— Rolla, Rolla, MO

### WEDNESDAY, OCTOBER 9

#### **PIGMENTS/DISPERSIONS**

#### (9:00-10:30)

PIGMENT DISPERSIONS: RECENT DEVELOPMENTS IN THE USE OF A-B BLOCK POLYMERS AS DISPERSANTS FOR NON-AQUEOUS SYSTEMS—Henry L. Jakubauskas, Research Associate, E. I. du Pont de Nemours & Co., Inc., Marshall R & D Laboratory, Philadelphia, PA

STRUCTURE AND PROPERTIES OF AMORPHOUS SILICA GEL IN COATINGS APPLICATIONS—Leon Kutik, Manager, Commercial Applications Laboratory, Technical Center, Davison Chemical Div., W. R. Grace & Co., Baltimore, MD

NATURAL SILICAS IN COATINGS—Walter J. Polestak, Group Leader, Paints and Polymers Laboratory, and Thomas D. Thompson, Director of Research, Specialty Applications, Georgia Kaolin Co., Springfield, NJ

#### SOLVENTS

#### (9:00-10:30)

SOLVENT FORMULATION FOR SAFETY IN TOMORROW'S WORLD—Harold L. Jackson, Research Associate, Intermediates Div., Petrochemicals Dept., E. I. du Pont de Nemours & Co., Inc., Experimental Station, Wilmington, DE

THE ROLE OF AZEOTROPY IN SPEEDING UP WATER/SOL-VENT EVAPORATION IN HUMID AIR—Albert L. Rocklin, Senior Research Chemist, Shell Development Co., Westhollow Research Center, Houston, TX (A Roon Awards competition paper)

THE PREPARATION AND EVALUATION OF ALKYD RES-INS THAT COMPLY WITH SOLVENT RESTRICTIVE REGU-LATIONS—James Stoffer, John Gordon, and Karen Beckmann, University of Missouri—Rolla, Rolla, MO

#### MATTIELLO MEMORIAL LECTURE

#### (10:30-11:30)

REFLECTIONS ON THE PHENOMENON OF FADING—Ruth Johnston-Feller, Consultant in Color Science, Research Center on the Materials of the Artist and Conservator, Mellon Institute, Pittsburgh, PA

#### AWARDS LUNCHEON

#### (11:45)

Grand Ballroom, Sheraton St. Louis Hotel

Presentation of these awards: George Baugh Heckel ... Paint Show ... Roon Foundation ... American Paint & Coatings Journal/A. F. Voss ... Materials Marketing Associates ... Program Committee ... Ernest T. Trigg

Featured Speaker: Jim Valvano, Head Basketball Coach, North Carolina State University

#### EFFECTIVE USE OF COMPUTERS IN R & D (2:00-5:00)

Moderator—Theodore Provder, Principal Scientist, Polymer Research and Computer Science Group, Glidden Coatings & Resins, Div. of SCM Corp., Strongsville, OH

ON-LINE INFORMATION RETRIEVAL: A POWERFUL IN-TELLIGENCE GATHERING TOOL FOR THE COATINGS IN-DUSTRY—Joanne Witiak, Information Specialist, Research Div., Rohm and Haas Co., Spring House, PA

APPLICATIONS OF COMPUTER DATA BASE MANAGE-MENT IN POLYMER AND COATINGS RESEARCH—Mark E. Koehler, Group Leader, Instrumentation and Computer Science, Glidden Coatings & Resins. Div. of SCM Corp., Strongsville, OH

THE NEXT STEP IN LABORATORY AUTOMATION: ROBOT-ICS—Steve Pull, Zymark Corp., Zymark Center, Hopkinton, MA

USE OF RS-1, "AN ELECTRONICS NOTEBOOK," IN A COATINGS LABORATORY—Timothy D. Costello, Research Chemist, Finishes & Fabricated Products Dept., E. I. du Pont de Nemours & Co., Inc., Experimental Station, Wilmington, DE

CAF-COMPUTER AIDED FORMULATION—Donald S. Andrade, President, DSA Consulting, Inc., Mission, KS

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

Annual Business Meeting of the Federation Installation of Officers, 1985-86





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# ABSTRACTS OF PAPERS

#### COATINGS TECHNOLOGY—HIGH TECH, LOW RESPECT

#### Marco Wismer, PPG Industries, Inc.

Despite our industry's size, sophistication and product value, coatings technology has been badly underrated. Today, for example, the western world's coatings industry represents \$22 billion in annual sales—a significant sum by anyone's measure. A coating's performance-to-cost ratio is higher than essentially any other product on the market. Its technology involves scientists whose back-grounds include polymer chemistry, organic chemistry, physical chemistry, colloid science, and many other fields.

The low public esteem of coatings technology results from a convergence of opposites; our industry's hidden successes, together with its conspicuous failures, have combined to undercut our professional respect. Our shortcomings include a comparatively low rate of R&D spending, a lack of commitment to research on a sustained basis, a preoccupation with costs at the frequent expense of quality, and a lack of marketing foresight. These deficiencies are real, and they need to be addressed within the industry itself. The external issues, however, present a different problem.

It is easy for the layman to identify a coating failure. But our successes are harder to spot. An effective, high-performance auto finish, for example, is seen as an attractive car, not as an attractive coating. Indeed, it is only through their least sophisticated component—color—that coatings are seen at all. There is a need to educate our technical peers, as well as the general public, to recognize our successes. But beyond that, it will require our renewed commitment to research, especially to leapfrog technology, to restore our market strength and the respect of our professional counterparts worldwide.

#### THE PIONEERING RESEARCH EXPERIENCE AT ROHM AND HAAS

#### William D. Emmons, Rohm and Haas Co.

Establishing, organizing, and managing entrepreneurial long range research programs within the confines of any large company is always a problem. Research establishments just like any other portion of a corporation are frequently inhabited by a conservative bureaucracy dedicated to the *status quo*. In 1973 Rohm and Haas established a Pioneering Research Group which was unique in a number of ways. Experience with this organization and its track record is described in some detail.

#### CREATIVE THINKING AND PROBLEM SOLVING— THEIR APPLICATION TO BUSINESS AND MANAGEMENT

#### John S. Ostrowski, The Valspar Corp.

Learning and problem solving are very important human behavioral traits and depend a great deal on some basic assumptions we make about the thinking processes and our interactions with the world around us, including our abilities to observe and communicate. The need for both creative and rational methods are reviewed, with the focus on using approximations as a valid method for learning and using the mind's imaging process for the verification of thinking progressions.

#### MAKING THE DUAL LADDER WORK IN MONSANTO'S TECHNICAL COMMUNITY

#### William E. Koerner, Monsanto Co.

Monsanto has had a Dual Ladder System for technical and management promotion in existence for about 35 years. The operation of the technical ladder (the Fellow Program) is summarized. Major emphasis is on the key issues involved in harmonizing the reward systems on the management and technical ladders to achieve reasonable equity between the two promotional ladders. Examples of common features and special perquisites on the two ladders are discussed.

#### PUTTING THE "R" INTO PRODUCT AND PROFIT

#### Peter V. Robinson, Glidden Coating & Resins, Div. of SCM Corp.

The advantages, problems and difficulties of converting *research* activity in a research and development laboratory into profitable products are explained. Many opportunities and barriers do exist; in many cases the personal, non-academic characteristics of the researcher are as important as their purely technical contributions.

#### MECHANICS OF ELECTROSTATIC ATOMIZATION, TRANSPORT, AND DEPOSITION OF COATINGS

#### Guy C. Bell, Jr. and Jerome Hochberg, E. I. du Pont de Nemours & Co., Inc.

The mechanics of electrostatic atomization, transport, and deposition of coatings were studied. Emphasis was given to electrostatic application of automotive finishes by a high-speed rotary atomizer. The dynamics of atomization and particle deposition were examined using high-speed video recording techniques. Spray droplet sizes and speeds were measured with laser light-scattering and Doppler methods. Charge/mass ratios were determined. The droplet parameters were related to application conditions (electrostatic voltage, fluid flow rate, mechanical forces) and to paint properties (electrical resistivity, surface tension, viscosity).

#### THE INFLUENCE OF FINE-PARTICLE SIZE EXTENDERS ON THE OPTICAL PROPERTIES OF LATEX PAINTS

#### Luigi Cutrone, Tioxide Canada Inc.

For many years it has been assumed that fine-particle size extenders can act as diluents or spacers to enhance the light scattering ability of titanium dioxide. Spacer theory suggests that coarse extenders crowd titanium dioxide particles together, reducing the



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L.T. Camilleri

E.E. Faust

ideal spacing at which the pigment affects maximum light scattering. When extender particles of a size closer to that of titanium dioxide are used, they move between the titanium dioxide particles and space them for greater efficiency. The concept is based on theory alone, and its validity has never been demonstrated. This study demonstrates that the spacer theory is not entirely correct because fine-particle size extenders do not enhance the scattering ability of titanium dioxide, but merely maintain the same level as binder is removed. Several fine-particle size extenders were used in the study and not one of them improved the scattering efficiency of titanium dioxide pigment in a well bound system.

#### PHOTODEGRADATION OF MELAMINE-ACRYLIC COATINGS

#### Richard C. Wilson, Monsanto Company

The ability of melamine-acrylic coatings to withstand exterior environmental conditions has been a topic of intense interest for many years. The growth of computer-aided instrumentation has allowed determination of even more subtle changes in chemical structure.

Chemical changes can be monitored in many ways. Focus is on the use of FT-IR and solid state 13C-NMR techniques to evaluate degradation pathways under both accelerated and Florida exterior exposures.

#### DYNAMIC PROPERTIES AND PERFORMANCE OF THERMOSET FILMS

#### L.W. Hill and K. Kozlowski, Monsanto Polymer Products Company

Films prepared from high solids acrylic or polyester polyols and etherified melamine formaldehyde (MF) crosslinkers have been characterized using an Autovibron instrument from Imass, Inc. Some aspects of cure mechanism, such as co-condensation versus MF self-condensation, have been elucidated based on analysis of dependence of glass transition temperature (Tg) on cure temperature and acrylic/MF ratio. Crosslink density of cured films is discussed in relation to values of the storage modulus (E') determined at temperatures well above Tg. Relationships between resin structure and dynamic properties of cured films are presented. For example, the effect of type of hydroxyl group, primary versus secondary, on extent of cure is shown.

Dynamic properties are also related to performance as measured by widely used tests of hardness, flexibility, impact resistance and solvent resistance. An approach to improving the difficult property balance between hardness and flexibility through control of loss modulus (E") values is described.

#### CATIONIC ELECTRODEPOSITION PRIMERS OVER ZINC COATED STEELS (PART I): EFFECT OF VOLTAGE AND SUBSTRATE ON COATING QUALITY

### Clifford K. Schoff and Huey-Jyh Chen, PPG Industries, Inc.

The automobile industry is turning more and more to zinc coated steels to improve corrosion protection. Considerable amounts of galvanized and zinc alloy coated steels presently are used in production and their proportion of the steel contained in an automobile will rise in the future. These surfaces, along with the rest of the vehicle, normally are primed with cationic electrodeposition primers and results have been excellent. However, under certain conditions cationic e.d. primers can give pits and craters over zinc alloy and galvanized steels.

The problem was reproduced over a variety of zinc coated steels and a wide range of physical and analytical techniques were applied to characterize the defects. Conventional cationic e.d. primers have a high tendency to crater, but the newer high build primers are more resistant to cratering. There are two mechanisms for formation of the defects: electrical discharge (dielectric failure of the wet film) in the bath and gassing. Defects are associated with the zinc on the surface, but with high build primers, the craters occur more often with deposition over zinc-iron alloys.

Craters can be prevented or greatly reduced by using high build cationic e.d. primers rather than conventional ones, operating at lower voltages, using smoother substrates, and controlling A.C. ripple to suppress high voltage peaks.

#### AN INVESTIGATION OF MICROENCAPSULATED FUNGICIDES FOR USE IN EXTERIOR TRADE SALES PAINTS

#### G. K. Noren, M. F. Clifton, and A. H. Migdal, DeSoto, Inc.

The fungicides, 2,3,5,6-tetrachloro-4-methylsulfonyl pyridine (1) and tetrachloroisophthalonitrile (2), were successfully encapsulat-

30

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ed using a technique based on the *in-situ* polymerization of a ureaformaldehyde resin. The microcapsules obtained by this process had an average particle size of 10 $\mu$  and consisted of a matrix containing between 55 and 65% fungicide. Microencapsulation allowed for increased control in reducing both the solubility and volatility of the fungicides. The encapsulated fungicides were incorporated into exterior latex paint formulations which were used for exposure tests in Illinois, Florida, and Louisiana.

Mildew rating data after 12 months' exposure indicated equal or better mildew protection could be obtained using the microencapsulated fungicides. After 24 months, some of the mildew ratings obtained were considered too low to allow accurate interpretation, but other results were encouraging, in that these results continued to indicate a tendency for increased mildew protection with the use of the microencapsulated fungicides.

#### URETHANE-FUNCTIONAL ALKALI-SOLUBLE ASSOCIATIVE LATEX THICKENERS

#### G. D. Shay and A. F. Rich, DeSoto, Inc.

The advantages and limitations of conventional cellulosic thickeners are reviewed along with those of the more recent alkali-soluble and nonionic associative thickener technologies. With particular consideration given to the structural and performance attributes of the latter, a new class of alkali-soluble thickener containing hydrophobically terminated urethane ethoxylate monomer (TMIX) was designed and investigated. The synthesis, characterization, and emulsion terpolymerization of the urethane functional macromonomer with ethyl acrylate and methacrylic acid are described. A Sheffe' experimental design simplex was employed for studying the physical properties of the thickener latices prior to neutralization and for the determination of optimum terpolymer compositions with respect to performance and economics in selected trade sales coating formulations.

#### PRINCIPLES AND RHEOLOGY OF ADHESION

#### A. N. Gent, The University of Akron

The mechanical strength of an adhesive bond depends upon both the chemical and the rheological properties of the adherends. Indeed, it is often difficult to separate the different contributions. Several studies have therefore been carried out using model joints prepared with different amounts of chemical interlinking at the interface between rubber and glass, between rubber and metal, and between one rubber and another.

These experiments are described and interpreted in terms of a characteristic threshold strength of the joint, governed by the type and extent of chemical interlinking, and augmented to a remarkable degree by inelastic behavior of the adherends. Application of these results to the strength of particle-filled and fiber-reinforced materials is also discussed.

#### PREDICTING PERFORMANCE OF COATINGS OPERATIONS WITH COMPUTER-ASSISTED FINITE ELEMENT MATHEMATICS

#### L. E. Scriven and C. W. Macosko, University of Minnesota, Minneapolis, MN

Complicated flow and rheology put coating operations beyond conventional mathematics. Dynamics of wetting and air entrainment put them at the fringe of established physics. However, newly developed methods of computer-assisted mathematics, together with advancing large-scale scientific computers are making

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it possible to calculate predictions of performance from governing principles, provisional mechanisms, and refined rheological characterizations. Comparisons with experiment then guide completion of the theory and ultimately validate computer codes for analyzing performance, improving operations, and designing applicators.

The same systematic approach can be brought to bear on rheological characterization itself. Illustrations are drawn from recent studies of curtain, roll, blade, and slide coating operations. This paper constitutes in part a review of the Coatings Flows research program at the University of Minnesota.

#### MODELING OF THERMOSET OVEN-CURING INCLUDING FLOW AND EVAPORATION EFFECTS USING FINITE ELEMENT ANALYSIS

### Robert L. Mullen, Case Western Reserve University and M. J. Braun, University of Akron

A finite element model for the flow of a coating during oven drying is presented. The model is based on a Reynold's equation, which has been modified to model non-Newtonian rheology. The







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use of the Reynold's equation significantly reduces the computational effort required to solve the model by reducing the dimension of the calculations. The thermal portion of the model includes the geometry of the substrate, along with the varying thermal properties of the coating. The energy and solvent loss due to evaporation is calculated by an "equation of state" approach similar to the AQUEVAP program [A. Rocklin & D. Bonner, JOURNAL OF COATINGS TECHNOLOGY, 1980].

The resulting model is applied to predict the coating flow due to variation in temperature caused by a structural rib on the underside of a flat plastic low thermal conductivity component.

The effect of initial film thickness as well as oven temperature/ humidity are quantified.

In addition, the role of surface tension vs. gravity in driving the flow is presented. Modification of the surface tension as a method to reduce curing time without inducing excessive coating flow is indicated.

#### POLYVINYLACETATE LATEX DESIGN AND RHEOLOGICAL PERFORMANCE IN LATEX PAINTS

#### Alfred Rudin, University of Waterloo (Ontario)

A cup and bob rheometer was used to characterize the rheological behavior of vinyl acetate-dibutyl maleate copolymer latices of emulsion paints. A simple experimental procedure was devised which correlates well with user opinions of performance. Latices were synthesized with a range of particle sizes. Thixotropic behavior of the paints was found to depend primarily upon emulsion polymerization ingredients.

Cellulosic and associative thickeners are compared and some reasons suggested for the observed differences in performance.

#### EFFECT OF MOISTURE-AGING ON DISPERSIBILITY OF CARBON BLACK, KAOLIN, AND TITANIUM DIOXIDE DISPERSED IN POLYBUTENE AND 1,2,6 HEXANETRIOL MEASURED BY SHEAR SENSITIVITY

### Jen-Ten Kuo, Raymond R. Myers, Carl J. Knauss and Richard J. Ruch, Kent State University

Shear viscosities of moisture-conditioned suspensions were measured over the range of 20-200 sec<sup>-1</sup> for pigment volume percentages of 10 to 13%. Titanium dioxide and kaolin were chosen as representative of hydrophilic pigments and carbon black as a hydrophobic pigment. Polybutene represented a non-polar vehicle; and 1.2,6-hexanetriol. a polar vehicle. The pigments were subjected to water vapor atmospheres of fixed relative humidity to generate adsorption isotherms and to prepare pigment surfaces of known moisture coverage.

The Bingham yield values increased with water coverage for the titania and clay, but decreased for the carbon black when they were dispersed in polybutene. In the triol, titania and clay were Newtonian but the carbon black was shear sensitive.

Effects, including those on the plastic viscosity, are discussed in terms of the role of the moisture and of the activation energies calculated over the range of 20° to 50°C.

#### THE CONTRIBUTIONS OF DYNAMIC UNIAXIAL EXTENSIONAL VISCOSITIES TO TRADE-SALE ROLL AND INDUSTRIAL ROLL AND SPRAY APPLICATION PERFORMANCE

#### R. H. Fernando and J. E. Glass, North Dakota State University

The dynamic uniaxial extensional viscosity (DUEV) of waterborne latex coatings thickened with a cellulose ether has been measured for the first time and related with the coating's spatter characteristics. The DUEVs of industrial coil coating formulations varying in their ribbing and misting characteristics have also been measured and related. Attempts to measure the DUEV of interior can coatings applied by spray were unsuccessful without the addition of high molecular weight poly(oxyethylene). Modification in small amounts resulted in an inverse relationship between the coating's tendency to mist and its DUEV.

In spray coating applications, the relationship is straightforward. In coil coatings, the phenomenon is more complex. Ribbing occurs when the viscous forces overcome the influence of surface tension in minimizing the fluid's free surface area. The radius and peripheral velocity of the rolls, and nip spacing between the rolls all contribute to the onset of ribbing. In individual polymer blend studies which permitted separation of shear and extensional components, the importance of DUEVs in facilitating an earlier onset of ribbing at a given nip spacing and roll velocity is illustrated. In total, the study delineates the primary importance of DUEV and nip spacing in determining the number of ribs at a given set of roll conditions, and the importance of DUEV at high roll velocities in web growth and misting. These and other effects are discussed in detail.

#### THE CORRELATION OF RESULTS FROM "COLOR MATCHING APTITUDE TEST" SCORES ABSTRACT

#### Birmingham Society for Coatings Technology

Data from tests performed by several organizations using the 1978 version of the Color-matching Aptitude Test Set were collated and compared with similarly collected data for the 1964 version of the test. Small but significant differences between the versions in the mean-individual color raw scores were noted, although the total scores in each case gave a normal Gaussian distribution. This enables a revised interpretation of scores to be drawn up for the 1978 Test version.

An age-dependence for mean scores was also observed, with the best mean scores being achieved by examinees in the 25-29 age group.

#### IMPLEMENTATION OF LABORATORY PAINT FORMULA CALCULATIONS ON AN APPLE II MICROCOMPUTER

#### Los Angeles Society for Coatings Technology

The design and coding of a program written in Applesoff<sup>®</sup> Basic to perform standard laboratory calculations is detailed. Hardware configuration and limitations are explained. The program stores information on over 1,000 raw materials on one diskette. The program stores approximately 100 formulae on one diskette. Standard calculations are performed on stored formulae utilizing stored raw material information. The program outputs to the monitor screen and, optionally, to a printer.

#### THE INFLUENCE OF RHEOLOGY MODIFIERS ON THE PERFORMANCE CHARACTERISTICS OF LATEX PAINTS

#### Montreal Society for Coatings Technology Technical Committee

A series of latex paints was prepared based on a range of commercially available rheology modifiers, including cellulose derivatives, alkali-soluble polymers and (HEUR) associative thickeners. An extensive range of rheological, optical and physical properties was determined in order to evaluate the relative performance characteristics of each system.

It was found that both the associative and alkali-soluble thicken-

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Federation of Societies for Coatings Technology 1315 Walnut Street Philadelphia, PA 19107 ers generally offered superior flow and leveling, and application properties relative to systems based on cellulosic polymers. When associative thickener/cellulose combinations were used, the overall performance of the system was found to be inferior relative to an associative thickened system, application properties in particular being adversely affected.

### THE NEW PAINT TECHNOLOGY FOR THE 80's AND BEYOND

#### Alan D. McInnes, Vapocure Limited

A commercially viable alternative to the traditional baking of industrial paint finishes is described, wherein a room temperature, 10-minute catalyzing cycle replaces a typical 30-minute 180°C baking schedule.

Specifically formulated acrylics, epoxies, alkyds and urethanes are exposed to a contained, mildly turbulent atmosphere containing controlled parts per million of specific catalysts to achieve rapid room temperature drying. Thus the way is opened for the painting of heat sensitive substrates, plastics, partially and fully assembled items, finally breaking the present industrial tyrany of high temperature paint drying on production lines.

#### EFFECT OF POLYESTER CARBOXYL AND HYDROXYL END GROUP BALANCE IN ISOCYANATE CURING POWDER COATINGS

### John J. McLafferty, Peter A. Figlioti, and Louis T. Camilleri, Ruco Polymer Corp.

The balance of hydroxyl and carboxyl end groups in polyesters for urethane powder coatings is a parameter which can significantly affect curing characteristics and performance of the coatings.

Reaction kinetics of these functional groups with aromatic and aliphatic isocyanates are reviewed. The practical effect of the functional group balance at a nominal reactive number is studied in a standard catalyzed powder formulation based on a resin series with caprolactam blocked isocyanate curatives. Resin viscosities and Tg are compared and flow behavior and gel times of laboratory prepared compounds are evaluated. Properties of the cured coatings are also examined.

#### HIGH SOLIDS HYBRID THERMOSET COATINGS

#### Ellsworth E. Faust, Celanese Chemical Company, Inc.

Preparations of very high active content coatings formulations are described. The coatings are hybrids of solution acrylic copolymers and reactive diluents. The diluents are mono-esters of triols such as trimethylolpropane with drying oil fatty acids which increase the hydroxyl content of the liquid coatings while at the same time reducing their solution viscosities.

Thermoset coatings systems prepared from these formulations exhibited both good flexibility and good chemical resistance. Solution viscosities of 2000 to 6000 centipoise at active content levels of 79 to 84% were achieved.

### THE EFFECT OF $\alpha$ -METHYL GROUPS ON ROOM TEMPERATURE CROSSLINKING IN ACRYLIC POLYMERS CONTAINING MAGME MONOMERS

#### H. R. Lucas, American Cyanamid Co.

Coating formulations, based on MAGME acrylic polymers and aimed at the automotive refinish market, were found to develop a rapid and high degree of cure with multifunctional primary amines at room temperatures. Curing at temperatures some twenty degrees below their glass transitions, these polymers exhibited large differences in their rate of hardness development and solvent resistance. Increasing the amount of  $\alpha$ -methyl groups along the backbone of the polymer was found to greatly improve the properties of the coatings.

Based on this work, a hypothesis has been proposed and supported by experimental data that these differences are largely due to the amount of inter- or intramolecular crosslinks that occur.

#### THE EFFECT OF COBALT DRIERS ON THE DRYING AND THE POST-CURE EMBRITTLEMENT OF PALE BODIED VARNISH LINSEED OIL IN MINERAL SPIRITS AND 1,1,1-TRICHLOROETHANE

#### Esin Gunay and Harvest L. Collier, University of Missouri-Rolla

A study is presented that demonstrates how steric features of different functional groups in cobalt-organic acids as driers affect the post-cure embrittlement of Pale Bodied Varnish Linseed Oil (PBVLO). Predictive correlation of decreased oxidative crosslinking during the post-cure reaction period for PBVLO films containing cobalt driers having a short-substituted carbon chain, a long-unsubstituted carbon chain, an aromatic ring about the cobalt metal, and an aromatic ring 6-carbon unit away from the carbon metal are made. Interpretation of how catalyst concentration, film dry-time, accelerated weathering, and solvent affect post-cure embrittlement is also discussed.

This study illustrates some aspects of the complexity of possible curing mechanisms. It also suggests that some film post-cure properties may be controlled by design for well-characterized formulations.

#### PIGMENT DISPERSIONS: RECENT DEVELOPMENTS IN THE USE OF A-B BLOCK POLYMERS AS DISPERSANTS FOR NON-AQUEOUS SYSTEMS

### Henry L. Jakubauskas, E. I. du Pont de Nemours & Co., Inc.

Theoretical considerations involved in the dispersions of pigments are reviewed, including a new class of pigment dispersants for non-aqueous solvent systems that has been developed by Du Pont. These dispersants are polymer molecules containing "polar clusters" attached to one end of non-polar polymeric chain. The stability of dispersed pigment particles is achieved by adsorption of the "polar cluster" on the pigment surface with the non-polar portion of the molecule providing a barrier for pigment-to-pigment interactions. The mechanism of stabilization of particles is steric (entropic).

Pigment deflocculation is shown to be a function of dispersant structure, molecular weight polar and non-polar segments, pigment polarity, and solvent polarity used.

#### STRUCTURE AND PROPERTIES OF AMORPHOUS SILICA GEL IN COATINGS APPLICATIONS

#### Leon Kutik, W. R. Grace & Co.

Amorphous silica gel is a unique type of synthetic silica which normally exhibits a high surface area, low density, and a porous structure. Each of these properties is controlled by the manufacturing process so that highly specialized products for a variety of coating applications result. Physical properties such as pore volume, surface area, and density are important considerations during the manufacturing process. Examples are shown how these parameters are modified to optimize the adsorption properties of water sensitive coating materials. Similarly, the effect of particle size, density, pore volume, and surface properties on the flatting efficiency of silica gel pigments are described. Flatting efficiency curves demonstrating the use of silica gel pigments in various industrial coating systems are presented. The use of recently developed silica gel thixotropes in various non-aqueous systems are also described, together with a comparison with other types of synthetic silicas.

#### NATURAL SILICAS IN COATINGS

### Walter J. Polestak and Thomas D. Thompson, Georgia Kaolin Co.

Silica extender pigments obtained from natural sources have been used in various coating systems for some time. A review of the microcrystalline silicas available to the paint formulator is included for reference purposes. Cost reduction and sheen control were recognized as attributes of ground natural silica.

Properties of the finer or micronized natural silicas, such as lower pigment abrasion and low oil absorption, are covered to indicate potential contributions to the traditional or conventional as well as new coating systems.

#### SOLVENT FORMULATION FOR SAFETY IN TOMORROW'S WORLD

#### Harold L. Jackson, E. I. du Pont de Nemours & Co., Inc.

Solvents in coating formulations of tomorrow will have to provide not only optimum solvency and evaporative properties, but also a higher degree of human safety and economy in use. This movement has already begun, as evidenced by growing efforts to replace ethylene glycol ethers and acetates with blends of solvents such as propylene glycol ethers and acetates and dibasic esters (DBE).

New solvent formulations for coatings of acrylic, epoxy, and polyester resins are discussed with emphasis on those which will provide the safety required in tomorrow's world.

#### THE ROLE OF AZEOTROPY IN SPEEDING UP WATER/SOLVENT EVAPORATION IN HUMID AIR

#### Albert L. Rocklin, Shell Development Company

Water/solvent azeotropes evaporate faster than water, but they do so without change in composition only in dry air. In humid air they lose cosolvent because the water evaporation is retarded. Blends which evaporate into humid air without change in composition must have higher cosolvent concentrations than the azeotrope. This presentation describes these blends and defines them as *pseudo azeotropes*. Their composition depends on relative humidity and, in each case, is close to the composition of the fastest evaporating blend at that humidity. Flanking the pseudo azeotrope there is usually a broad range of blends that evaporate faster than water at the same humidity. The calculated by an established program for computing water/solvent evaporation, so that for any humidity range a compromise

blend can be selected that is advantageously close to the pseudo azeotropes within that humidity range and, therefore, will have optimally fast evaporation within that range.

Azeotroping efficiencies of cosolvents are compared in computed evaporation rate studies of binary and ternary water/ solvent blends over a wide range of humidities. Selection of optimum evaporating blends is illustrated. This should be useful in dealing with evaporation during air dry and ambient temperature flash-off of water-reducible coatings.

#### THE PREPARATION AND EVALUATION OF ALKYD RESINS THAT COMPLY WITH SOLVENT RESTRICTIVE REGULATIONS

### James Stoffer, John Gordon and Karen Beckmann, University of Missouri-Rolla

The tightening of EPA solvent emission regulations has spurred the use of compliance solvents in coatings. A comparison of 1,1,1trichloroethane with mineral spirits in white architectural coatings was performed to determine the effects of the compliance solvent.

The fast evaporation rate of 1,1,1 trichloroethane adversely affected the stability of the coatings. But these coatings dried faster and exhibited improved gloss retention and scrub resistance. Most importantly, the Volatile Organic Compounds content (VOC) was reduced from 3.9 to 0.7 pounds per gallon with the use of compliance solvents.

### REFLECTIONS ON THE PHENOMENON OF FADING

#### **Ruth Johnston-Feller, Consultant**

Evaluation of the degree of fading on exposed paints by visual means alone obscures the underlying physical behavior of the pigments. It is shown that "Mother Nature is logical" and that, when hiding is complete, and the dispersion is uniform, the rate of fading is constant and is not appreciably affected by many of the variables of paint formulation, such as pigment volume concentration. On this basis, the observed visual changes are also predictable. When hiding is incomplete, however, the rate of fading increases and is dependent on the nature of the substrate. Illustrations of these phenomena are presented.

#### ON-LINE INFORMATION RETRIEVAL: A POWERFUL INTELLIGENCE GATHERING TOOL FOR THE COATINGS INDUSTRY

#### Joanne Witiak, Rohm and Haas Co.

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#### APPLICATIONS OF COMPUTER DATA BASE MANAGEMENT IN POLYMER AND COATINGS RESEARCH

### Mark E. Koehler, Glidden Coatings and Resins, Div. of SCM Corp.

The recently acquired ability of the scientist to have easy access to uncomplicated computerized data base management utilities without the necessity of, and the constraints connected with, involving systems personnel in these projects has provided the opportunity to store, retrieve and analyze data in ways which would not have been practical or even possible before.

The data base applications range from very large, complex systems which may have a life of many years, to fairly small, short-term, personal data bases. This presentation attempts to describe a range of applications of data base management implemented in Glidden laboratories. These applications include managerial applications, such as project monitoring and patent disclosure tracking; information retrieval applications, such as a research report index, lab notebook tracking and file indexing; and technical data retrieval and analysis applications involving exposure test data and laboratory experimental data.

### THE NEXT STEP IN LABORATORY AUTOMATION: ROBOTICS

#### Steve Pull and James N. Little, Zymark Corporation

A new technology, robotics, already being used in other fields, is slated to have a major impact in automating operations and procedures performed in chemistry laboratories during the eighties. An introduction to laboratory robotics, which combines the technologies of chemistry, analytical instrumentation, computers and robotics, is presented.

Laboratory automation, once limited to computerized data reduction, now includes sample handling and sample preparation, wet chemistry procedures, laboratory process control and instrumental analysis.

Laboratory robots, utilizing programmable computers, can be easily reprogrammed to do a variety of laboratory procedures and, thus, does not require a great number of identical, repetitive operations to justify the investment in capital and time.

Examples are given in automated sample preparation for chromatography, spectroscopy, and other chemical laboratory analyses, as well as automating physical testing procedures. Comparisons are given for automated versus manual procedures in terms of time and precision.

#### THE USE OF RS/1, "AN ELECTRONIC NOTEBOOK," IN A COATINGS LABORATORY

#### Timothy D. Costello, E. I. du Pont de Nemours & Co., Inc.

RS/1, the Research System, is an integrated software package intended for use by scientists and engineers. It is designed as an "electronic notebook," thus it contains built-in tools for data entry, editing, graphics, and statistical analyses. In addition, the system commands are simple English phrases which describe the desired function. Many of the system functions can be run in either a menu-driven form or by typing a single one-line command. This flexibility means that a novice should be able to use the program without any prior knowledge, yet the experienced user is able to use shortcuts inside the environment.

Information is stored in tabular format, which simplifies manipulation of blocks of data by placing them in the same row or column. In addition, RE/1 includes the ability to produce anything from simple scatter diagrams to 3D-pictures using these same blocks of data. Curve fitting and the results of statistical analyses also can be superimposed directly on these plots. Since RS/1 contains drivers for a variety of output devices the labelling and annotation of a plot are very simple and uniform.

RS/1 is more than just a means of storing and retrieving information because it also includes a stand-alone programming environment. Therefore, procedures can be written inside of RS/1 that carry out repetitive tasks or perform a certain calculation across an entire block of experimental values. RPL, the Research Programming Language, can be used to perform all the functions built into RS/1 along with the ability to verify and convert entered data. Examples of both the simple and advanced uses of RS/1 are presented, along with a discussion of some of the difficulties encountered while using the program.

#### CAF-COMPUTER AIDED FORMULATION

#### Donald S. Andrade, DSA Consulting, Inc.

CAD/CAM (computer aided design/computer aided manufacturing) systems for modeling have become an integral part of many industries. These systems give the user the ability to study relationships of properties and to see the effect of changes when elements and properties are modified without actual construction of the product. To be able to apply a similar concept to laboratory bench formulation, we must first be able to mathematically describe and control color, gloss and extenders, dispersion and mill ratios, stoickiometry involving resins, and solvent analyses. The discussion presents an overall flow chart of the programs that are necessary for modeling, as well as comments on some of the mathematics involved.

When bulking is viewed as a part of the formulation modeling process with its rigorous mathematical demands, including flexibility of integration with the above programs, it becomes a separate function from accounting and marketing. With this in mind, the bulking process with its many complexities and pitfalls for formulators is discussed from a laboratory point of view. Finally, it is shown that CAF is the only approach that will allow the formulator a chance to quickly investigate many sides of a problem and choose the most likely path for solution.

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Fillite USA, Inc.         725           Filterite/ Brunswick Tech, Filt. Systems         636           Filter Specialists, Inc.         1620           Floridin Co.         1231           Fricke Enterprises         212           Fryma, Inc.         124
Fillite USA, Inc.       725         Filterite/Brunswick Tech. Filt. Systems       636         Filter Specialists, Inc.       1620         Floridin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124
Fillite USA, Inc.         725           Filterite/ Brunswick Tech. Filt. Systems         636           Filter Specialists, Inc.         1620           Floridin Co.         1231           Fricke Enterprises         212           Fryma, Inc.         124
Fillite USA, Inc.         725           Filterite/ Brunswick Tech. Filt. Systems         636           Filter Specialists, Inc.         1620           Floridin Co.         1231           Fricke Enterprises         212           Fryma, Inc.         124           GAF Corp.         125           Paul N. Gardner Co. Inc.         435
Fillite USA, Inc.       725         Filterite/ Brunswick Tech. Filt. Systems       636         Filter Specialists, Inc.       1620         Floridin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kalin Co.       1001
Fillite USA, Inc.       725         Filterite / Brunswick Tech. Filt. Systems       636         Filter Specialists, Inc.       1620         Floridin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       1001         Globe Trading Co.       138
Fillite USA, Inc.       725         Filterite/ Brunswick Tech. Filt. Systems       636         Filter Specialists, Inc.       1620         Ifordin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       1001         Globe Trading Co.       138         Goodyear Tire & Rubher Co.       724
Fillite USA, Inc.       725         Filterite/ Brunswick Tech. Filt. Systems       636         Filter specialists, Inc.       1620         Floridin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       138         Goodyear Tire & Rubber Co.       724         Gorman-Ruon Co.       122
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Fillite USA, Inc.       725         Filterite/ Brunswick Tech. Filt. Systems       636         Filter Specialists, Inc.       1620         Flordin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       1001         Globe Trading Co.       138         Goodyaar Tire & Rubber Co.       724         Gorman-Rupp Co.       122         W.R. Grace & Co., Davison Chem. Div.       1616         Grefco, Inc.       1616         Grefor, O.       162
Fillite USA, Inc.       725         Filterite/ Brunswick Tech. Filt. Systems       636         Filter specialists, Inc.       1620         Flordin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       1001         Glober Trading Co.       138         Goodyear Tire & Rubber Co.       724         Gorman-Rupp Co.       122         W.R. Grace & Co., Davison Chem. Div.       1616         Grefo, Inc., Dicaperl & Dicalite Depts.       622
Fillite USA, Inc.       725         Filterite/ Brunswick Tech. Filt. Systems       636         Filter specialists, Inc.       1620         Flordin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       1001         Glober Trading Co.       138         Goodyear Tire & Rubber Co.       724         W.R. Grace & Co., Davison Chem. Div.       1616         Grefco, Inc., Dicaperl & Dicalite Depts.       622         Haake Buchler Instruments. Inc.       1025
Fillite USA, Inc.       725         Filterite / Brunswick Tech. Filt. Systems       636         Filter specialists, Inc.       1620         Flordin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       138         Goodyear Tire & Rubber Co.       724         Gorman-Rupp Co.       122         W.R. Grace & Co., Davison Chem. Div.       1616         Grefco, Inc., Dicaperl & Dicalite Depts.       622         Haake Buchler Instruments, Inc.       1029         Halox Piements, Div.       1517
Fillite USA, Inc.       725         Filterite/ Brunswick Tech. Filt. Systems       636         Filterite/ Brunswick Tech. Filt. Systems       636         Fordin Co.       1620         Fordin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       1001         Globe Trading Co.       138         Goodyear Tire & Rubber Co.       724         Gorman-Rupp Co.       122         W.R. Grace & Co., Davison Chem. Div.       1616         Grefor, Inc., Dicaperl & Dicalite Depts.       622         Haake Buchler Instruments, Inc.       1022         Haake Pigments, Div. Hammond Lead Prods.       1517
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Fillite USA, Inc.       725         Filterite/ Brunswick Tech. Filt. Systems       636         Filter specialists, Inc.       1620         Flordin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       435         Goodyear Tire & Rubber Co.       724         Gorman-Rupp Co.       128         W.R. Grace & Co., Davison Chem. Div.       1616         Grefco, Inc., Dicaperl & Dicalite Depts.       622         Haake Buchler Instruments, Inc.       1025         Halox Pigments, Div. Hammond Lead Prods.       1517         Harshaw/Filtrol       304         Helios Container Systems.       418         Henkel Corp., Polymers Div.       824         Hencules Incorporated       911
Fillite USA, Inc.       725         Filterite Brunswick Tech. Filt. Systems       636         Filterite Brunswick Tech. Filt. Systems       636         Fordin Co.       1231         Fricke Enterprises       212         Fryma, Inc.       124         GAF Corp.       125         Paul N. Gardner Co., Inc.       435         Georgia Kaolin Co., Inc.       1001         Globe Trading Co.       138         Goodyaar Tire & Rubber Co.       724         Gorman-Rupp Co.       122         W.R. Grace & Co., Davison Chem. Div.       1616         Grefor, Inc., Dicaperl & Dicalite Depts.       622         Haake Buchler Instruments, Inc.       1022         Hakes Pigments, Div. Hammond Lead Prods.       1517         Herson My/Filtrol       300         Helios Container Systems.       418         Henkel Corp., Polymers Div.       822         Hercules Incorporated.       911         Heubach Inc.       233
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#### CATALYSTS

Phillips Chem. Co. & Catalyst Resources

#### CHEMICAL INTERMEDIATES

Angus Chemical Co. Ashland Chemical Co., IC&S Div. Buckman Laboratories, Inc. Chemtech Industries, Inc. Dow Corning Corp. Eastman Chemical Products, Inc. Hercules Incorporated Lubrizol Corp. Mobay Chemical Corp. Phillips Chem. Co. & Catalyst Resources Rohm and Haas Co. A.E. Staley Mfg. Co. Union Carbide Corp., Specialty Polymers & Composites Div. R. T. Vanderbilt Co., Inc. Virginia Chemical Co. Wacker Chemical Co.

#### COLORANTS

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#### EXTENDERS

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FLATTING AGENTS W.R. Grace & Co., Davison Chem, Div.

LATICES AND EMULSIONS Air Products & Chemicals, Inc. ICI Americas, Inc. Polyvinyl Chemicals Industries, Inc. Reichhold Chemicals, Inc. Rohm and Haas Co. Union Carbide Corp. Union Chemicals Div., Union Oil Co. Witco Chemical Corp., Organics Div.

#### OILS (DRYING/NON-DRYING) Spencer Kellogg Prod./NL Chemicals

A.E. Staley Mfg. Co.

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Aceto Chemical Co., Inc. BASF Wyandotte Corp. Buckman Laboratories, Inc. Columbian Chemicals Co. Degussa Corp. EM Industries, Inc. Halox Pigments, Div. Hammond Lead Prods. Harshaw/Filtrol Heubach Inc Hitox Corp. of America J.M. Huber Corp. ISC Alloys Ltd. Mearl Corp. Minerals Pigments Corp. Mobay Chemical Corp. NJZ Colors NL Chemicals/NL Industries, Inc. Pfizer, Inc., MPM Div. PPG Industries, Inc. Reichard-Coulston, Inc. Sherwin-Williams Chemicals

#### PIGMENTS, ORGANIC

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#### PIGMENTS, METALLIC

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#### PROPELLANTS

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### OTHER CONVENTION INFORMATION

#### PAINT INDUSTRIES' SHOW

The 50th Anniversary Paint Industries' Show will be held in conjunction with the Annual Meeting. The 1985 Paint Show is the largest, by far, in Federation history with 240 companies occupying over 56,000 sq. ft. of exhibit space at Cervantes Convention Center.

The Show—biggest and best of its kind in the world—presents unique and attractive displays devoted to a wide variety of raw materials, production equipment, containers, laboratory apparatus and testing devices, and services furnished to the paint and coatings manufacturing industry.

The purpose of the Show is to enable registrants to learn of the latest developments in these products and services and also to provide the opportunity to discuss them with the top technical/ sales personnel of the exhibiting firms.

Exhibit hours will be 12:00 to 5:30 pm on Monday, October 7; 9:30 am to 5:30 pm on Tuesday, October 8; and 9:30 am to 3:00 pm on Wednesday, October 9.

#### **REGISTRATION FEES**

Advance registration is available for \$50 for members and \$65 for non-members. Fee for spouses' activities is \$35 in advance.

There is a special registration fee of \$25 each for retired members and their spouses. This applies to advance registration only.

On-site registration is \$60 for full time and \$40 for one day for members. Non-member fees are \$75 for full time and \$50 one day. Spouses registration is \$45 on-site.

#### SPOUSES PROGRAM

The spouses program on Monday will be a wine 'n cheese social in Hall A of the Convention Center. There will be giveaway and door prizes, courtesy of several supplier companies. The continental breakfast on Tuesday and Wednesday will be in the ballroom of the Sheraton. A tour of outstanding sites in St. Louis and a luncheon will comprise the Spouses Tour on Tuesday.

#### FEDERATION ANNUAL AWARDS LUNCHEON

The Federation Awards Luncheon will be held Wednesday at the Sheraton St. Louis 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 1985 Paint Industries' Show), the Roon Awards Competition awards, other Annual Meeting Awards and special presentations.

Featured speaker at the Luncheon will be Jim Valvano, Head Coach of the North Carolina State University basketball team.

In commemoration of the "Big 50" Golden Jubilee Paint Show, 50-year members of the Federation present in St. Louis will be among the honorees. They will be seated in a special area and will be introduced.



#### **CO-HEADQUARTERS HOTELS**

Co-headquarters for the Annual Meeting will be the Sheraton St. Louis Hotel and the Marriott Pavilion Hotel. Other hotels with blocks of rooms set aside for the Annual Meeting are: Bel Air Hilton, Best Western, Clarion, Embassy Suites, Holiday Inn Market St., Holiday Inn Riverfront, Mayfair, Omni-Union Station, Radisson St. Louis, Rodeway Inn, and University Plaza.

#### PROGRAM STEERING COMMITTEE

Joseph A. Vasta, of the DuPont Co., F&FP Dept., Wilmington, DE, is the Chairman of the Federation's Program Committee.

Assisting Chairman Vasta in development of the Annual Meeting program are: Percy Pierce (Vice-Chairman), PPG Industries, Inc., Allison Park, PA; Taki Anagnostou, Akzo Coatings America, Inc., Troy, MI; Richard Eley, Glidden Coatings & Resins Div., SCM Corp., Strongsville, OH; William H. Ellis, Chevron Research Co., El Segundo, CA; James Lee, Lanchem Corp., E. St. Louis, IL: Stanley LeSota, Rohm and Haas Co., Spring House, PA; John S. Ostrowski, Valspar Corp., Minneapolis, MN; and David Richie, Midland Div. of Dexter Corp., Waukegan, IL.

#### HOST COMMITTEE

The Federation's two Societies in Missouri (St. Louis and Kansas City) will serve as the official hosts for the Annual Meeting and Paint Show. The General Chairman is Howard Jerome, of Spatz Paint Industries, Inc. Subcommittee Chairmen are: Spouses—Mrs. Howard (Gene) Jerome; Program Operations— John Folkerts, of Future Coatings; Registration Area—Joseph W. Wrobel, Jr., of Ciba-Geigy Corp.; Information Services—William Reckel, of Amteco, Inc.; and Liaison—Nick Dispensa, of Davis Paint Co.

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1985 Annual Meeting Paint Industries' Show October 7, 8, 9 Cervantes Convention Center St. Louis, MO

### OPENING SESSION Monday, October 7

#### **Keynote Address**

"Tung Oil to Group Transfer Polymerization" John P. McAndrews

An industry leader, John P. McAndrews is Group Vice-President— Finishes and Fabricated Products of E.I. du Pont de Nemours & Co., Inc., Wilmington. Joining du Pont in 1947, Mr. McAndrews has held the positions of Laboratory Manager, Director of Fabric Sales Div., Assistant Director of Marketing for Automotive and Industrial Products, Director of Marketing and, later, President of Remington Arms, and was named to his current position in 1983.



John P. McAndrews

### MATTIELLO LECTURE Wednesday, October 9



Ruth M. Johnston-Feller

#### "Reflections on the Phenomenon of Fading" Ruth M. Johnston-Feller

Ruth M. Johnston-Feller has more than 30 years' experience in industrial color technology. Author of more than 45 technical articles, Mrs. Johnston-Feller has taught numerous courses on color and has lectured on color science in many universities. She is the recipient of the FSCT's Armin J. Bruning Award, Dry Color Manufacturer's Award, and the Inter-Society Color Council's Macbeth Award. Mrs. Johnston-Feller is currently a consultant in color science to the Mellon Institute at Carnegie-Mellon University, Pittsburgh.

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#### Guidelines for using HALOX pigments for greater corrosion resistance or tannin-stain blocking.

They're at the 1985 Paint Show, Booth 1517, Cervantes Convention Center, St. Louis, MO, October 7, 8, 9.



#### HALOX PIGMENTS

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### For more information on HALOX rust inhibitive pigments

If you are not going to this year's Paint Show, here's how to get detailed information on any of Halox's full line of pigment products, including product data sheets, formulations, application information and research reports. Your letterhead request will bring an immediate response. Or contact the HALOX Pigment representative nearest you:

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Houston, TX

713-688-7722

405-685-1746

Phoenix, AZ

602-894-9761

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Western New York Call Pittsburgh Collect 412-367-8990

J.W. Copps, Inc. Menomonee Falls, WI 414-251-3825

Corbet Chemicals Inc. Newark-New York 201-992-6981

Cypress Color & Chemical, Inc. Boston, MA 617-329-8424

Dorsett & Jackson, Inc. Los Angeles, CA 213-268-1815

F.R. Hall, Inc. St. Louis, MO 314-725-2600

Kansas City, MO 816-221-6713

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E.W. Kaufmann Co. Baltimore, MD 301-321-0458

Kraft Chemical Company, Inc. Chicago, IL 312-345-8210

Mehaffey and Daigle, Inc. New Orleans, LA 504-733-7831

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### Government and Industry

#### Merger and Acquisition Activity Up In Chemicals, Paints & Coatings Industry

Merger/acquisition activity in the Chemicals, Paints & Coatings industry increased 44 percent in 1984, reports W. T. Grimm & Co., a leading merger and acquisition consultant, in its "1984 Mergerstat Review."

The firm's annual comprehensive 205page guide to merger and acquisition activity revealed that the number of such transactions in the Chemicals, Paints & Coatings industry rose from 55 in 1983 to 79 in 1984, ranking the industry 11th in W. T. Grimm's 50-industry list. In terms of dollar values, the industry ranked tenth in 1984, rising from \$1.1 billion in 1983 to \$2.6 billion in 1984.

Because commodity chemicals producers are plagued by excess plant capacity, slow-changing technology, and foreign competition, they are diversifying into the more profitable specialty chemical market and divesting bulk oriented product lines. The five largest chemical transactions in 1984 were all divestitures. Beatrice Cos. sold its chemical operations to Imperial Chemical Industries PLC of Britain for \$750 million. Liquid Carbonic Corp., a subsidiary of Houston Natural Gas Corp., was sold to CBI Industries, Inc. for \$407 million. Georgia-Pacific Corp. sold some commodity-chemical operations to a management group for \$270 million. SCM Corp. acquired the titanium dioxide producing units of Laporte Industries PLC of Britain for \$125 million, and Superfos AS of Denmark acquired Royster Co. from Universal Leaf Tobacco Co. for \$113 million.

Headquartered in Chicago, W. T. Grimm & Co. has specialized in the merger, purchase or sale of companies for over 30 years. For more information about its "Mergerstat Review," write Research Department, W. T. Grimm & Co., 135 S. LaSalle St., Chicago, IL 60603.

#### NL Industries Acquires Spencer Kellogg

NL Industries, New York, NY, and Textron, Inc. have signed a definitive agreement providing for the acquisition of Spencer Kellogg, a division of Textron, by NL Industries, Inc. for an undisclosed sum. The agreement is subject to normal governmental review.

Spencer Kellogg will be operated as part of NL Chemicals, a leading worldwide supplier of titanium dioxide pigments and specialty chemicals. Headquartered in Buffalo, NY, Spencer Kellogg is a leading supplier of specialty resins to the coatings, inks, electrical and adhesives industries.

#### Akzo Enters R&D Agreement with Metal Coatings, Inc.

In response to automakers' requirements for car bodies to resist rust-through for up to 10 years, a joint research and development agreement aimed at improving the corrosion resistance of automotive sheet steel has been announced by Akzo Coatings America, Troy, MI, and Metal Coatings International, Cleveland, OH.

Akzo and Metal Coatings currently provide a coatings system for corrosion resistant steel called Zincrometal<sup>®</sup>, which is cold rolled steel coated with Metal Coatings' Dacromet<sup>®</sup>, a chromium-based basecoat treatment, and Zincromet<sup>®</sup>, a weldable, zinc-rich primer topcoat developed by Akzo. The joint research effort seeks to: develop a new family of coated steels with improved corrosion resistance; further refine the manufacturing process of Zincrometal<sup>®</sup> to accommodate continuously annealed steels; and improve the manufacturing characteristics of the final coated steel products.

#### Hercules Constructs New Resin Facility

Hercules Incorporated, Wilmington, DE, has begun construction of a new C-5 hydrocarbon resin facility at its existing Jefferson hydrocarbon resin complex located near Pittsburgh, PA.

This plant addition is the most recent in a series of consolidations undertaken to transfer hydrocarbon resin production facilities from the firm's previous site at Clairton. Completion of construction is expected by the end of the third quarter, 1986.

#### **Du Pont Expands Automotive Development Center**

To strengthen its support of the worldwide automotive market, Du Pont Co., Wilmington, DE, will expand its Troy, MI, Corporate Automotive Development Center. New facilities include a building for the development of technology to make thermoplastic auto body parts, an office and applications complex to house the company's refinish training center, and a state-of-the-art, on-line coating facility.

The enlarged center will enable Du Pont to focus on materials and systems for the development of polymeric auto body parts and new finishes.

"The consolidation brings together Du Pont's broad base of automotive expertise in polymers, finishes, fibers, and advanced composites for structural parts and electronics," said Henry B. Milligan, Director of Automotive Development. He added, "It will enable us to more fully meet the rapidly changing technology and product needs of the automotive industry."

The firm's Refinish Training Center, established in 1965, will be moved from Flint, MI, to the Troy Center. An applications testing facility for the experimental spraying of panel and parts of cars will be included in the new training complex.

#### International Paint Opens U.S. Powder Coating Plant

International Paint-Powder Coatings recently opened its first powder coatings plant in the U.S. Headquartered in London, England, International Paint has 44 factories and operations in 29 countries, producing high-technology, marine, protective and industrial coatings. Additional powder coatings plants are located in England, France, Germany, Brazil, Australia, and Korea.

#### ICI Americas Purchases Pigment Dispersions, Inc.

ICI Americas, Wilmington, DE, has acquired Pigment Dispersions, Inc. (PDI) of Edison, NJ. PDI is a privately-owned manufacturer of specialty dispersions for urethanes and thermoset materials and liquid colorants for thermoplastics. It will become a business unit of ICI Americas Specialty Chemicals business. The current management of PDI, under the direction of Frank E. Eisenhower, will continue to operate the business within ICI Americas.

#### Union Carbide Names Nuodex As Paint Thickener Distributor

Union Carbide Corp., Danbury, CT, has appointed Nuodex, Inc. as national distributor of its cellulosic and synthetic paint thickeners. Nuodex, which has offices in Piscataway, NJ; Chicago, IL; and Commerce, CA, will distribute Cellosize hydroxyethyl cellulose and UCAR SCT associative thickeners to the coatings industry.



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### FSCT and Coatings Industry to Celebrate 50 Year Paint Show Anniversary in St. Louis

From the first Paint Show in 1932, where 25 exhibitors showed their products and wares to the Federation members attending the Annual Meeting at The Mayflower in Washington, D.C., to the Fiftieth Paint Show, which will feature the goods of over 200 exhibitors at the Convention Center in St. Louis, October 7-9, 1985, the Federation's Paint Industries'Show has provided an informational mecca for the international coatings community.

Over the years the Paint Show has grown substantially to become the biggest and best of its kind in the world, presenting beautiful exhibitor displays devoted to the wide variety of raw materials, production equipment, and services to the paint and coatings manufacturing industry.

Attendees from almost every state in the Union, from Canada and Mexico, and from 45 other foreign countries will gather in St. Louis in October to celebrate the Fiftieth Anniversary of the Paint Industries' Show.

Following is a listing of the locations of the Annual Meeting and Paint Industries' Show of the FSCT:

1932-Washington, DC 1933-Chicago 1934-Washington, DC 1935-Washington, DC 1936-Chicago 1937-Cincinnati 1938-Atlantic City 1939-Chicago 1940-Washington, DC 1941-Chicago 1942-1945-Cancelled due to World War II 1946-Atlantic City 1947-Atlantic City 1948-Chicago 1949-Atlantic City 1950-Chicago 1951-Atlantic City 1952-Chicago 1953-Atlantic City 1954-Chicago 1955-New York 1956-Cincinnati 1957-Philadelphia 1958-Cleveland 1959-Atlantic City 1960-Chicago 1961-Washington, DC 1962-St. Louis 1963-Philadelphia 1964-Chicago 1965-Atlantic City





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DISULFONIC ACID
# DSC and DMA Analysis of the Curing Of a Liquid Epoxy Resin with Diamines

A. Toussaint, P. Cuypers, and L. D'Hont Coatings Research Institute (CoRI)\*

The curing of a liquid epoxy resin with primary diamines is followed by Differential Scanning Calorimetry (DSC) and by Dynamic Mechanical Analysis (DMA).

The DSC experiments show that the kinetic parameters change with curing temperature, extent of conversion of the reactive groups, and the presence of catalysts.

The DMA experiments point out that the curing process seems to proceed through five steps: microgel formation, macrogel formation, macroscopic gel point, (macroscopic crosslinking), and vitrification. But, depending upon the curing conditions, some of these steps may occur simultaneously and hence may not be observed separately.

#### INTRODUCTION

Increasing attention has been devoted recently to the technological properties of crosslinked resins in relation to the initial components, their stoichiometric ratio, and the conditions of the curing procedure. This has occurred mainly in the field of the so-called high solids paints or solventless paints. It is now well established that the formation of a high crosslinked film, characteristic of high solids paint types, is a very complex phenomenon leading to films having, in most cases, inhomogeneous structures. These inhomogeneities affect, just as pigments do, the physical properties of the films (the coatings) and depend on the chemical structure of the coreactants as well as the curing conditions.<sup>1-6</sup>

Some studies have shown that polycondensation reactions, in which molecules of functionality higher than three are involved, can lead to microgel formation of colloidal size, often incompatible with the reactional medium, either immediately or after having attained a certain size. Compatibility and size are dependent on the monomer size and chemical nature, and on the curing conditions. These latter conditions are clearly seen on the TTT diagrams given by Gillham<sup>4</sup> showing the gel, vitrification, and phase separation times versus curing temperatures.

The curing conditions are of great importance for the crosslinking density, for the morphology of the network being formed, and consequently, upon the mechanical properties. It should be of interest for the industry to be able to determine the optimal curing conditions (time and temperature) granting the desired physical properties and, particularly the mechanical ones, by easy and safe methods.

In this work we have used Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA) to follow the extent of the curing reaction and the development of the network structure.

#### DIFFERENTIAL SCANNING CALORIMETRY (DSC) EXPERIMENTS

#### Materials

The products used were: EPOXY RESIN: Araldite GY 250 (Ciba-Geigy)



<sup>\*</sup>Avenue Pierre Holoffe, B-1342, Limelette, Belgium.



Figure 1-Typical shape of a dynamic thermogram

equivalent weight was determined by oxirane group analysis = 207 g. The n value was determined by NMR analysis = 0.1.

DIAMINES: Isophorone diamine (IDP) from Veba-Chemie AG. = cycloaliphatic diamine, mixture of two stereoisomers. Equivalent weight: 42.6 g.



Trimethyl hexamethylene diamine (TMD) from Veba-Chemie AG.: mixture of two isomers (2,2,4 and 2,4,4). Equivalent weight: 39.6 g.



CATALYST: 2,4,6 -tris (dimethyl amino methyl phenol) (DMP30)



#### Instrumentation and Experimental Descriptions

The instrument used was the Mettler DSC 30 coupled with a TM Mettler TC10 programmable microprocessor and a Print Swiss Matrix printer. It was used to determine the enthalpy and the kinetic parameters (overall order, activation energy, front factor) of the reaction.

With few exceptions, we used equivalent amounts of epoxy and amines, knowing that an epoxy group is monofunctional towards one active hydrogen. Thermograms were recorded at a heating rate of 10° min<sup>-1</sup> from 0°C to 250°C, the temperature at which no more exotherms are detectable. Glass transition temperatures of the samples were determined after their curing by lowering the temperature to  $-20^{\circ}$ C (or lower) and then scanning at a heating rate of  $10^{\circ}$  min<sup>-1</sup>.

#### Thermograms and Kinetic Parameters Calculations

The determination of the kinetic parameters is usually done by isothermal methods, but this requires numerous thermograms and therefore makes the method tedious. On the other hand, enthalpy measurements are often tarnished by errors in such a way that part of the evolved reaction heat is not recorded or measured until the sample has attained its equilibrium temperature. These errors are the greatest with very reactive systems or very slow reactions. These are the reasons why the tendency is to determine the kinetic parameters by means of a single thermogram recorded by scanning calorimetry7-12 (dynamic method).

All the kinetic measurements are based on the fundamental equation (1) relating the conversion rate of the reactive species  $d\alpha/dt$ , at constant temperature, to a function of concentration and a rate constant k:

$$= k_T \cdot f(\alpha)$$
 (1)

 $(d\alpha/dt)_{\tau}$ where  $\alpha$  is the degree of conversion defined by:

$$\alpha = \frac{C_o - C_t}{C_o}$$
(2)

with Co and Ct, the concentration of the reactive species at time o and time t, respectively. a is time and temperature dependent and varies from 0 to 1.  $f(\alpha)$  is usually expressed as:

$$f(\alpha) = (1 - \alpha)^n \tag{3}$$

and k<sub>T</sub> by an Arrhenius expression:

$$k_{\rm T} = k_{\rm o} \cdot \exp\left(-Ea/RT\right) \tag{4}$$

where: n = the reaction order

 $k_o =$  the preexponential factor in s<sup>-1</sup>

- Ea = the activation energy  $(J \cdot mole^{-1})$
- R = the gas constant (8,314 J·mole<sup>-1</sup>· $^{\circ}$ K<sup>-1</sup>)
- T =the absolute temperature (°K).

If the recorded heat flow is only due to the phenomenon being studied, we may assume that the evolved heat rate (dh/dt) is directly proportional to the reaction rate (or to the conversion rate  $d\alpha/dt$ ). It follows that the area under the curve of the DSC thermogram is at every moment proportional to the conversion degree, which means to the reacted groups. A dynamic DSC thermogram has the typical shape shown in Figure 1, in which ho is the total heat evolved during the curing process, h is that produced up to time t. It must be pointed out that ho is not necessarily the maximum heat, hom, corresponding to a state in which all the reacting groups have been consumed (for the stoichiometry used). This is the reason why we must be careful when calculating the conversion degree. At any instant the conversion degree is given by

0

$$\alpha = h/h_{om}$$
(5)

but one can define a relative conversion degree by

$$r_r = h/h_o$$
 when  $h_o < h_{om}$ . (6)

The acquisition system of the Mettler instrument (T, t, dh(T,t)/dt), calculates  $k_o$ , Ea, and n of the best fit to the experimental results by a regression technique. The instrument is also capable of calculating the reaction enthalpy and the degree of conversion under given experimental conditions.

#### The DSC-treatment is strictly valuable only if:

(1) the Arrhenius law, valid for one isotherm, is also valid at other temperatures or at least in the recorded temperature range;

(2) the magnitude of  $f(\alpha)$  for a given value of conversion degree is temperature independent; in other words, the reaction mechanism is temperature independent;

(3) the reaction is of first or n<sup>th</sup> order; and

(4) the reagents are in equivalent amounts.

Also, because of the uncertainty about the fulfillment of the first requirements, the calculated kinetic parameters must be considered as apparent values and not as true ones. In fact, there is a basic mistake in the choice of the kinetic equation, since it is well known that the curing reaction of epoxy-amine can proceed through catalytic and autocatalytic processes. In that case, the kinetic equation must be rewritten, instead of equation (1), as<sup>13-15</sup> =

$$\frac{d\alpha}{dt} = k_1 c (1-\alpha)(r-\alpha) + k_1 \alpha (1-\alpha)(r-\alpha)$$
(7)

where

- $\alpha$  = fraction of epoxy group having reacted at time t,
- c = catalyst concentration,
- r = initial ratio between active hydrogens and epoxy groups,

 $k_1$  and  $k_2$  = rate constants.

#### **Experimental Results**

ENTHALPY OF REACTION: It is well known that for systems with mean functionalities higher than two, the reaction stops before it comes to completion.<sup>16-18</sup> The main reason for this is the difficulty for the reactive species to diffuse through the polymer matrix once the gel point is reached. If one wants to gain complete reaction of one type of reactive group, it is necessary to use an excess of the other reactive group. The optimum composition, expressed by r = initial ratio between active hydrogens and epoxy groups, is the one which liberates the greatest amount of heat. This optimum composition also gives us the enthalpy of reaction  $\Delta H_o$  of one epoxy group with H amine. *Table* 1 summarizes the experimental results.

In *Table* 1, as well as in those following,  $h_o$  is the heat of reaction  $(J \cdot g^{-1})$ ,  $\Delta H_o$  the reaction enthalpy per epoxy equivalent  $(kJ \cdot eq^{-1})$ , C the degree of conversion calculated for the epoxy groups (taking the value of  $h_{om}$  equal to 466 Jg<sup>-1</sup>), n the apparent reaction order, E and lnk<sub>o</sub>, respectively, the activation energy (kJ.mole<sup>-1</sup>) and the

#### Table 1—Experimental Kinetic Parameters

PD/GY 250 (without catalyst)	h <sub>o</sub>	$\Delta H_o$	с	n	Е	lnk <sub>o</sub>	Tg (°C)
r = 1	366	91.31	0.73	2.72	84.33	22.04	116
r = 1.1	433	110	0.93	3.01	97.75	25.73	138
r = 1.3	466	125	1.00	2.72	97.79	25.70	134
$r = 1.5 \ldots$	445	119	0.95	2.05	92.50	24.63	122
TMD/GY 250 (without catalyst)							
r = 1	398	98.15	0.80	1.96	92.82	25.11	71
r = 1.1	442	113	0.94	1.95	91.61	24.86	98
r = 1.3	467	122	1.00	1.81	87.67	23.77	97
$r = 1.5 \ldots$	454	118	0.99	1.32	83.48	22.67	77

Napierian logarithm of the front factor (s<sup>-1</sup>). The reaction order is calculated for the range  $\alpha = 0.1$  to 0.8.

#### Conclusions

The maximum heat evolved per equivalent epoxy is obtained for r = 1.3 in both cases. This has been verified by I.R. spectroscopy (910 cm<sup>-1</sup>) which confirmed the complete disappearance of epoxy groups for r > 1.2, whereas the peak is still observable for r < 1.2.

For r = 1, the degree of conversion is about 80%.

 $\Delta$ H per equivalent epoxy has practically the same value in both cases and these values are in agreement with those found in the literature.<sup>7, 9, 18</sup>

The kinetic parameters n, E, and  $lnk_o$  are dependent on the stoichiometric ratio r. The order diminishes with increasing value of r, indicating either a modification in the reaction mechanism or the non-validity of the basic kinetic equation.

The Tg of the films has the highest values for  $r \approx 1.1$ . This is normal since for r < 1.1, the crosslinking is incomplete and for r > 1.2, an excess of NH<sub>2</sub> or NH groups is present which suggests the existence of branched or dangling chains, having a plastifying effect.

We must emphasize that the reaction between epoxy and diamine is a very complicated one and much work has been devoted to its comprehension.<sup>18-26</sup> The classical interpretation is the following: the non-catalyzed reaction leads to the opening of the oxirane ring with formation of a secondary hydroxyl group and a secondary amine hy-

Table 2—Influence of Catalyst (DMP30) Content on the
Kinetic Constants

IPD (%	/GY 250 Catalyst)	h	ΔH	с	n	Е	lnk <sub>o</sub>	Tg (°C)
0		366	91.31	0.73	2.72	84.33	22.04	116
1		376	93.82	0.75	2.71	104.60	28.53	134
5		425	106.09	0.85	2.40	109.24	30.11	111
10		400	99.85	0.80	1.24	99.69	26.95	86
тм	D/GY 250							
0		398	98.15	0.80	1.96	92.22	25.11	71
1		405	99.87	0.82	1.49	81.87	21.53	75
5		429	105.79	0.87	1.25	81.17	21.21	88
10		405	99.87	0.82	0.78	80.22	20.87	71

#### Table 3—Influence of the Heating Rate on the Apparent Kinetic Constants

r=1 (without catalyst)	h	Δ <b>H</b>	с	n	Е	Inko
2 K.min <sup>-1</sup>	282	69.54	0.57	1.77	103.25	33.33
5 K.min <sup>-1</sup>	400	98.64	0.81	1.72	84.73	23.08
10 K.min <sup>-1</sup>	398	98.15	0.80	2.00	90.70	24.63
20 K.min <sup>-1</sup>	410	101.10	0.83	1.95	85.52	22.45
r=1 (with catalyst)						
2 K.min <sup>-1</sup>	296	72.99	0.60	0.54	98.57	28.32
5 K.min <sup>-1</sup>	413	101.85	0.83	0.46	73.40	18.84
10 K.min <sup>-1</sup>	408	100.62	0.82	1.15	81.73	21.37
20 K.min <sup>-1</sup>	401	98.89	0.79	1.31	86.53	22.65
r = 1,3 (without catalyst)	h	ΔH	С	n	Е	Inko
2 K.min <sup>-1</sup>	398	102.86	0.84	1.47	97.37	27.75
5 K.min <sup>-1</sup>	427	110.36	0.90	1.63	87.52	24.04
10 K.min <sup>-1</sup>	444	114.76	0.94	1.57	83.92	22.48
20 K.min <sup>-1</sup>	464	121.48	0.99	1.60	75.85	19.47
r = 1,3 (with catalyst)						
5 K.min <sup>-1</sup>	425	109.84	0.90	0.91	71.89	18.62
10 K.min <sup>-1</sup>	464	119.92	0.98	0.83	65.24	16.20
20 K.min <sup>-1</sup>	410	105.97	0.87	1.12	72.57	18.63

System GY 250/TMD

drogen; the latter is still capable of reaction by opening another epoxy group to give rise to another secondary hydroxy group and a tertiary amine, so finally a crosslinked structure is formed.

It is well known that the reaction is catalyzed by the presence of hydroxyl bearing groups (alcohols, phenols) and in general by all those having an electrophilic or proton donating character (water, Lewis & Brønsted acids). In the absence of catalysts, the reaction acquires a pronounced auto-catalytic character because of the formation of the secondary hydroxyls. There also can occur a competitive catalytic effect between hydroxylic and amine hydrogens as well as hydrogen bond formation between them; this leads to a reduction of either the catalytic effect or the reactivity of the amine. Furthermore, tertiary amines are known to be catalysts for secondary reactions, such as etherification and self-polymerization of epoxy groups, especially when aliphatic amines are used.

In summary, reaction rates and reaction mechanisms (and, therefore, the reaction order) will be dependent on the chemical nature of the amines used, on the nature and level of catalysts, on the stoichiometry of the system, and on the curing temperature. Formally, the order of an autocatalyzed reaction is three, whereas it is two for a catalyzed system.<sup>24</sup> Practically, however, it has been shown with model molecules that the order does vary with temperature<sup>21</sup> from 1.8 to 1.2, with the temperature lying in between 10 and 60°C. Experience shows that in normal conditions primary and secondary amines have very similar reactivities<sup>26, 13</sup> and that ether linkage formation is nearly absent with aliphatic amines and catalyzed systems.<sup>27, 28</sup>

INFLUENCE OF THE CATALYST CONTENT: Results are reported in *Table* 2 for systems where r = 1, and catalyzed with DMP 30 at various levels.

The conversion degree increases with the catalyst level to reach a maximum of 5% and then decreases slightly. The same affords for the activation energy.

The order of the reaction decreases with the increase of the catalyst level.

It must be pointed out that the dynamic thermograms of the TMD systems are characterized by nearly symmetrical peaks whereas those obtained with uncatalyzed IPD systems show asymmetrical peaks which are the superposition of two exotherms and leading to the conclusion that two types of reaction occur, each one probably with different kinetic parameters. This phenomenon tends to weaken with the increase of catalyst. This will by no means signify that the reaction becomes simpler, but that the catalyst has changed the kinetics of the reactions.

INFLUENCE OF THE HEATING RATE: The experimental results are reported in *Table 3*. Four heating rates were studied. The apparent order of reaction tends to increase with increasing heating rate whereas the front factor and the activation energy pass through a minimum or decrease.

VARIATION OF THE REACTION ORDER WITH THE DEGREE OF CONVERSION: The overall (or apparent) reaction orders were calculated for three conversion degree intervals:  $\alpha_1$ (0.1-0.8);  $\alpha_2$  (0.1-0.5);  $\alpha_3$  (0.5-0.8) which correspond respectively to the overall order, the one before the gel point, and the one after the gel point (the gel point for a system with r = 1 is calculated using the equation of Macosko and Miller<sup>28</sup>: it is attained for a conversion degree  $\alpha = 0.577$  in epoxy or amine hydrogens). The results are reported in *Table* 4 for the system GY250/ TMD as examples and show great differences between them.

#### DYNAMIC MECHANICAL ANALYSIS

If the DSC gives us information on the kinetics of the crosslinking reactions, it does not give us information on the network structure, nor on the resulting mechani-

Table 4—Values of the Kinetic Parameters in a Dynamic Experiment for Different Values of Intervals of the Conversion Degree

GY250/TMD	α <sub>1</sub> = <b>0.1-0.8</b>		$\alpha_2 = 0.1 - 0.5$			$\alpha_3 = 0.5 - 0.8$			
r = 1	n	E	Ink <sub>o</sub>	n	E	lnk <sub>o</sub>	n	E	Inko
uncatalyzed	1.94	98.54	26.31	1.46	85.10	22.33	2.98	175.30	51.38
catalyzed	1.18	88.70	22.05	0.53	67.82	16.58	2.04	149.73	43.77



Figure 2—Evolution of the dynamic properties of GY250/IPD system in stoechiometric amount without catalyst during isothermal curing at 50°C

cal properties. It is therefore also very useful to follow continuously the variation of the rheological properties during curing. One knows that viscosity and dynamic mechanical properties are particularly sensitive to the increase of molecular weight, to chain entanglement, and to the crosslinking density.

#### Instrumentation and Experimental

We used a viscoelasticimeter "Metravib" working at eight fixed possible frequencies: 7.8, 15.6, 31.2, 62.5, 125, 250, 500, and 1000 Hz. The measuring cell can work between -100 and +250°C. The instrument calculates the rigidity complex modulus and the phase angle. These values allowed us to compute the components of the complex shear modulus G which is directly related to the viscosity  $\eta$ . The measuring cell used was a cylindrical pot sealed on the base of the instrument and filled with the liquid mixture to be examined. A centered cylindrical piston plunges in this pot and moves up and down with given frequency and amplitude. All experiments were conducted isothermally.

#### **Experimental Results**

*Figures* 2 and 3 are examples of isothermal curves obtained showing variations of the real part G' and imaginary part G'' of the complex shear modulus  $(N \cdot m^{-2})$  and the loss tangent in function of time for the mixture GY250/IPD (r = 1) without catalyst and cured at 50°C



Figure 3—Evolution of the dynamic properties of GY250/IPD system in stoechiometric amount without catalyst during isothermal curing at 110°C

<b>Table 5—Values</b>	of k1 and k2 Versus Curing	Temperatures
	And Catalyst Levels	

	Systems							
Experimental Conditions	GY 250/IPD		GY 250/TMD					
without catalyst cure t°	k <sub>1</sub>	k <sub>2</sub>	k	ı k <sub>2</sub>				
21	$2.5.10^{-4}$ 4.166.1	$0^{-4}$	$4.86.10^{-4}$	$5.41.10^{-4}$				
50	$1.77.10^{-3}$ $1.361.1$	$0^{-3}$	$2.396.10^{-3}$	$1.299.10^{-3}$				
	$4.26.10^{-3}$ 1.822.1	$0^{-3}$	9.375.10-3	$2.50.10^{-3}$				
—110	1.25.10 <sup>-2</sup> 2.66.1	0 - 2	$1.83.10^{-2}$	$3.54.10^{-3}$				
with catalyst DMP30								
0.5 % 50	$1.208.10^{-3}$ $1.229.1$	$0^{-3}$	$2.25.10^{-3}$	$1.583.10^{-3}$				
1 % 50	$2.00.10^{-3}$ 1.666.1	$0^{-3}$	$3.33.10^{-3}$	$2.10.10^{-3}$				
5 % 50	2.33.10-3 2.958.1	$0^{-3}$						
10 % 50	$4.04.10^{-3}$ 3.5.10	) - 3	1777-1272	—				

and 110°C, respectively. One can see that:

(1) the G' curve has a double sigmoïdal shape;

(2) the first plateau (between  $t_1$  and  $t_2$ ) of the G' curve is all the better observable since the curing temperature is low. The curve is characterized by linear parts having different heights and slopes. The length of the intermediate plateau depends on temperature, on nature, and on amount of catalyst;

(3) the tg  $\delta$  decreases rapidly at the beginning from an infinite value ( $\delta = 90^\circ$ ) to a finite value and shows one or two maxima depending on the experimental conditions. These maxima are better marked the higher the curing temperature. The experimental points of the Figures are given in *Tables* 4 and 5. The reactants are mixed in equivalent ratio.

#### DISCUSSION

For a description of the symbols used in this discussion, see Appendix (also see *Figure* 2).

It is well known that the curing of thermosetting systems is a complex phenomenon leading to the formation of heterogeneous structures consisting of microgels whose dimensions and number are dependent on the curing conditions, linked together by many or few crosslinked chains.<sup>2, 5, 30</sup> Therefore, microgels can occur before the macroscopic gel point is attained, and this one can also occur for a higher degree of conversion than the theoretically calculated one. If we refer to *Figure* 2, the crosslinking process can be conceived as follows:

In a first step, between t = 0 and t = t<sub>o</sub>, the reaction leads to the formation of linear chains and chains of small dimensions, branched at both ends, that are elastic due to the increasing molecular weight. This raises the viscosity G'' (G'' =  $\eta \cdot 2\pi f$ ) and justifies the small increase of G'.

From  $t_o$  to  $t_1$ , elastic and loss moduli G' and G" increase drastically. During that period, the linear and branched chains continue to grow and some of the branched chains can at the same time react locally together or intramolecularly to form microgels of small dimensions. This results in a sudden raising of the viscosity

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(G") and of the elastic modulus (G'). At the same time a very drastic drop of the loss tangent,  $tg\delta$ , is observed, as expected. We define  $t_o$  as the induction time or the time corresponding to the first appearance of microgels. In this stage the material must be considered as a liquid phase in which microgels are dispersed but not yet as an infinite molecule loosely crosslinked. In fact, extraction tests (CH<sub>2</sub>Cl<sub>2</sub>, THF) showed that a small fraction of the medium was insoluble. Of course this insolubility increases with time, since reactions continue to occur at the surface of the microgels (increasing their dimensions) or inside the microgels by diffusion of reactive species.

In the third phase between  $t_1$  and  $t_2$ , the modulus G' and G' show no, or at least very little, variation. This can be explained as follows: some of the microgels, growing in number and size (diameter: 10-15  $\mu$ m) can react together either by external reactive groups and form aggregates or via a branched or linear extending chain. If this union suddenly increases the molecular weights, its influence on the mean molecular weight between crosslinks is very small. Consequently, the influence on the elastic modulus which depends on that is reduced. We believe that the formation of isolated aggregates should begin at  $t_1$  up to  $t_2$ . These aggregates can become insoluble in the medium leading to a phase separation phenomenon.

The fourth region is between  $t_2$  to  $t_3$ . At  $t_2$ , the number, size, and shape of the aggregates, as well as the dimensions of the other growing macromolecules and the degree of conversion, are such that they react with each other and gelation takes place. The medium transforms itself in a viscoelastic material. We call t2 the macroscopic or viscoelastic gelation time. One can associate the first maximum of the tg $\delta$  curve with the macroscopic gelation as Gillham does. Moreover one can see that the times t<sub>a</sub> and  $t_b$  at which  $tg\delta = 1$  are very close to  $t_1$  and  $t_2$ . It follows that the gel time can be indifferently defined as  $t_2$ ,  $t_b$  or  $t(tg\delta_{max})_1$ . This interpretation is justified by the fact that  $tg\delta$  starts to increase from  $t_4$  which is close to  $t_1$ . attains a maximum at  $t(tg\delta_{max})_1$ , close to  $t_2$ , and decreases afterwards. Furthermore, in the region between t<sub>1</sub> and t<sub>2</sub> we expect phase inversion: the oligomers are now dispersed in the gel phase. From t<sub>2</sub> and t<sub>3</sub>, extraction tests show that the material (network) swells and there is very little soluble material left. Up to t<sub>3</sub>, reactions between

Table 6—Comparison	Between	Times	(Minutes	) to Gelation
Determined by I	DMA and	Calcul	ated by D	SC.ª

	1	DMA	DSC experiments			
Systems	cure t°C	t <sub>2</sub>	t(tg $\delta$ = 1) t(tg $\delta_{max}$ ) <sub>1</sub>		1	2
GY 250/TMD without catalyst	50	56	54	47	402	204
-	80	12	12	-	18	14
GY/TMD with 1% DMP30	50	32	29	37	142	69
	80	8	11		10	9

(a) For the definition of  $t_2$ ,  $t(tg\delta = 1)$  and  $t(tg\delta_{max})_1$  see text. For DSC column 1: time calculated with the kinetic parameters calculated for the interval  $\alpha_1 = (0, 1{-}0.8)$ ; column 2: time calculated with the kinetic parameters calculated for the interval  $\alpha_2 = (0, 1{-}0, 5)$ 

reactive groups continue on, increasing the numbers of crosslinks and consequently the elastic modulus G'. It is the second ascending part of the curve. Crosslinking goes further until stopped at  $t_3$ , either by complete consumption of the reactive groups or by vitrification of the material depending on whether the curing temperature is above or below the Tg. This is also confirmed by the appearance of a second maximum in the curve tg\delta at t(tg\delta\_max)\_2, the quasi constancy of G', and the fall of G'' above  $t_3$ .

If a plateau (or a slow increase of G') between  $t_1$  and  $t_2$  is not observed in the curve (G', t), then either there is no microgel formation or the microgel formation and gelation occur at the same moment. This problem needs further study and is included in a future program. It also means that  $t_1$  and  $t_2$  are confused. In this case there are only one or two moments when  $tg\delta = 1$ . This unique value, or the first one if two exist, corresponds fairly well to what is happening at  $t_2$ .

#### Implication on the Physical Properties

After the gelation point and up to vitrification, the material can be considered as a rubber and therefore its elastic modulus is directly proportional to the effective number N of crosslinks per unit volume:

$$G' = \emptyset RTN$$
(8)

where  $\emptyset = a$  front (Tobolsky) factor;

R = the ideal gas constant;

T = the absolute temperature.

So far, the changes of G' versus time, in the time intervals  $(t_o, t_1)$  and  $(t_2, t_3)$ , which look to be linear, can be expressed mathematically as:<sup>29, 31</sup>

$$\frac{d (\lg G')}{dt} = \frac{d (\lg N)}{dt} = k_i$$
(9)

with i = 1 and 2 respectively for the linear part between  $t_o-t_1$  and  $t_2-t_3$ .

In these time intervals, the number of crosslinks increases exponentially. k is expressed in mole  $m^{-3} \cdot s^{-1}$ for N. However, the only value having a true significance is  $k_2$ . In fact, in the first ascending part of the curve,  $k_1$ does not have the same signification as  $k_2$ , because the medium is not yet an elastomer but is a microgel dispersion in a liquid medium. However, the  $k_1$  value is in some way related to the rate of formation and the number of microgels. Values of  $k_1$  and  $k_2$  can be found in *Table* 5 for two systems, catalyzed and non-catalyzed.

It can be seen from the analysis of the experimental results that:  $k_1$  and  $k_2$  increase with temperature;  $k_1$  increases more than  $k_2$ . Both systems studied, GY250/TMD and GY250/IPD, show a similar behavior, except that  $(t_2-t_1)$  is different.

The range  $(t_2-t_1)$  is smaller and decreases more rapidly with temperature for the system GY250/TMD than for the system GY250/IPD. This could mean that in the case of crosslinking with TMP, the formation of aggregates (time range  $t_1$  to  $t_2$ ) and the macroscopic gelation tend to occur simultaneously, thus giving rise to a film of more homogeneous structure, if not non-isotropic, than the one obtained with IPD.

The addition of catalyst only slightly increases k1 and

 $k_2$  in both cases. The effect is more sensitive with IPD than with TMD. This might be an indication of a different reactivity regarding the two amine functionalities, IPD versus TMD.

Catalysts reduce the times  $t_o$ ,  $t_1$ , and  $t_2$  but seem to have less influence on their intervals especially for  $(t_2-t_1)$ , characteristic of microgel formation. The value of G' is not changed at the vitreous plateau  $(t > t_3)$ . The times to gelation and vitrification are reduced by the use of catalysts.

#### COMPARISON OF GEL TIMES

*Table* 6 gives the gel times determined by DMA on one hand and those calculated for the system GY250/TMD by DSC, using the kinetic parameters previously determined for the conversion degree intervals ( $\alpha_1 = 0.1$ -0.8) and ( $\alpha_2 = 0.1$ -0.5) on the other hand.

One can see that there are no correlations between the gel times except for the results from the kinetic constants calculated for the interval ( $\alpha = 0.1$ -0.5) and those of DMA curing at 80°C (and higher t°). Regarding the fact that the kinetic parameters do vary with temperature, amount, and type of catalyst, and also with the extent of reaction, they should be used very carefully when predicting the curing process. Nevertheless it must be said that when calculating the extent of reaction, gel point, and so on, for isothermal curing conditions from kinetic parameters determined by dynamic DSC, the agreement is all the better the higher the curing temperature.

#### CONCLUSION

We have shown that the enthalpy of reaction  $\Delta H$  per equivalent epoxy has the same value for both systems (GY250/TMD and GY250/IPD). Conversion of the reactive groups is not complete when they are used in equivalent amounts although it increases with the heating rate in dynamic DSC experiences, and with the use of catalysts. Conversion has never been higher than 95-97%. The completion of the conversion can only be obtained for a ratio (equiv. active hydrogen/equiv. epoxy) r > 1, 1. Conversion is also a function of temperature (in isothermal curing) relative to the Tg of the fully cured resin; the more close the Tg and the temperature of curing are to each other, the higher the conversion.

The overall apparent kinetic constants, determined by dynamic DSC experiments, do vary with experimental conditions; the reaction order increases with heating rate while the frequency factor tends to decrease. They also vary with the stoichiometry and with the degree of conversion. This means that the kinetic constants can vary with both temperature and (during curing) degree of conversion, and can be quite different before and after the gel point is reached, depending on the isothermal curing temperature.

One must, therefore, be very circumspect when using the apparent kinetic constants determined by dynamic DSC to predict gel points and times required to reach a given degree of conversion in isothermal curing processes.

The shape of the curves lg G', lg G'' and tg  $\delta$  obtained

by means of DMA experiments allows us to assume a scheme for the crosslinking process during the isothermal curing of solventless epoxy-polyamine resins.

The solventless liquid mixture (epoxy-diamine) transforms itself into a solid through five steps, each characterized by a time:

- (1) microgel formation:  $t_o t_1$ ;
- (2) aggregation of microgels—macrogels:  $t_1 t_2$ ;
- (3) macroscopic gel point: t<sub>2</sub>;
- (4) macroscopic crosslinking:  $t_2 t_3$ ; and
- (5) vitrification point: t<sub>3</sub>.

These steps and related times are influenced by catalysts and the curing temperature, a feature that is important for the following reasons. The formation of microgels and their aggregation can lead to phase separation and appearance of microdomains. It is well known that the existence of microdomains has a great influence on the mechanical properties in the elastomer field. The same should likely hold in these cases. The formation of these domains and their growth is considered to continue until gelation (or vitrification) occurs. Of course, phase separation is also regulated by the compatibility of the growing molecules in the reactive medium, but is probably favored if cyclic structures are allowed to be formed. As pointed out by Gillham (reference 32, Figure 1b), it is easy to understand that different morphologies arise from cure at different temperatures, because of the influence of temperature on the competition between thermodynamic (compatibility) and kinetic factors (which are dependent on curing temperature and degree of conversion). For example, if curing is realized at temperatures above or near the  $Tg_{\infty}$  of the fully cured resin, no phase separation occurs. If microgels are nevertheless produced in the early reaction phase they will remain compatible with the medium and will be of very little dimension, finally dispersed in the bulk. On the other hand, curing below  $Tg_{\infty}$  can lead to phase separation with the number and size of the domains depending on the curing temperature and on the time to (reach) gelation. Once the gelation point is reached, the structure or the morphology of the film is fixed.

How these structural inhomogeneities are influencing paint properties, such as elasticity, hardness, flexibility, ultimate mechanical properties, abrasion resistance, impact resistance, and permeability, is still insufficiently known. These relationships will be studied in the near future, as will be the influence of pigmentation.

#### ACKNOWLEDGMENT

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#### APPENDIX —

- $t_o$  = the time corresponding to the intersection of the asymptote belonging to the first ascending branch G' and the horizontal to the abscissa axis for G' at time t = 0.
- $t_1$  = the time corresponding to the intersection between the asymptote of the first ascending branch of the curve G' and that one of the first plateau.
- $t_2$  = the time corresponding to the intersection of the asymptote to the first plateau and that one belonging to the second ascendant part of the curve G'.
- $t_3$  = the time corresponding to the intersection of the asymptote of the second ascendant part of the curve G' and the second plateau.
- $t(tg\delta_{max1}) = time$  at the first maximum of the tg\delta's curve.
- $t(tg\delta_{max2}) = that of the second maximum.$
- $t(tg\delta = 1) = times at which the tg\delta values take the value 1 i.e.,$ when G'=G''. Analogous times can be defined taking thecurve G'' as reference.

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TOLUENE	-	_	-	-	61	61	
XYLENE	50	50	-	_	_	_	
EE ACETATE	50	-	53	-	13	-	
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# Preparation of Acrylic Oligomers For High Solids Coatings Using Hydroxy-Functional Mercaptan Chain Transfer Agents

R.A. Gray Phillips Petroleum Company\*

Acrylic oligomers intended for application in high solids acrylic coating formulations were prepared by free radical solution polymerization using hydroxyfunctional mercaptan chain transfer agents. Hydroxyfunctional mercaptans that are commercially available or that could easily be made commercially available are emphasized.

Properties of the oligomers produced were investigated. These included molecular weight, polydispersity, solution viscosity, and residual odor. Properties of cured coatings resulting from high solids formulations of the oligomers and methylated melamine resins were also investigated.

#### INTRODUCTION

Pressure on the coatings industry to reduce emissions of volatile organic compounds has prompted considerable effort to develop high solids coating systems. Included are the thermoset coatings based on soluble hydroxyfunctional acrylic resins. One approach to producing higher-solids coatings is to utilize the same type polymers used in conventional systems but at reduced molecular weights. The many factors to be considered in such an endeavor are discussed in a comprehensive article on high solids coatings by Hill and Wicks.<sup>1</sup>

The low molecular weight hydroxy acrylic resins, or oligomers, for high solids coatings must yield coating formulations with viscosities low enough to be easily sprayed. In addition, the oligomers must contain a satisfactory number of chemically active hydroxyl groups to undergo subsequent chain extension and crosslinking when cured. Presumably, there is an optimum hydroxyl group content for the acrylic oligomers used in each high solids system. Whatever this number, certainly undesirable are oligomers that contain only one, or are completely devoid of, hydroxyl groups. Such oligomers are capable of neither chain extension nor crosslinking and may adversely affect the properties of the resulting coatings. Unfortunately, the probability of obtaining oligomers with inadequate hydroxyl content increases as the average molecular weight is reduced.<sup>1,2</sup>

Acrylic oligomers with suitable properties for high solids coatings can be obtained from free radical polymerizations using chain transfer agents, high polymerization temperatures, or a combination of both.<sup>3-6</sup> In our studies, we have investigated a specific free radical polymerization technique for the preparation of acrylic oligomers for high solids coatings. This technique involves the use of a highly effective mercaptan chain transfer agent that also contains hydroxyl functionality. We envisioned that this dual functionality would both reduce oligomer molecular weight and incorporate additional hydroxy functionality into a significant number of oligomer molecules.

The chain transfer reaction may be depicted as<sup>7</sup>

$$Mn' + XA \xrightarrow{K_{tr}} MnX + A'$$
(1)

where Mn<sup>•</sup> is a propagating polymer chain, XA is a chain transfer agent, X is the species transferred, A<sup>•</sup> is a new radical capable of reinitiating polymerization, and  $k_{tr}$  is the chain transfer rate constant. If XA in equation (1) is a

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chain transfer agent with a molecular structure such that A' contains a hydroxyl group, this functionality is imparted to the new propagating polymer chain. When A' is the predominant radical, then a substantial number of the new polymer chains should contain a hydroxyl group that originated as part of the chain transfer agent. An hydroxy-functional mercaptan should affect molecular weight reduction since it has been shown that the chain transfer rate constant for a hydroxy-functional mercaptan, such as 2-mercaptoethanol, is equivalent to that of an alkyl mercaptan, such as 1-mercaptobutane.<sup>8</sup>

The objective of this investigation was to determine the utility of hydroxy-functional mercaptans as chain transfer agents in the preparation of hydroxy acrylic oligomers for high solids coatings. This was accomplished by comparing hydroxy-functional with nonhydroxy-functional mercaptans. The comparison included evaluation of the mercaptans as molecular weight modifiers in the preparation of hydroxy acrylic oligomers and evaluation of the properties of coatings resulting from the oligomers.

#### EXPERIMENTAL

#### **Oligomer Synthesis**

The hydroxy acrylic oligomers were prepared by free radical solution polymerization under nitrogen using 2,2'-azobisisobutyronitrile (AIBN) as the initiator and a mercaptan chain transfer agent. Polymerizations were conducted in a multi-necked flask equipped with a thermometer, condenser, addition funnel, and magnetic stirrer while partially submerged in a hot oil bath. Monomers, AIBN, and chain transfer agent were combined in the addition funnel and added dropwise over a two-hour period to the refluxing solvent. After monomer addition, the final temperature was maintained for an additional two hours. Except for experiments designed to study the effect of polymerization temperature, the solvent used was methyl isobutyl ketone (MIBK), the initial temperature was 100°C (212°F) and the final temperature was 120°C (248°F). Other solvents used, along with the resulting polymerization temperatures, are noted when applicable.

Ingredients used in a typical polymerization were methyl methacrylate (MMA) (32.0g; 0.32 mole); butyl acrylate (BA) (35.9g; 0.28 mole); 2-hydroxyethyl methacrylate (HEMA) (20.8g; 0.16 mole); AIBN (1.65g; 0.01 mole); mercaptan chain transfer agent (0.01-0.06 mole); and MIBK (38.0g; 0.38 mole). When the HEMA concentration was changed, the same ratio of MMA to BA as given above was maintained. All monomers contained inhibitors added by the supplier and were used as received without further purification. Solvents and AIBN were reagent grade chemicals. The mercaptan chain transfer agents were either commercial samples or the distilled products of laboratory scale syntheses.

#### **Properties and Characterization of Oligomers**

Oligomer viscosities were determined at 23.9°C (75°F) with a Brookfield Viscometer, and oligomer molecular weights were determined by gel permeation chromatography (GPC) using polystyrene standards for calibration. The molecular weights of a select few oligomers were also determined by vapor-phase osmometry. Values by GPC were 1.4-1.5 times that obtained by osmometry.

#### **Coating Formulations**

Coating formulations were comprised of the oligomer solutions in MIBK, Cymel 303<sup>®</sup>\* crosslinking resin and p-toluenesulfonic acid catalyst. Catalyst concentration was 1.0 weight percent based on the crosslinking resin and was added as a 10 weight percent solution in 2butanol. Solids content of formulations ranged from 73.3 to 79.5 weight percent depending on the amount of Cymel 303 crosslinking resin employed.

#### **Preparation of Coatings**

The coatings were applied using a wire-wound applicator to untreated steel panels that had been coated with an epoxy-ester primer/surfacer. Following a five minute minimum solvent flash, the coated panels were placed in an air circulating oven at either  $120^{\circ}C$  ( $248^{\circ}F$ ) for  $140^{\circ}C$  ( $284^{\circ}F$ ) for 20 minutes. Cured coating thickness was nominally 1.0 mil. All cured coatings were aged at room temperature for a minimum of 48 hours prior to the determination of coating properties.

#### **Determination of Coating Properties**

The coating properties evaluated were hardness, flexibility, solvent resistance, and moisture resistance. Hardness was determined by pencil hardness and designated as the hardest pencil that failed to mar the surface. Flexibility was determined with a conical mandrel apparatus and reported as crack length in millimeters from the panel edge contacted by the small end of the mandrel. Solvent resistance was determined by rubbing the surface of the coating with a cotton swab saturated with methyl ethyl ketone (MEK) and noting the number of double rubs at which loss of coating gloss or integrity occurred. Moisture resistance was measured in a device closely resembling the Cleveland Humidity Chamber. In this apparatus, the coating was exposed to water vapor from 43.6°C (110°F) water which condensed on the coated side of the test panel. Exposed panels were inspected at regular intervals for loss of gloss or blistering.

#### **RESULTS AND DISCUSSION**

#### Mercaptans

The three hydroxy-functional mercaptans and two nonhydroxy-functional mercaptans selected for the investigation are listed below.

<sup>\*</sup>Cymel is a registered tradename of American Cyanamid Co.

NONHYDROXY-FUNCTIONAL MERCAPTANS: Methyl 3-Mercaptopropionate (MMP) CH<sub>3</sub>O<sub>2</sub>CCH<sub>2</sub>CH<sub>2</sub>SH; and 1-Mercaptodecane (MD) CH<sub>3</sub>(CH<sub>2</sub>)<sub>8</sub>CH<sub>2</sub>SH.

2-Mercaptoethanol (ME) was included since it is the most commonplace hydroxy-functional mercaptan. 3-Mercaptopropanol (MP) was selected because, unlike 2-mercaptoethanol, the functional groups are not attached to adjacent carbons and it is readily prepared by the addition of hydrogen sulfide to allyl alcohol. The choice of 2-hydroxyethyl 3-mercaptopropionate (HEMP) was based on the remoteness of the hydroxyl and mercapto groups, structural features in common with the monomers and the ease of preparation via the addition of hydrogen sulfide to 2-hydroxyethyl acrylate. Methyl 3-mercaptopropionate (MMP) was selected because it is not simply an alkyl mercaptan and because of its availability. 1-Mercaptodecane (MD) was chosen because it is a primary mercaptan with a boiling point in the desired range.

#### Effectiveness of Mercaptans As Molecular Weight Modifiers

The effectiveness of the five mercaptans as molecular weight modifiers was determined in polymerizations of 42.1/36.8/21.1 mole percent MMA/BA/HEMA containing a fixed concentration of initiator (AIBN) and increasing amounts of the mercaptan chain transfer agents. Figure 1 shows the oligomer molecular weights obtained with the five primary mercaptans plotted as a function of mercaptan concentration. For comparison, also included in Figure 1 are similar data resulting from the use of a tertiary mercaptan, 2-mercapto-2-methyl octane (t-nonyl mercaptan), which has a chain transfer constant in acrylic polymerizations of about one-half that of primary mercaptans.9 The primary mercaptans, whether hydroxy functional or not, were equivalent in their effectiveness as molecular weight modifiers and were definitely more effective than the tertiary mercaptan. Polydispersity of the oligomers decreased from 1.9 to 1.4 as the concentration of mercaptan chain transfer agent increased. Molecular weights and solution viscosities of the oligomers obtained using varying concentrations of MP as the chain transfer agent are shown in Table 1. In general, the molecular weight data is typical of that obtained with each of the five primary mercaptans.

The number-average molecular weight (Mn) of a polymer is the product of the number-average degree of polymerization (Pn) and the molecular weight of the monomer. The influence on the number-average degree of polymerization by a chain transfer agent is depicted by equation (2).<sup>7</sup>

$$\frac{1}{Pn} = \frac{1}{(Pn)_{o}} + C_{s} \frac{[S]}{[M]}$$
(2)

where  $(Pn)_o$  is the value of Pn in the absence of the chain transfer agent,  $C_s$  is the transfer constant of the agent, [S] is the concentration of chain transfer agent, and [M] is the concentration of the monomer. Thus a linear relationship should exist between the reciprocal degree of polymerization at constant initiator concentration and the chain transfer agent/monomer ratio. The transfer constant of the agent ( $C_s$ ) is given by the slope of this line.

#### Table 1—Molecular Weights and Solution Viscosities Of Oligomers Obtained Using 3-Mercaptopropanol (MP) As the Chain Transfer Agent

MP Concentration, mphm <sup>a</sup> Mw	Mn	Mw/Mn	Solution Viscosity <sup>b</sup> @23.9°C, (75°F), cps
0 20,900	11,400	1.9	19,400
1.3 10,800	6,000	1.8	3,850
2.6 7,200	4,300	1.7	1,875
3.9 5,700	3,500	1.6	1,300
5.2 4,400	3,000	1.5	720
6.6 3,600	2,400	1.5	460
7.9 3,100	2,200	1.4	300
(a) Moles per hundred moles of monomer (b) Essentially 70 weight percent solids.	ri		

A plot of 1/Pn versus [S]/[M] of the data in *Table* 1 is shown in *Figure* 2. In determining the Pn values, the molecular weight of the monomer used was 116 or the average molecular weight of the monomers in the three component mixture. As predicted by equation (2), a linear plot was obtained. The slope of the plot, or C<sub>s</sub>, of 0.52 is within the 0.4-0.8 range of values for methyl methacrylate polymerization reported for such primary mercaptans as 2-mercaptoethanol, 1-mercaptobutane, 1mercaptopentane, 3-mercaptopropionic acid, and methyl 2-mercaptoacetate.<sup>9</sup>

Molecular weight and solution viscosity data obtained from polymerizations that employed the highest level



Figure 1—Effect of mercaptan chain transfer agent on the molecular weight of oligomers obtained (Constant AIBN concentration)



Figure 2—Effect of 3-mercaptopropanol as the chain transfer agent on the degree of polymerization of the monomer mixture

(7.9 mphm) of each of the mercaptans are shown in *Table* 2. Except for solution viscosity, the properties of these oligomers are quite similar regardless of the mercaptan used. The higher solution viscosities of the oligomers obtained with the hydroxy-functional mercaptans are likely the result of increased hydroxyl functionality imparted to these oligomers by the mercaptans. The effect of oligomer hydroxyl content on viscosity is also apparent when the HEMA monomer concentration is increased from 21.1 to 34.2 mole percent in the polymerization recipe (*Figure* 3). Further, the viscosity increase in going from a nonhydroxy to a hydroxy-functional mercaptan appears greater when higher levels of HEMA monomer are used.

Although most polymerizations were carried out in refluxing MIBK at 110-120°C (230-248°F), a cursory investigation was conducted using only one of the mercaptans, HEMP, to determine the effect of polymerization temperature on molecular weight. For these studies, initiator and HEMP concentration were 1.3 and 7.9 mphm, respectively. Polymerization temperatures of approximately 80, 115, 150, and 170°C (176, 239, 302, and

Table 2—Molecular Weights and Solution Viscosities Of Oligomers Resulting From 7.9 mphm<sup>a</sup> Of Mercaptan Chain Transfer Agent

Mercaptan	Mw	Mn	Mw/Mn	Solution Viscosity <sup>b</sup> @23.9°C, (75°F), cps		
ME	3,200	2,100	1.5	320		
MP	3,200	2,100	1.5	300		
HEMP	.3,100	2,200	1.4	310		
MMP	3,100	2,100	1.5	200		
MD	. 3,200	2,100	1.5	160		

(a) Moles per hundred moles of monomer

(b) Essentially 70 weight percent solids.

#### Table 3—Best Coating Properties Obtained From Cured Oligomer/Cymel 303® High Solids Formulations<sup>a</sup>

Mercaptan	Chain	Transfer	Agent
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	Hydro	oxy-Fund	Nonhydroxy- Functional					
Coating Property	ME	MP	HEMP	MMP	ΜD			
Hardness, pencil	2H	3H	2H	Н	Н			
Solvent resistance, MEK double rubs	>200	>200	>200	150	150			
Flexibility, conical mandrel bend, length of crack, mm	0	0	0	0	0			
Moisture resistance, no apparent loss of gloss or blistering after 10 days	Pass	Pass	Pass	Pass	Pass			
(a) Cure conditions are 140°C (284°I	F) for 20 m	inutes.						

(a) Cute Conductors at 140 C (2017) for 20 minutes. Formulation: HEMA Concentration in Oligomers—21.1 mole percent. Oligomer to Cymel 303 Crosslinking Resin—2.33 to 1 by weight Solids Content of Formulation—77 weight percent.

338°F) were investigated by using the following respective polymerization solvents under reflux conditions: methyl ethyl ketone, MIBK, methyl amyl ketone and Carbitol<sup>®</sup>.\* As anticipated,<sup>3</sup> *Figure* 4 shows that the molecular weight and polydispersity of the oligomers obtained decreased with increasing polymerization temperature. Coatings were not prepared from the oligomers obtained at polymerization temperatures other than 110-120°C (230-248°F).

#### **Properties of Coatings**

Film properties were determined for coatings prepared from formulations containing acrylic oligomers and Cymel 303 crosslinking resin (commercial grade of hexamethoxymethylmelamine). Since the functionality of the oligomers and the ratio of oligomers to crosslinking resin can affect film properties, films were prepared from formulations that encompassed a range of both of these variables. The weight ratios of oligomer to crosslinking resin in the formulations varied from 5.6:1 to 1.5:1; and the mole percent HEMA used to prepare the oligomers varied from 13.8 to 34.2, with the amounts of MMA and BA adjusted to maintain a constant MMA/BA ratio.

Of the variations tried, the best coatings properties were obtained with oligomers containing 21.1 mole percent HEMA monomer and where the weight ratio of oligomer to crosslinking resin was 2.33:1 (*Table* 3). Formulations containing more crosslinking resin than the 2.33:1 ratio gave brittle coatings and those with less crosslinking resin yielded coatings with reduced hardness and reduced solvent and moisture resistance. Oligomers containing less than 21.1 mole percent HEMA produced coatings that exhibited reduced hardness and solvent resistance while those containing more than 21.1 mole percent HEMA gave coatings with reduced flexibility.

<sup>\*</sup>Carbitol is a registered tradename of Union Carbide Corp.

As the data in Table 3 show, coatings prepared using oligomers made with hydroxy-functional mercaptans exhibited superior hardness and solvent resistance compared to those made with nonhydroxy-functional mercaptans. One explanation for the better hardness and solvent resistance exhibited by the films prepared utilizing hydroxy-functional mercaptans is that they contain fewer oligomers with inadequate hydroxyl functionality. Coatings prepared from oligomers that are devoid of, or have insufficient, hydroxyl functionality tend to be softer and less solvent resistant due to inadequate crosslinking and due to the tendency of the noncrosslinked oligomers to act as plasticizers.<sup>1,10</sup> Thus, the use of hydroxy-functional mercaptans as chain transfer agents in the preparation of acrylic oligomers for high solids coatings may be a method of minimizing one of the major problems incurred with very low molecular weight hydroxy-functional acrylic oligomers: the presence of oligomers with inadequate hydroxyl functionality that adversely affect performance properties of the resulting coatings.

Although the majority of the coatings that contained oligomers prepared with the hydroxy-functional mercaptan chain transfer agents exhibited adequate hardness and solvent resistance, not all exhibited the flexibility of those shown in *Table* 3. Additional flexibility data are presented in *Table* 4 for coatings prepared using various cure temperatures, oligomer/crosslinking resin ratios, and HEMA monomer contents. This data indicates that the flexibility of the coatings was affected by the particular hydroxy-functional mercaptan used to prepare the oligomers employed.

The order in which the hydroxy-functional mercaptans were found to impart improved coating flexibility is HEMP>MP>ME. It is interesting to note that the intermolecular distance between the hydroxyl and mercapto groups for these three compounds follows the same order; the longest spacing is present in HEMP and the shortest in ME. Thus, the relative location of the hydroxyl group imparted by the hydroxy-functional mercaptan to the other hydroxyl groups in the oligomer will be greater for HEMP than for MP and greater for MP than for ME. Relative positions of the hydroxy functionalties in an



Solution Viscosity of Oligomers @ 23.9°C (75°F), cps

Figure 3—Effect of mercaptan chain transfer agent on solution viscosity

#### PREPARATION OF ACRYLIC OLIGOMERS



Figure 4—Effect of polymerization temperature on Mw of oligomers with 2-hydroxyethyl 3-mercaptopropionate (HEMP) as the chain transfer agent

oligomer that is subsequently crosslinked is known to be one of the factors that can influence the flexibility of the resulting coating.<sup>11</sup> Based on this, HEMP would be expected to produce coatings with the most elastically effective crosslinks and the best flexibility characteristics.

#### **Odor of Oligomers**

Odor is a property that is difficult to evaluate because evaluations are based on subjective judgments and the results obtained are difficult to relate verbally. Further, what is deemed acceptable in one area is completely unacceptable in another.

The mercaptans used in this investigation were all malodorous materials. However, the oligomers obtained exhibited little odor of the type generally attributed to sulfur

#### Table 4—Effect of the Hydroxy-Functional Mercaptans Used to Prepare Oligomers on the Flexibility of Resulting Coatings

			Flexibility, Conical Mandrel Bend, length of crack in mm Hydroxy-Functional Mercaptan			
Cure Temperature, °C (°F)	Wt. Ratio of	HEMA, Mole % of Monomers				
	Crosslinking Resin		ME	MP	HEMP	
140 (284)	4:1	16.9	15	3	3	
140 (284)	3:1	16.9	30	10	10	
140 (284)	2.33:1	16.9	45	38	4	
140 (284)	5.6:1	13.8	11	0	0	
140 (284)	4:1	13.8	17	12	0	
140 (284)	3:1	13.8	20	15	10	
120 (248)	4:1	21.1	18	2	0	
140 (284)	4:1	21.1	45	1	0	
120 (248)	1.5:1	21.1	19	10	9	

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compounds. The hydroxy-functional mercaptans and MMP yielded oligomers with less odor than did the alkyl mercaptans. The oligomers with the least odor were those resulting from HEMP.

#### CONCLUSIONS

Acrylic oligomers for high solids coatings were prepared by free radical solution polymerization of methyl methacrylate, butyl acrylate, and 2-hydroxyethyl methacrylate using hydroxy-functional and nonhydroxy-functional mercaptan chain transfer agents. Experimentation was designed to compare the relative effectiveness of these two types of mercaptan chain transfer agents as molecular weight modifiers. Also, the physical properties of coatings prepared from oligomers modified by the two types of mercaptans were compared.

The hydroxy-functional and nonhydroxy-functional mercaptans were equally effective as molecular weight modifiers yielding oligomers with a number average molecular weight in the range of 2000 and a polydispersity in the range of 1.5. Evaluation of thermoset coatings prepared from the oligomers and a melamine resin indicated that coatings utilizing hydroxy-functional mercaptans had better hardness and solvent resistance than coatings utilizing nonhydroxy-functional mercaptans. A trend in the coatings utilizing the hydroxy-functional mercaptans suggests that increasing the distance between the hydroxyl and mercapto groups in the mercaptan improves coating flexibility.

The odor of the oligomers prepared using the mercaptan chain transfer agents was less than might be anticipated. Oligomers from polymerizations employing the hydroxy-functional mercaptans were less odorous than those employing 1-mercaptodecane.

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# Effect of Solvent and Solvent Concentration On the Internal Stress Of Epoxide Resin Coatings

Masaki Shimbo, Mitsukazu Ochi, and Katsumasa Arai Kansai University\*

The magnitude and the mechanism for occurrence of internal stress were investigated for the solventbased bisphenol-A type epoxide resin coatings cured with triethylenetetramine. For these coating systems, internal stresses were not observed when the system was in the rubbery region. Rather, they were generated during cooling from the glass transition temperature of each system to room temperature. The magnitude of the internal stress depends on the concentration of the residual solvents, regardless of solvent type. Furthermore, the measured internal stress was in good agreement with the calculated value based on the strain and the modulus of coatings.

#### INTRODUCTION

Polymeric coatings are widely utilized for protection and decoration of various substrates. However, the coatings usually shrink during solvent evaporation or as the curing reaction proceeds. This shrinking results in internal stresses being generated within the coatings. The shrinkage and internal stress cause cracking, loss of adhesion, and other defects in the coatings. For epoxide resin coatings, many investigations<sup>1-6</sup> have been carried out to prevent the generation of these defects. However, in most of these investigations, the coatings have been cured at room temperature, so that the curing reaction has not reached an equilibrium state. Under such conditions, the values of internal stress were different from investigation to investigation. As a result, the mechanism for occurrence of internal stress in the coatings was not readily evident. The effect of solvents on the internal stress in the coatings has also been studied. However, the action of solvents is not exactly defined, because of a lack of the

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comparison of the internal stress in the solvent-based systems with that of the solvent-free systems.

We have studied the mechanism for generation of internal stress in the solvent-free, cured bulk systems and coating systems. The volume contraction and internal stress of bulk systems was measured by photoelastic<sup>8</sup> and strain gauge<sup>7, 9-16</sup> techniques for epoxide resin cured with aliphatic amines, <sup>6-8</sup> acids, <sup>11, 12</sup> aromatic amines, <sup>13, 14</sup> and alicyclic anhydride.<sup>9</sup> The effect of addition of the inorganic fillers, <sup>13, 14</sup> liquid rubbers, <sup>10</sup> and a spiro-ortho ester type resin<sup>15</sup> on internal stress generation was also investigated. For coating systems, the linear shrinkage and internal stress of epoxide resin coatings cured with aliphatic amines,<sup>7, 18</sup> acids,<sup>17</sup> reactive diluent,<sup>18</sup> and 2-methoxyethanol<sup>19</sup> were studied.

In the present paper, the mechanism for occurrence of internal stress is studied for epoxide resin coating systems that contain various amounts of methyl ethyl ketone (MEK), toluene, or 2-methoxyethanol and for the solvent-free coatings. Moreover, procedures for reducing the internal stress in the coatings are discussed.

#### EXPERIMENTAL

#### Materials

The epoxide resin used was liquid bisphenol-A type epoxide resin (Epikote 828, epoxide eq.:190 $\pm$ 5,  $\overline{Mn}$ :380).





Figure 1—Dynamic mechanical properties of epoxide resin coatings. Epoxide resin: Bisphenol A DGE (n = 0.1, Epikote 828). Curing agent: triethylenetetramine (TTA). Curing conditions: 80°C 4hr/130°C 4hr

Triethylenetetramine,  $H_2N$ -(CH<sub>2</sub>-CH<sub>2</sub>-NH)<sub>3</sub>-H, was used as curing agent. MEK, toluene, and 2-methoxyethanol were used as solvents. These curing agents and solvents were E.P. grade materials and were used without further purification.

#### **Curing of Epoxide Resin**

Epoxide resin was compounded stoichiometrically with curing agents wherein one epoxide group corresponded to one active hydrogen of an amino group. The indicated amounts of solvent were added to the compound. The mixtures were stirred at 80°C for about five minutes and were then coated on the aluminum substrate and allowed to cure at 80°C for four hours and at 130°C for four hours in a forced-draft oven. It was confirmed for all cured systems that the conversion of epoxide groups could not be changed by further curing at 130°C. Therefore, it was concluded that the curing reaction had reached an equilibrium state.

#### Measurements

Specimen shape and the procedure for measuring the internal stress of coatings were described in the previous paper.<sup>7</sup> Aluminum foil (thickness: 200 $\mu$ m; width: 15mm; length: 110mm; Toyo Aluminum Co. Ltd.) was used as the substrate. The aluminum substrate was cleaned with trichloroethylene and then subjected to air-drying. A strain-gauge (KFC-10-C-1-11, Kyowa Electric Co. Ltd.) was bonded on one side of the substrate. Epoxide resin compounds were coated on the other side of the cleaned substrate with an applicator. The thickness of the coatings was adjusted to be 200 ± 15  $\mu$ m.

As the epoxide resins are cured, the aluminum substrate is curved by the contraction or shrinkage of the system. The internal strain was measured and reduced to the internal stress as described in the previous paper,<sup>7</sup> by using the equations.<sup>25, 26</sup>

$$\sigma_{obsd.} = \frac{Eh_1^3}{12bh_2} \cdot \frac{2}{\rho(h_1 + h_2)} \left[ 1 + \frac{1}{3} \left( \frac{h_1}{h_1 + h_2} \right)^2 \right]$$
$$\rho = \frac{(1 + \epsilon)h_1}{2\epsilon}$$

where  $\sigma_{obsd.}$  is the internal stress and E,  $\rho$ ,  $h_1$ , b and  $\epsilon$  are Young's modulus, radius of curvature, thickness, width, and strain of the aluminum substrate, respectively. Thickness of the coating is given by the quantity  $h_2$ .

As other aspects of the study were carried out according to the procedures described in the previous paper,<sup>7</sup> details of the techniques are abridged. The gel content was determined by the procedure of Nielsen.<sup>27</sup> The epoxide and primary amino group concentrations were measured by the Bell method.<sup>28</sup> The tertiary amino group was determined according to the method of Luňák.<sup>29</sup> Dynamic mechanical properties were measured with a torsion pen-

	Initial Solvent Concentration phr (w/w%)	Residual Solvent Concentration phr (w/w%)	Sample Thickness (mm)	Gel Fraction (%)	Total	Conversion of Epoxide Group (%)		
Solvent						Reacted with Primary Amine	Reacted with Secondary Amine	Formation of Ether and Others
Nil (Solvent-free)	0(0)	0(0)	0.25	100	94	31	28	35
Methyl ethyl ketone	15(11.8)	4.5(3.5)	0.23	97	94	33	29	32
Toluene	15(11.7)	6.2(4.9)	0.23	100	93	33	28	32
2-Methoxyethanol	15(11.8)	6.8(5.3)	0.20	100	97	33	28	36
	10(8.3)	5.0(4.1)	0.23	99	95	33	32	30
	5(4.4)	1.8(1.5)	0.22	98	95	32	34	29

Table 1—Chemical Composition of Epoxide Resin Coatings

Epoxide resin: Bisphenol A DGE (n = 0.1, Epikote 828); Curing agent: Triethylenetetramine (TTA); Curing condition: 80°C 4hr / 130°C 4hr

dulum according to ASTM D 2236-64T. Linear expansion coefficients were calculated from the slope of sample length versus temperature.<sup>7</sup>

#### **RESULTS AND DISCUSSION**

#### Chemical Composition Of Epoxide Resin Coatings

Chemical characteristics of the epoxide resin coatings, solvent concentration, and cured-system characteristics are given in *Table* 1. As described above, the curing reaction for these coatings had reached the equilibrium state. Concentrations of residual solvents in this state are 4.5 phr(3.5 wt%), 6.8 phr(5.3 wt%), and 6.8 phr(5.3 wt%) for the coatings compounded with MEK, toluene, and 2-methoxyethanol, respectively, when the initial concentration of the solvents is 15 phr(ca. 11.7 wt%). The gel contents to be 100% and 97-100% for the solvent-free system and three solvent-added systems, respectively. Consequently, it is assumed that all cured coatings formed nearly complete networks.

Conversion of epoxide groups for all coatings is 93-97%. Furthermore, 31-33% and 28-34% of the epoxide groups reacted with the primary and secondary amino groups, respectively. Estimation of network structure from the conversion of the functional groups has been discussed elsewhere.<sup>10, 12, 17, 21, 24</sup> According to the discussion, we consider that 64-71% and 29-36% of the crosslinking points in these networks are composed of the amine and ether linkages, respectively.<sup>10, 22, 23</sup>

#### Dynamic Mechanical Properties Of the Epoxide Resin Coatings

Dynamic mechanical properties of the epoxide resin coatings are shown in *Figure* 1. Since the measurements were carried out in the range 0.3-3.0 Hz, the temperature where the damping had a maximum value was regarded as the glass transition temperature  $(T_g)$ . The  $T_g$  of each system is shown in this Figure, as  $T_{gn-15}$ ,  $T_{gt-15}$ ,  $T_{g2n-15}$ ,



Figure 2—Residual solvent concentration vs curing time and  $T_{\alpha}$  of epoxide resin coatings. Legend same as *Figure* 1

and  $T_{g_{2m-5}}$ . The suffixes indicate the solvent type and its initial content. For example,  $T_{g_{m-15}}$  refers to the  $T_g$  of the system that contained 15phr of MEK, etc.  $T_{g0}$  refers to the  $T_g$  of solvent-free system. As would be expected,  $T_g$  and damping maximum of the epoxide resin coatings are decreased with the damping peaks broadening when the systems contained solvent. These results demonstrate that there is residual solvent in the coatings after curing. The change of the concentration of residual solvents

	Residual Solvent Concentration	Glass Transition Temperature (°C)	Linear Shrinkage in Glassy Region (%)	Internal Stress σ (obsd.) (MPa)	σ <sup>a</sup> calcd. (MPa)	σ calcd.	
Solvent	phr(w/w%)					Internal Stress	
Nil (Solvent-free)	0(0)	130	0.65	6.72	8.12	1.21	
Methyl ethyl ketone	4.5(3.5)	112	0.49	5.15	6.13	1.19	
Toluene	5.5(4.3)	105	0.46	4.90	5.75	1.17	
2-Methoxyethanol	6.8(5.3)	104	0.45	4.64	5.58	1.20	
	5.0(4.1)	109	0.51	5.00	6.01	1.20	
	1.8(1.5)	121	0.57	5.92	6.96	1.19	

#### Table 2—Calculation of Internal Stress in Epoxide Resin Coatings from Thermal Expansion

<sup>a</sup> Calcd. from  $\alpha_{calcd} = \int_{-\infty}^{-\infty} \frac{Tg}{2.66G(\alpha_r - \alpha_a)dt}$ 

 $\alpha_r$ : Coefficient of thermal expansion of epoxide resin coatings

 $\alpha_a$ : Coefficient of thermal expansion of aluminum plate.



Figure 3—Internal stress of epoxide resin coatings. Legend same as Figure 1

during curing is shown in *Figure* 2(A). It is apparent that significant amounts of solvent remain in the films even after post-curing. Specifically, about 30%, 42%, and 34-50% of the initial MEK, toluene, and 2-methoxyethanol remain in the cured coatings, respectively (*Table* 1).

The effect of residual solvent on the  $T_g$  of the cured systems is shown in *Figure* 2(B). Over this low solvent concentration range, an approximately linear relationship existed between the  $T_g$  of coatings and the concentration of residual solvent, regardless of the type and initial concentration of the solvents. The results indicate that 5.3% residual solvent decreases  $T_g$  at about 26°C (*Table* 2).

#### Internal Stress In the Epoxide Resin Coatings

The changes in the magnitude of the internal stress in the coatings are shown in *Figure* 3(A). Internal stresses in all coatings were almost absent during cooling from the curing temperature (130°C) to the  $T_g$  of each coating (rubbery region), and they increased linearly with decreasing ambient temperature in the lower temperature region (glassy region).

Since the deflection points of the internal stress-temperature curves correspond to the  $T_g$  of each system, these curves were rewritten by regarding  $T_g$  as an original point, as shown in *Figure* 3(B). This Figure shows that the relationship between the internal stress in the coatings and the temperature difference from  $T_g$  can be expressed by a straight line, even if the variety and the amounts of the solvents are different in each system. We conclude from this result that internal stresses in these epoxide



Figure 4—Internal stress vs temperature difference from T<sub>g</sub> and residual solvent concentration of epoxide resin coatings. Legend same as *Figure* 1

resin coatings are negligible in the rubbery  $(>T_g)$  region and increase at a constant ratio with decreasing ambient temperature in the glassy  $(<T_g)$  region.

The internal stress at room temperature was plotted against the temperature difference between  $T_g$  and room temperature ( $T_g$ -t) as shown in *Figure* 4(A). An essentially linear relationship existed between the internal stress and  $T_g$ -t. This result shows that the internal stress at room temperature for each coating depends on the temperature difference between  $T_g$  and room temperature, regardless of the variety and the initial solvent content.

A plot of internal stress vs residual solvent concentration is given in *Figure* 4(B). In the plot, a nearly linear relationship also existed between the internal stress and the residual solvent concentration. This is reasonable, because the  $T_g$  of each system depends on the concentration of residual solvents [*Figure* 2(B)]. Therefore, it is concluded that the level of internal stress in these coatings depends only on the residual solvent concentration, even though different solvents and different initial solvent contents are involved.

#### Shrinkage in the Epoxide Resin Coatings

It is understood that the internal stresses in the coatings are influenced by the shrinkage. Thus, linear shrinkages of the epoxide resin coatings during cooling from the curing temperature  $(130^{\circ}C)$  are shown in *Figure* 5(A). The linear shrinkages in the temperature range from curing temperature to the T<sub>g</sub> of each system are expressed by a straight line even though different solvents and initial solvent contents were used. In the temperature range

INTERNAL STRESS OF EPOXIDE RESIN COATINGS

below  $T_g$ , the shrinkage had a different linear relationship for each system.

To explain the behavior of shrinkage in these systems, the plots of the linear shrinkage in the glassy ( $\langle T_g \rangle$ ) region vs temperature are shown in *Figure* 5(B). The shrinkage in the glassy region increased linearly with decreasing temperature for all cured systems. The shrinkage-temperature plots, rewritten by considering the  $T_g$  of each system as an original point, are shown in *Figure* 5(C). In this Figure, the relationship between the shrinkage in the glassy region and temperature was expressed by a straight line, regardless of the type of solvent and initial solvent content.

The linear thermal expansion coefficient,  $\alpha_r$ , of these systems is shown in *Figure* 6(A) and, of course, are reminiscent of diatometric measurements conducted through the glass-rubber transition range with various materials. The value of  $\alpha_r$  changed remarkably in the Tg region, while it remained essentially unchanged in the glassy region. These curves were replotted by considering the Tg of each system as an original point as shown in *Figure* 6(B). In the glassy region, the value of  $\alpha_r$  is little affected by the variety and initial contents of the solvents. This is the reason why the relationship between the linear shrinkage in the glassy region and temperature difference from Tg was expressed by a straight line in *Figure* 5(C).

Plots of the magnitude of the shrinkage in the glassy region vs temperature difference between the  $T_g$  of each system and room temperature,  $T_g$ -t, are shown in *Figure* 



Figure 5—Linear shrinkage of epoxide resin coatings. Legend same as Figure 1



Figure 6—Thermal expansion coefficient of epoxide resin coatings. Legend same as Figure 1

7(A). A linear relationship exists between the shrinkage and  $T_g$ -t. This shows that the shrinkage in the glassy region depends only on the temperature difference between the  $T_g$  of each system and room temperature, regardless of the variety and initial contents of the added solvents.



Figure 7—Linear shrinkage in glassy region vs temperature difference from T<sub>g</sub> and residual solvent concentration of epoxide resin coatings. Legend same as *Figure* 1



Figure 8—Internal stress vs linear shrinkage in glassy region of epoxide resin coatings. Legend same as Figure 1

The relationship of the linear shrinkage in the glassy region to the concentration of the residual solvents is plotted in *Figure* 7(B). A linear relationship also existed between the linear shrinkage in the glassy region and the concentration of the residual solvents. In the concentration arge of the residual solvent studied, the thermal expansion coefficient remained essentially constant in the glassy region (*Figure* 6), while the Tg of each system was governed only with the concentration of the residual solvent in the glassy region can be attributed to the residual solvent sudied, even if the type and the initial contents of the solvents are changed.

#### Mechanism for the Occurrence Of Internal Stress And Estimation of the Internal Stress

In the epoxide resin coatings that contained different solvents and initial solvent contents, both the internal stress and shrinkage in the glassy region depended on the temperature difference from  $T_g$  of each system, as shown in *Figure* 4(A) and 7(A), respectively. Thus, the relationship of internal stress to shrinkage in the glassy region is plotted in *Figure* 8. A linear relationship exists between the internal stress and shrinkage in the glassy region. From these results, we conclude that the internal stress in the coatings is governed by the shrinkage in the glassy region.

Estimation of the internal stress induced in the epoxide resin coatings is shown in *Table 2*. The internal stress is directly proportional to the linear shrinkage in the glassy region (*Figure 8*). Thus, if  $\epsilon$  is the strain caused in the coatings by their contraction and E the tensile modulus of the coatings, it should be possible to calculate the internal stress,  $\sigma$ , as the product of  $\epsilon$  and E. The magnitude of  $\epsilon$ during cooling from T<sub>g</sub> to room temperature (30°C) is calculated based on the linear thermal expansion coefficient of the coatings and the substrate according to the following equation:  $\epsilon = (\alpha_r \cdot \alpha_a)x$  (T<sub>g</sub>-30), where  $\alpha_r$  and  $\alpha_a$  are the linear thermal expansion coefficient of the solutings and the aluminum substrate, respectively. The shear modulus, G, rather than the tensile modulus, E, of the coatings was measured in this work (*Figure 1*). The value of E of the coatings could be calculated from G by using an assumed value of 0.33 for Poisson's ratio,  $\nu$ , and the following equation:  $E = 2G(1 + \nu) = 2.66G$ . In addition, the value of E changes with temperature. Thus, the internal stress,  $\sigma_{calcd.}$ , is calculated from the following equation:

$$r_{calcd.} = \int \frac{Tg}{30} 2.66G(\alpha_r - \alpha_a) dt$$

C

The values of  $\sigma_{calcd}$ , estimated from the above equation are shown in *Table* 2. The ratio of the calculated internal stress with experimental value is  $1.19 \pm 0.01$  for all the coatings. Accordingly, it is clear that the internal stress in the coatings is due to the shrinkage in the glassy region.

#### Method for Reducing The Internal Stress

It is clear from the results of this study that the internal stress generated in these epoxide resin coatings is proportional to (a) the shrinkage in the glassy region (*Figure* 8), (b) temperature difference from the  $T_g$  of each coating to room temperature [*Figure* 4(A)], and (c) the concentration of the residual solvents in the coatings [*Figure* 4(B)]. In addition, it is also clear that (d) the  $T_g$  of each coating depends on the concentration of the residual solvent, and (e) the linear shrinkage in the glassy region depends on the temperature difference from  $T_g$  to room temperature [*Figure* 7(A)] and the residual solvent concentration [*Figure* 7(B)].

It is concluded from these results that (a) the decrease in  $T_g$ , (b) the reduction of the linear shrinkage in the glassy region, and (c) the increase of the residual solvent concentration are effective as the methods for reducing the internal stress in the coatings.

T<sub>g</sub> OF THE COATINGS: The T<sub>g</sub> of the coatings cured with aliphatic amines<sup>7, 10, 18</sup> and acids <sup>11, 12, 17</sup> is lower than that of the coatings cured with aromatic amines, <sup>7, 16, 17</sup> alicyclic acids, <sup>15</sup> and tertiary amines.<sup>7, 16, 17</sup> If the aliphatic curing agents include the longer methylene sequence, the T<sub>g</sub> of the coatings shifts to the lower temperature. In addition, it is possible to decrease the T<sub>g</sub> of the systems by changing the network structure through the addition of accelerator,<sup>7, 10, 11, 12</sup> and by the addition of reactive diluents.<sup>13</sup>

SHRINKAGE OF THE COATINGS: A mineral filler having a small expansion coefficient can be used to reduce the shrinkage and internal stress. Addition of the mineral filler surely decreases the total shrinkage of the cured resins.<sup>6, 13, 14</sup> However, if the content of the filler is over some critical value, the internal stress increases dramatically because of the increase of the modulus of the system.<sup>13, 14</sup> On the other hand, addition of the liquid rubber decreases the modulus of the cured epoxide resin and thus reduces the internal stress of the system.<sup>16</sup>

Spiro-ortho esters either expand or contract very little during the polymerization process, and the mechanism for reducing the internal stress by the addition of the spiro-ortho esters has been published.<sup>15</sup> In addition, we will report the reduction of shrinkage and internal stress by changing the chemical structure of epoxide resins and the decrease of the internal stress by the addition of plasticizers in another paper.

EFFECT OF RESIDUAL SOLVENTS: Increase of the residual solvent concentration decreases the internal stress [*Figure* 4(B)], the linear shrinkage [*Figure* 7(B)], and the T<sub>g</sub> [*Figure* 2(B)] of cured epoxide resins. However, it is presumed that this method causes some troubles, because the internal stress and shrinkage increase with the evaporation of solvent even after curing. In fact, it was reported for polyester coatings that the internal stress and shrinkage increase with evaporation of residual solvent during thermal aging after curing.<sup>30</sup>

#### CONCLUSIONS

Bisphenol-A type epoxide resin coatings containing various amounts of MEK, toluene, and 2-methoxyethanol were cured with triethylenetetramine. The magnitude and the mechanism for occurrence of internal stress in the coatings were investigated in detail. The following conclusions were obtained.

(1) The internal stress in these coatings is almost absent in the rubbery region and is mainly induced by cooling shrinkage that occurs when cooling in the glassy region. The internal stress depends on the strain and the modulus of coatings. Accordingly, the internal stress generated in the rubbery region is negligible, because the modulus of coatings is very low in this region. On the other hand, the modulus in the glassy region is about two decades higher than that in the rubbery region and the motion of the network segments is restricted in this region. Thus, in the glassy region where relaxation times are very long, the shrinkage is not dissipated and high internal stress results.

(2) In the solvent-added epoxide resin systems, considerable amounts of solvent were retained in the coatings, even after the curing reaction reached an equilibrium state. The concentration of the residual solvents governed the internal stress, the shrinkage in the glassy region, and the glass transition temperature of the coatings, regardless of the variety and the initial contents of the solvents.

(3) For all coating systems, the experimental values of the internal stress were in good agreement with the calculated values based on the strain and modulus of the coatings.

(4) Methods for reducing the internal stress in the coatings were discussed based on the general rule found in this work.

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# The Evolution of Reduced Emissions Coatings

J. Larry Jameson Inmont Corporation\*

Making automotive coatings that are high in quality, strong in performance, appealing to the eye and, importantly, in conformance with increasingly stringent air quality regulations are the primary challenges facing the automotive coatings industry today.

Although many factors have influenced U.S. automotive coatings technology in the last two decades, only a few have significantly impacted on the overall course of this technology.

They include: customer demand for performance upgrading; governmental air quality regulations; a competitively driven need for aesthetics improvements; economic considerations; and, perhaps, energy relationships.

While these factors, or combinations of them, have shifted in importance over time, the single most influential force to change coatings technology in the U.S. has been the regulation of atmospheric emissions.

Active research and development aimed at controlling organic emissions from automotive and other industrial coatings began in the U.S. about 20 years ago. California, a state long concerned with pollution control, started the technological ball rolling with passage of local and state regulations such as Los Angeles, California Pollution Control District Rule 66.

Early regulations like these initially restricted the chemical composition of solvent systems in coatings. Now these state standards have evolved into actual quantitative restrictions that paved the way for broad federal legislation on—and the evolution of—coatings with reduced organic solvents.

These early California regulations of the mid-1960's sparked the development of the first water-borne enamels that were used worldwide in the auto industry. Commercialized in 1975, these low emission waterborne materials are still being used at automotive assembly plants at Van Nuys, Calif. and Oklahoma City, Okla. This technology established a standard for all reduced emissions products under development for the last 10 years.

The water-borne enamel technology with Volatile Organic Components or VOC emissions of 2.8 pounds per gallon of coating as applied has been established as the level of environmental acceptability in low emissions coatings technology. This is the standard for competing topcoat alternatives.

The standards don't stop with topcoats. Other VOC performance levels have been set for electrocoat systems, spray applied body primers, and repair coatings material. U.S. auto manufacturers must meet this federal standard of emissions reduction by 1987.

Before 1987, intermediate standards that set specific VOC emissions maximums have been established by local and state agencies for individual assembly plants. The impact of compliance with these intermediate emissions standards—to say nothing of the target objectives—has been broad and significant. It has been a factor in all automotive coatings materials development and in processing application methods engineering. Following is a summary of technological developments aimed at satisfying Clean Air legislation.

#### **Electrodeposition Coatings**

Although electrodeposition coatings represent the most effective technology available to control emissions, no single element of the total automotive coatings system was exempted from playing a role in reducing organic emissions.

Because electrodeposition represented a technical alternative that offered the benefits of automation, efficient application, and improved corrosion resistance, it replaced—and thereby reduced emissions levels from—solvent-borne dip and spray primer systems throughout the industry.

From a quality vs environmental perspective, it was a "win-win" solution.

In the late '70's and into the early '80's, the VOC level of commercial electrodeposition primer systems remained at about 2.2 pounds per gallon. Still lower emissions levels of about 1.8 pounds per gallon were achieved through the development of lower emission materials and control of operating bath parameters.

Further substantial reductions to the current operating level of 1.2 pounds of VOC were the result of research and development into lower solvent content polymer processing and wet film plasticization through polymer changes rather than solvent content.

Advanced development now in place is expected to yield even further VOC reductions . . . down to levels of 0.5 pound or less!

Presented at the 12th International Conference on the Finishing of Automobiles, Cannes, France, on June 7, 1985.

<sup>\*1255</sup> Broad Street, Clifton, NJ.

It is important to note that, despite these substantial reductions in VOC and accompanying improvements in environmental quality, performance properties in terms of physical durability of film aesthetics have not been compromised.

#### Sprayable Body Primer-Surfacers

Emissions reduction of sprayable body primer surfacers took place along two paths: high solids or water-borne technologies.

Technical evolution resulted in waterborne primers, which offered potentially greater emissions reductions, being commercialized primarily in plants using water-borne enamels.

High solids solvent-borne primers, which were easier to utilize in plants using low-solids, solvent-based primers, were adopted extensively in most passenger car and truck plants.

The use of high solids, low VOC primer surfacers applied by high voltage electrostatic centrifugal atomization, has produced much lower emission levels from spray priming operations.

This combination offers the advantages of improved product utilization, better cost/benefit performance *and* lower emissions.

#### **Topcoat Systems**

To examine the evolution of topcoat technology, the experience of General Motors is instructive for two reasons: GM opreduction efforts. This impact has been so profound that it has produced a revolution in topcoat technology in the U.S. which has affected all U.S. automakers.

A number of coatings systems were developed to respond to the emissions standards. These included water-borne enamels, high solids two component urethanes and powder coatings.

GM studied these options and met the earliest standards by adopting the waterborne enamel system which was first developed and introduced by Inmont Corporation in 1975. Some ten years later these same low emissions enamels are still used by GM today at two plants.

As individual states have enacted emissions legislation, and as the issuance of Federal standards approaches, extensive research and development programs are exploring alternatives to existing coatings technology. The charge: to meet current, and projected, emissions standards and further improve the aesthetic quality of the topcoat finish. It's a real challenge.

The auto industry's need for relatively rapid change in order to satisfy impending regulatory issues has given coatings manufacturers an opportunity for significant technological innovation. Advanced coatings technology has been the result.

The original technological plan developed by Inmont placed emphasis on both high solids—low solvent enamels, and high solids base coat/clear coat systems. However, the program quickly evolved into a concentrated effort to optimize high solids base coat/clear coat technology.

Preliminary systems that offered applied base coat/clear coat volume solids levels of

#### "...the single most influential force to change coatings technology in the U.S. has been the regulation of atmospheric emissions."

erates a large percentage of all assembly plants in the U.S. and when the new regulations were established, GM was using a lacquer system.

From an emissions perspective, and especially for GM, right after the earliest Clean Air regulations were introduced, topcoat was the major contributor.

More than any other element of the total coatings systems, the topcoat has been most significantly influenced by emission 32% and 44% respectively, and a composite VOC level of 4.4 pounds per gallon, have now evolved into even lower emissions materials of 40% and 54% base coat/ clear coat applied volume solids respectively, and composite VOC levels of 3.6 pounds per gallon.

Driven by emission reduction objectives, this planned replacement of thermoplastic solution and dispersion acrylic lacquer technology with thermosetting high solids base coat/clear coat enamels is perhaps the most significant change in coatings technology in recent history. This change was significant. It has produced a major redirection of materials technology, the introduction of advanced application methods, *and* the expectation of real improvement in physical performance and appearance of automotive coatings.

#### Some Solutions

Thus the need was established and the solution defined, all that remained was the matter of implementing this historic and significant program. These, then, were the problems. Following are some of the coatings industry's reactions to them.

The pressure for rapid technology change often stimulates creativity. Evolutionary change, on the other hand, is generally orderly and permits modifications well supported with extensive data. Periodic revolutionary change, however, can force real, but creative, solutions for urgent, complex problems.

The organic emissions problem in the U.S. is a classic example. The time frame was short and the problem at hand was significant and technically complex.

In terms of emissions levels, automotive OEM coatings materials and systems developed over the past fifteen to twenty years were now under critical analysis.

The days of minor modifications and adjustments had come and gone. There were no easy answers.

Evolutionary advances of most coatings systems which had been prompted by early requirements like Rule 66 were already in place. They were approaching their technological limits.

As indicated, electrodeposition coatings technology had reduced emissions levels to 1.2 pounds VOC per gallon applied. This compared to earlier levels of 2.2 and 1.8 pounds. Low solids sprayable body primer surfacers had advanced almost annually to incrementally higher applied solids and lower VOC levels.

Most recent advancements produced substantial changes in the resin chemistry of these primers, allowing introduction, between 1979 and 1982, of coatings applied at volume solids levels of 55 to 60% with VOC levels as low as 3.0 pounds per gallon.

This represented a substantial improvement over earlier conventional lower solids primers applied at 35 to 40% volume solids and VOC levels as high as 5.0 pounds per gallon.

Incremental advancements in applied volume solids of thermoset acrylic enamel topcoats had continued almost since their introduction in the early 1960's. For thermoplastic acrylic lacquer systems used on all General Motors passenger car lines, the evolutionary progress was nothing short of dramatic.

Acrylic lacquer topcoat based on solution acrylic resin chemistry was first commercialized in the early 1960's as a major durability upgrade replacement for nitrocellulose lacquers.

Solution lacquers with applied volume solids levels of 10 to 12% and VOC levels averaging 6.8 pounds per gallon were used on GM passenger cars in the United States until 1979.

The advanced development of dispersion chemistry based polymers significantly extended prior technological limits in terms of applied volume solids. Through the ensuing period, following introduction of the 17% volume solids dispersion lacquers, systems offering 22% and 27%, and VOC levels of 5.5 and 5.1 pounds per gallon, respectively, have evolved.

Having nearly reached the technological limits of existing systems to reduce organic emissions, "fast track" advanced development programs were initiated to seek even better alternatives. Initially, "the solution" appeared to be high solids thermoset acrylic enamel topcoat systems.

These enamels, offering average applied volume solids levels of 46 to 50 percent and VOC levels of 3.6 to 3.9, were introduced into all General Motors truck plants, as well as both AMC plants and some Ford plants.

Advancements in polymer chemistry helped create these relatively low, but narrowly controlled molecular weight resins, and permitted these high solids enamel products to be developed.

The primary areas of concern, however, in the use of resinous components approaching oligomeric nature were with applied system rheology and physical/mechanical performance.

Specifically, these concerns were:

- Aluminum pigment orientation in metallic colors—and general resultant color and gloss limitations, primarily in light metallic color ranges, and,
- (2) Film control characteristics including sag resistance, excess flow from character lines, and edge pull-away.

The laboratory was where some of these critical application performance problems were solved. It was there that a novel, very highly efficient dispersion chemistry-based rheology control polymer was developed. Using low levels of these high efficiency flow control agents made for a quantum leap forward.

Research efforts were rewarded with substantial improvements in: aluminum pigment orientation; cleanliness of color; color travel in light metallic color ranges; and applied film flow control properties.

Even these significant technological

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strides, while noteworthy, were to fall short of meeting appearance quality standards that were yet to evolve.

Physical performance and exposure durability of the low molecular weight solution polymer-based systems were primarily maintained through optimization of system reactivity and crosslink density. Additionally, high solids enamel formulations utilizing generally low molecular weight solution resins have become somewhat more highly dependent on proper selection and use of advanced developments in ultraviolet light absorbing chemicals.

The years 1981 and 1982 saw aggressive adoption of thermoset high solids enamels in early conversions of most commercial vehicle assembly lines as an evolutionary response to the stringent Clean Air legislation target dates.

During the same time that U.S. automakers and their OEM suppliers were grappling with the intense, governmentally-imposed regulatory pressures to reduce emissions, the marketplace produced equally extreme pressures. It demanded substantial improvements in the physical appearance characteristics of automobile finishes.

"Fit and finish" concerns from car buyers and carmakers suddenly assumed equal footing with environmental concerns emanating from the government. Among the driving forces behind this latter aesthetics quality issue were:

- (1) The substantial growth of import sales in the U.S.;
- (2) The resultant increase in global sales competition; and
- (3) The overall issue of comparative quality in all aspects of the automobile.

Clearly, the quality and selling appeal of the finish—the first thing a car customer sees—became a major concern.

For the U.S. automaker, this meant a growing sensitivity to customer satisfaction. Out of shadows emerged a far more discerning and quality-conscious customer. It became apparent that the aesthetic quality of the base coat/clear coat finish regularly provided on cars imported from Europe or Japan could only be equalled with a base coat/clear coat system.

Non-clear coated high solids enamels, particularly in light metallic color ranges, presented inherent weaknesses when compared with base/clear materials in both color capability and gloss. These problems were rooted in rheology control related factors that impacted on color cleanliness, metallic travel, and surface texture, as discussed earlier.

Collectively, these critical issues forced the acceleration of high solids base coat/ clear coat technology as a more appropriate answer to the problems of low emissions on the one hand, and higher quality on the other.

Again, it is interesting to examine GM's movement from lacquer to an enamel system.

This conversion involves the change of all General Motors passenger car assembly lines from a 27% volume solids—5.1 pounds VOC thermoplastic acrylic dispersion lacquer system, to high solids, base coat/clear coat thermosetting enamels averaging near 50% applied volume solids and VOC levels approaching 3.6.

This revolutionary conversion in technology was initiated with the introduction in 1981 of high solids base coat/clear coat enamel at the General Motors Bowling Green Assembly plant. It has proceeded to date through the conversions or start-ups of six other GM plants.

The technology has continued to rapidly advance and now includes systems offering the capability of further, substantial reduction in VOC yet with improved appearance, better durability, and additionally, elastomeric coating qualities.

The commercialization of high solids base coat/clear coat in the U.S. now includes some 18 paint lines; seven at GM, six at Chrysler, three at Ford, and two at American Motors. Conversion to high solids base coat/clear coat enamels in U.S. assembly plants has allowed compliance with current emission mandates and a measurable improvement in the quality of OEM finish appearance and durability.

#### Summary

The high solids base coat/clear coat technology has provided an innovative and practical solution to a complicated, multifaceted problem. However, as always for OEM coatings producers, considerable opportunity for further advancement remains.

A number of primary issues are currently being addressed in the critical area of topcoat systems. In a way, our situation reminds me of the remark made many years ago by an American labor leader who was asked by a reporter, "What does labor want?"

"More," said Samuel Gompers.

And more is what U.S. automobile manufacturers and the U.S. car buying public want...and it is what the coatings industry is going to give them, in terms of:

- Significant reductions in organic emissions levels
- Upgrading of color latitude and styling potential in metallic colors.
- Improvements in film smoothness and resultant gloss, distinctness of image, and film clarity
- · Better coating system durability and,
- Advancements in materials usage efficiency and general systems economics.

These critical issues continue to chal-

lenge our ability to help improve our environment by meeting Clean Air standards while, at the same time, offering car buyers the outstanding quality they rightly demand and deserve.

The time frame for meeting these pressing demands remains compressed.

The pressure for technological change to solve these immediate and near term problems offers unique opportunities for technically creative solutions.

The famous U.S. inventor, Thomas Edison, once observed that creativity involved a small amount of inspiration and a great deal of *perspiration*. Regardless of whether by inspiration, perspiration, or both, the automobile manufacturing and the OEM coatings industries welcome these challenges.

Together, as we have in the past, we'll meet them today...and tomorrow.

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The corrugated box standards are as follows:

- Bursting Test—175 pounds per square inch
- (2) Minimum combined weight facings—75 pounds per thousand square feet
- (3) Gross weight—maximum of 40 pounds.

The aluminum coated fibre two-inch tape (used inside of the box) is fibre reinforced; rubber type adhesive; with aluminum coating on top of the tape; and is similar to "Shurtape—Hickory N.C. 28603."

The white box two-inch sealing tape for outside sealing of the box must have tensile strength—22 pounds per square inch; adhesion—25 ounces per inch; thickness—2.6 mils; elongation—40%; and rubber type adhesive.

Outside of the box, the seams and bottom folding flaps should be thoroughly sealed with a two-inch tape similar to the sealing tapes. The inside seams and all horizontal and vertical corners should be sealed with a two-inch tape similar to the aluminum coated fibre tape.

Dimensions of the inside area of the box to be painted (A = area; l = length; w =width; h = height) are:

A = (lw) + 2 (lh) + 2 (wh)

 $A = (12^{7}/_{8} \times 8^{3}/_{4}) + 2 (12^{7}/_{8} \times 5^{1}/_{2})$ 

- $+ 2 (8^{3}/_{4} \times 5^{1}/_{2})$
- A = 112.70 + 141.68 + 96.25

A = 350.63 square inches.

The box taped should weigh 200 to 250 grams and when painted with 18-20 dry mils will be 375 to 450 grams. The paint used to coat the box (350.63 square inches) will be 175 to 200 grams. Allow taped "Box Tester" to age one week before painting.

The specifications of the paint III that was used for the demonstration of the "Benham-Gachette Ponding Water Box Tester" are:

(1) T.N.V. = 73%; (2) P.V.C. = 48%; (3) Lbs. per gallon = 13.47; (4) Volume solids = 60%.

We recommend an 18 to 20 mils (dry) paint film be applied to the "Box Tester" for the ponding water test. The wet film thickness should be 30 to 40 mils based on the volume solids of the paint to be tested.

To calculate wet and dry mil film thickness on the "Box Tester" we used paint III = 13.47 lbs. per gallon = 6115.38 grams per gallon. Brushing on five coats of paint over five days, we applied 181 grams of dried paint on the box. 181 divided by 6115.38 = 0.029598 gallons. 1 gallon = 231 cubic inches  $\times$  0.029598 = 6.837138 cubic inches. Volume = 350.63 square inches  $\times$  M. (Note: M = mil thickness.)

 $6.837138 = 350.63 \times M$ 

6.837138 ÷ 350.63 + M

0.01950 = M. Thus, dry film thickness is 19.5 mils and wet film thickness is 19.5  $\div 0.60 = 32.5$  wet mils.

Table 1—Average Water Loss							
Test	Date	Inches	Inch	Water	Days	Average	
	(1985)		LOSS	Ounces To Refill	Tests	Lost <sup>b</sup>	
A	2/6	210/16	-		_	_	
В	2/11	2%16	4/16	9	5	1.80	
С	2/15	25/16	5/16	12	5	2.40	
D	2/19	2%/16	4/16	9	5	1.80	
E	2/25	2%16	5/16	12	6	2.00	
F	3/4	25/16	9/16	16	7	2.28	

(a) The average daily depth loss was 0.30 inches.

(b) The average daily volume water loss was 2.07 ounces. Note: If no leaks show up in the wetting of the exterior of the box or on the kraft paper in the tray the "Box Tester" is sitting on, the loss will be evaporation or water loss into the swelling of the paint film. This should be determined each 30 days of testing. (See section on measuring water absorption into the paint film.)

Introduced at the Annual Meeting of the Southern Society of the Federation of Societies for Coatings Technology, March 27-29, 1985, in Atlanta, GA.



Figure 1

To measure water volume loss, use a light weight plastic six-inch ruler to measure the water depth. Fill box one half full



Figure 2—Close up of Benham-Gachette Ponding Water Box I and Box II. Paint I lost one inch of water in three hours. It lost all water overnight with the entire exterior of the box soggy and disintegrating

of water  $(2\frac{1}{2}-3'')$ . The example test on paint III used  $2\frac{5}{3}$ ". After each five day period, measure the depth of the water change and refill to the initial filled mark. The graph for paint III is as follows in *Table* 1.

For measuring any water absorption into the paint film, empty all water from the "Box Tester" after 30 days and allow it to dry overnight. Weigh the box, then check weight versus the freshly painted box (on paint III it was 388 grams). The box was 207 grams and the paint was 181 grams.

The formula used follows:

- Initial weight of box painted initial weight of box = initial weight of paint.
- (2) Weight of box painted (dried) initial weight of box = weight of paint immersed (after 30 day test).

- (3) Weight of paint on 30 day tested box - weight of paint initially = film water gain (fwg).
- (4) On paint III, the fwg was 0 grams or 0%.\*
- (5) Factor for paint exposed to water immersion:
  - (a) Area of box is 350.63;
  - (b) Area above water is 118.94;
  - (c) Area submerged is 231.69;
  - (d) Water gain + initial paint weight = % moisture pick up;
  - (e) Multiply (5d) by 1.513343 to get the correct % of moisture swelling after 30 days for paint 111.

#### Inexpensive Method For Determining Liquid Volume in a Tank

MARTIN BOARDMAN

Bel-Mar Paint Corp. 2790 West 3 Court Hialeah, Florida 33010

Fasten at least two pulley wheels to a wood block. Glue or fasten this wooden block (with pulleys) at the top of the tank between the tank opening or orifice, and the vertical side of the tank. The pulley wheels should extend slightly over the sides of each drop-down. This wood block can be fastened to the tank with a thick coating of alkyd resin and be allowed to dry.

A strong string should be used to ride over the pulley wheels, down into the tank and down along the outside of the tank. A small, empty, capped glass bottle should

<sup>\*</sup>The "Box Tester" lost I gram after 30 days. This is 0.2578% based on the "Box Tester" or 0.5525% based on the paint applied. This could be due to the loss of moisture in the cardboard box or volatile residual chemicals in the paint film.



Figure 3—A close up of Box III and Box IV. Paint IV did not lose water except for evaporation ( $\frac{4}{16}$  to  $\frac{6}{16}$ ") for 48 hours. Then it lost 1" in 72 hours and emptied in 96 hours. The box was soggy and started to disintegrate

be tied at its neck with one end of the string. Then place the bottle into the tank to ride up and down on the surface of the liquid in the tank. The outside tank string should also be tied to the neck of another glass bottle (or other desired weight) of a lesser weight than the inner tank bottle. This outside tank bottle acts to keep the string taut and to indicate the liquid volume inside the tank. When the outside bottle is at the bottom of the tank, the tank is full. When the outside bottle is at the top of the tank, the tank is empty.

#### **Quick Non-Volatile Determination**

MARTIN BOARDMAN

Bel-Mar Paint Corp. 2790 West 3 Court Hialeah, Florida 33010

Prepare the two samples of weighed dishes for non-volatile determination procedure in the customary manner. Place the dishes or samples to be run on a surface that will not burn or be affected by heat. The heating element should be a common heating bulb (250 watt) inserted into a goose neck desk type reading lamp. Place the surface of the bulb exactly three inches away and above the sample dishes. Switch on the lamp for approximately 15 minutes. The liquid matter in the sample dishes should be stirred (with preweighed stirrers) two or three times during the heating procedure to break the skimming that may form and make the liquid more uniform.

The test may be considered finished when samples appear to be "dry" and no longer free flowing. The heating time may be prolonged or abridged depending upon the sample quantity and volatile matter to be run off.

This quick procedure is within 1 or 2% accurate.

#### Quick Pigment Color Rub-Up Determination

#### MARTIN BOARDMAN

Bel-Mar Paint Corp. 2790 West 3 Court Hialeah, Florida 33010

Instead of using linseed oil or other usual slow drying liquids as the media in a pigment color rub-up, the use of vinyl toluene alkyd in VMP naphtha as the liquid media will give very rapid color determination because of the faster drying.

#### **Quick Cool for Lab Samples**

LOIS GOLDSMITH

Ocean Chemicals, Inc. P.O. Box 1667 Savannah, Georgia 31402

Place weighted lined quart can in large container of water. Place in freezer until frozen solid and ready to use.

When needed, remove weights and partially fill the quart can with hot water, spin the can until loose enough to remove should take a few seconds. When can is removed, add enough cold water for a bath for the sample that is to be cooled down. Use as long as needed—all day if necessary. Store back in freezer when not in use after replacing the quart can and weights as before. Lasts all day!

Supplies needed to build: 1 large stainless steel bowl; 1 lined quart can; Mill balls to weight can; and Water.

### Let's Can It! (or what can you do with an extra 25-50 bucks a day?)

STEPHEN J. LAKATOS

Akzo Coatings America, Inc. 6369 Old Peachtree Rd. Norcross, Georgia 30071

No, I'm not going to talk about a pickle factory, or preserving greens for the winter. However, I am going to present a way to save some dollars that are escaping through the cracks of the floor. How many times have you attempted to follow the monastrol red footsteps through the plant? How much was spilled and stepped into? One ounce of pigment, \$2.00? Four ounces of pigment, \$8.00? Have I got your interest yet? How about monastrol red, blue, and hansa yellow B.O.N.'s? All are in the \$8.00 to \$16.00 and better range and an ounce or two of these pigments spilled on the floor rapidly adds up to big bucks.

Work horse titanium dioxide, at 60-70 cents per pound, spread around a given work area at the rate of one or two pounds makes a considerable mess. Who said white is clean?

I have estimated that for every bag of black pigment poured into cowels premix 2-3 oz. is lost into the atmosphere. Aside from the actual loss of 10 cents a bag in pigment, hiding power and batch contamination, there is the expense of clean-up. In this case black is not beautiful.

Now, do I still have your interest? Good! All you have to do is go out and buy a couple of zinc coated garbage cans. Take your bags of pigment and deposit them into the can and carefully cut away the entire top of the bag (this eliminates opening and closing the bag, creating more dust). Then buy a scoop for each color, to avoid contamination. When the batch maker takes a scoop of the pigment to weigh, he holds the can lid under the scoop, swings over to the scale and carefully deposits the pigment in the staged batch. If pigment spills out of the scoop it either drops into the can or the lid—not on the floor!

What about the pigments that come in the cardboard drums? No sweat! Just get a five gallon can lid and use it the same way as the garbage can lid. It's not perfect, but it will save dollars, aggravation, and clean up time.

Some additional tips. If you are not doing this yet, put grinding resin in the bottom of the tank, add the thinner and wetting aids, and then carefully deposit the organic pigments in the thinner. The pigment is quickly wetted and when you turn on the mixer, powdering of the pigment is either eliminated or drastically reduced. OSHA will love you.

One last tip. In the drummed pigments, cut away that excess paper and throw it out, it only causes powdering both when closing and opening the inner bag.

Using these suggestions won't enable you to raise your stock dividends by a dollar a share but they might pay the membership fee at the local club with a few bucks left to invest in poker.

One final remark. No new or innovative idea will work anywhere without steady gentle prodding of management. Don't let up the pressure but don't raise your voice. Rather than brow beating them, be gentle with employees when initiating new and better habits.

### Color Code System To Optimize Lab Time

JAMES W. LEE

Akzo Coatings America, Inc. 6369 Old Peachtree Rd. Norcross, Georgia 30071

This is an extension of last year's system to minimize time on retrieving resins and pigments, but I have carried it one step further to retrieving additives and how to color code them so they can be more accessible without having to go through each and every container.

Here, paint all the containers white and then take a stencil gun with different color tapes, and stencil the code and the product name on them. For example,

Additives							(	20	C	L	0	R	(	Code
Stabilizers													1	Black
Chemicals	•	•	•	•	•		•	•	•	•	•			Blue

## SOUTHERN SOCIETY

Additives	COLOR CODE
Driers	Green
Flow Control Age	nt Yellow
Suspending Agent	Orange
Thickeners	Red
<b>Dispersing Agents</b>	Brown
Plasticizers	Gold
C	1

So at a glance you can locate the color code of the additive you want and only have to go through a few containers.

#### Handfilling Five Gallon Pails

#### STAN TRUELOVE

SCM Organic Chemicals Corp. P.O. Box 389A N Ave. Jacksonville, Florida 32201

PROBLEM: Hand fill five gallon pails of block filler in a more timely and safer fashion.

OLD SYSTEM: (1) Hand stamp production numbers, plant, and date codes on labels

for batch. (2) Glue labels to buckets. (3) Hook Finex up under desired tank and hand fill pails to desired weight. (4) Cut off paint, put a lid on and crimp. (5) Hand stack onto pallet and move pallets to Shipping.

OLD RESULTS: This system would take approximately 16 man hours to perform and would require the employee to lift approximately 35,000 pounds during this time.

RESULTS NEEDED: To find a way to use the existing Elgin filler to fill the hand fill pails.

PROBLEM: Because of the viscosity and the abrasive nature of block filler we weren't able to fill it with the existing Elgin because it would ruin the cylinder walls.

SOLUTION NEEDED: If we could use existing machinery we could eliminate hand stamping labels, putting on labels, and palletizing pails.

SOLUTION: We decided to pump the paint into the existing five gallon line Finex area to strain paint. Then pump this paint overhead to a ball valve over the existing five gallon filler. This allowed us to use all the existing machinery. We have used check weighing on this system and have cut our man power to six hours per block filler batch.

Open Forum is an experiment in communications designed to give readers the opportunity to share creative concepts in all aspects of coatings. Suggested topics include color, formulation and manufacture, testing, and selection of raw materials. These "tricks of the trade" need not be prepared formally such as research papers, but should, however, be thorough in their preparation and presentation. Submissions should be sent to "Open Forum" Editor, Journal of Coatings Technology, 1315 Walnut St., Philadelphia, PA 19107.

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## LIAISON

## A Brief Look at the Australian Surface Coatings Industry

[This thumbnail sketch of the Australian coatings industry was published as Newsletter #4 of the Oil & Colour Chemists' Association International in mid-1985.

The Editor/Coordinator of OCCA International is E.T. Backous, 73 Edgecliffe Blvd., Collary Plateau, N.S.W. Australia 2098. He is a member of OCCA Australia.

The article was prepared by Kevin Duffy, of the Australian Paint Manufacturers Federation.

We are grateful to Messrs. Backous and Duffy for permission to reprint excerpts from the original piece in the JCT.—Ed.]

#### Nature of the Industry

As is the case in most industrialized economies, the market for surface coatings in Australia is divided clearly into two major segments: (1) Architectural and Decorative Paints and (2) Industrial Paints and Coatings.

About half of the industry's production takes the form of the familiar architectural and decorative paints. Growth in architectural and decorative paints basically follows the growth in population and is only slightly affected by other economic influences. Over the last ten years whilst the production of solvent-thinned house paints has shown a gradual decline, from 42 million litres in 1974/75 to 30 ML in 1983/84, there has been a marked increase from 28 ML in 1974/75 to 54 ML in 1983/84.

The other major sector of the industry comprises paints and coatings for industrial application which are formulated to meet special conditions encountered in numerous industrial and special end-uses. Total output of industrial paints and coatings has been in the range of 60 to 65 ML during the past 10 years. Demand for industrial paints varies from year to year depending on the general state of the economy. Accordingly, the market for industrial paints was hit badly by the 1983 recession, dropping from 68 ML in the previous year to below 60 ML.

The total market for paint products in Australia is virtually static and forecast growth is less than 1% per annum over the longer term.

#### **Contributions to the Economy**

The paint manufacturing industry is one of basic economic importance because its products contribute greatly to the progress, growth, and prosperity of our modern life style. The industry makes a significant contribution to the Australian economy through its purchase of locally-produced materials. The majority of purchases of raw materials used by the industry are sourced locally. Overall, the industry is intake of raw materials, services, and supplies provide important markets for other Australian producers.

In 1982/83, the latest year for which Australian Bureau of Statistics are available, the turnover of the industry amounted to \$582.6 million. This figure represents about 1% of the turnover of the total Australian manufacturing sector.

Value added for 1982/83 amounted to \$219 million. With employment being 5,803 as at June 30, 1983, value added per employee for 1982/83 amounted to \$37,780. This figure compares favorably with a wide range of other Australian industries.

#### Structure of Industry

On the basis of most recent data available from the Australian Bureau of Statistics, i.e., 1982/83, the industry was made up of 125 establishments. This figure excludes single establishments employing less than four persons, which in 1982/83 amounted to 36 establishments employing a total of 76 persons.

The larger companies within the industry, some of which employ over 1,000 persons, produce an extensive range of surface coatings for application in just about all fields. The medium and smaller-sized manufacturers generally produce a more limited range of products, and many specialize in a particular sector of the market. For instance, some manufacturers concentrate on production of architectural and decorative paints, and some specialize even further by confining their distribution to a limited geographic area. Other manufacturers specialize in the production of particular types of industrial finishes, many of which are made to meet the specific requirements of their particular customers.

The majority of factories is located in Sydney and Melbourne, adjacent to Australia's principal centers of population and industry.

### Level of Technology And Rate of Innovation

New and improved developments in paint technology mostly come from the laboratories of raw material manufacturers, but, in general, much development work is still required on the part of the local paint manufacturer in order to meet his particular customer's needs.

Australia's contribution to world paint technology is minor in terms of the number of published patents. Very few Australian companies undertake basic research, relying instead on licensed know how or technology emanating from their overseas affiliated or associated companies.

However, some noteworthy Australian technologies in recent times have included:

"Spindrift" polymer beads used in emulsion paints to reduce cost at equivalent performance.

"Compolex" composite polymer latexes giving pre-determined properties to water-borne paints.

"Vapocure" a gaseous catalyst allowing rapid room temperature curing of polyurethane type industrial paints.

The industry is well served by professional bodies, in particular the industry's learned society, the Oil and Colour Chemists' Association, Australia.

#### **Recent Industry Rationalization**

In recent years, companies within the paint industry have responded well to market pressures and substantial restructuring has been undertaken by the industry in order to keep pace with changes within the Australian economy.

Mirroring developments in the manufacturing sector of the Australian economy, production, sales, employment, and capital expenditure within the paint industry deteriorated sharply during 1982 and into the first half of 1983.

Whilst some sectors of the manufactur-

ing industry have since shown improvement, there are marked divergencies in both performance and outlook between the various sectors of manufacturing industry. In turn, demand for paints and coatings by these various sectors has undergone marked change.

Over the last few years there has been a trend within the metal trades industry for a higher proportion of components and/or finished products to be produced off shore. Invariably, where such products require painting, they are painted overseas. This trend has led to a loss of market potential for Australian paint manufacturers. An outstanding example is the case of the automotive industry where today a higher proportion of vehicles and vehicle components is being imported. The result of this trend is demonstrated by the reduction in production of automotive primers and undercoats from a level of 5.2 million litres in 1979/80 to 4.3 million litres in 1983/84-representing a reduction over this period of some 16.5%.

Sectors of the manufacturing industry associated with large scale capital investment represented by the general engineering and heavy engineering plant and equipment sectors, are lagging in the recovery process. On the other hand, those segments of industry connected with the rural, housing consumer products and light industrial equipment, are showing improvement.

Significant restructuring activities have been undertaken by all major and a number of medium-sized paint manufacturers during the last few years. This activity has taken on a number of forms including the introduction of new technology, closure of some plants, and rationalization of product ranges. Since 1981 there have been 20 partial and/or total plant closures within the industry.

#### **Control of Hazardous Chemicals**

Bernard Rubens, who has been actively involved in developing these controls, has kindly prepared a summary of the present "state of affairs."

Although both industry, government and the general community have shown concern with the control of hazardous chemicals in Australia, control has been slow and haphazard due to the Australian system of governments with one central government and six separate governments in the member States.

The most significant move to control hazardous chemicals was a voluntary scheme to create a list of hazardous chemicals in Australia; authorities were not keen to accept the TOSCA (USA) or ECOIN (EEC) lists but to develop a list of materials actually in use in Australia. The voluntary scheme has been replaced by a mandatory system, the Australian Inventory of Chemical Substances (ACIS). A core inventory (ACOIN) has been published and development of the complete ACIS is progressing.

Occupational Health & Safety has not been neglected in the overall program; a very advanced scheme developed by government, industry and trade unions is coming into law.

Pollution control and the landing and disposal of wastes are also controlled with stringent safety standards.

In all areas there has been increasing liaison between government, industry, trade unions, and consumer organizations to produce effective and productive legislation. This legislation is not yet in place in every State in Australia but progress is accelerating as model legislation to the best international standards is drafted and processed into law.





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## Society Meetings

### BIRMINGHAM ..... MAY

#### "Two-Component Spraying Technology"

A certificate of Club Honorary Membership was presented to Stanley Ray. Mr. Ray joined the Club in 1947. He was awarded Honorary Life Membership in 1963 and became Club President in 1965.

Reg Pyatt, of Binks Bullows Ltd., discussed, "Two-Component Spraying Technology."

Using a slide presentation to display examples of two-component spray finishes and the necessary equipment, Mr. Pyatt enumerated several advantages to the finishes: high coatings resistance; flexibility; economy; coatings durability; and high solids. An explanation of the methods used to achieve two-component spraying followed. DAVID M. HEATH. Secretary

## CLEVELAND ..... MAY Awards Night

Officers elected for the 1985-86 year include: President—Scott Rickert, Case Western University; President-Elect—Madelyn K. Harding, Sherwin Williams Co.; Secretary—Richard Eley, Glidden Coatings & Resins Div.; Treasurer—Ed Bishop, Jamestown Paint & Varnish Co.; Assistant Treasurer—Bob Brumfield, Day-Glow Color Corp.; and Member-at-Large—Ilona Nemeth, Sherwin-Williams Co.

Twenty-five year pins were presented to Harry Scott, of Glidden Coatings & Resins Div.; Lou Hromadka, of Gibson-Homans Co.; and Howard Rupender, of Harshaw/ Filtrol Partnership.

Richard Eley, currently the Society's Assistant Treasurer and Educational Committee Chairman, was presented with an award in recognition for outstanding service to the Society and the industry.

The speaker for the evening was Leonard Dick, of PPG Industries, Inc. Mr. Dick discussed "COLOR AND STYLING."

MADELYN K. HARDING, Secretary

## LOS ANGELES ......JUNE "Operation of a Vertical Sawmill"

A moment of silence was observed for Carl King who died recently.

The following slate of officers was elected to serve for the year 1985-86: President—Mike Gildon, Guardsman Chemicals; Vice-President—Henry Kirsch, Trans Western Chemicals, Inc.; Sceretary—Melinda Rutledge, of Allo Chemicals; Treasurer—Ray DiMaio, Koppers Co., Inc.; and Society Representative—Jan P. Van Zelm, Byk Chemie.

Romer Johnson presented Jan Van Zelm with an Outstanding Service Award for his



## **Constituent Society Meetings and Secretaries**

BALTIMORE (Third Thursday—Eudowood Gardens, Towson, MD). ED COUN-TRYMAN, Bruning Paint Co., 601 S. Haven St., Baltimore, MD 21224. Virginia Section—Fourth Wednesday, Ramada Inn-East, Williamsburg, VA.

BIRMINGHAM (First Thursday—Strathallan Hotel, Birmingham, England). D.M. HEATH, Holden Surface Ctgs. Ltd., Bordesley Green Rd., Birmingham B9 4TQ England

CHICAGO (First Monday—meeting sites vary). RAYMOND CZICZO, Reliance Universal, Inc., 1915 Industrial Ave., Zion, IL 60099.

CDIC (Second Monday—Sept., Jan., Apr., June in Columbus; Oct., Dec., Mar., May in Cincinnati; and Nov., Feb. in Dayton). SAMUEL KRATZER, D&L Paint Co., 215 Brownsville Ave., Liberty, IN 47343.

CLEVELAND (Third Tuesday—meeting sites vary). RICHARD ELEY, Glidden Coatings & Resins, Div. of SCM Corp., D.P. Joyce Research Center, P.O. Box 8827, Strongsville, OH 44136.

DALLAS (Thursday following second Wednesday—Executive Inn, near Lovefield Airport). FREDERICK T. BEARD, Glidden Coating & Resins, Div. of SCM Corp., 1900 North Josey Ln., Carrolton, TX 75006.

DETROIT (Fourth Tuesday—meeting sites vary). PETER KNIGHT, Dow Chemical Corp., 2020 Dow Center, Midland, MI 48640.

GOLDEN GATE (Monday before third Wednesday—Alternate between Sabella's Restaurant on Fisherman's Wharf and Francesco's in Oakland, CA).

HOUSTON (Second Wednesday—Sonny Look's, Houston, TX). JAMES W. JUDLIN, Devoe Reynolds, Inc., 676 Kerbyville Rd., Houston, TX 77003.

KANSAS CITY (Second Thursday—Cascone's Restaurant, Kansas City, MO). STEVEN JOHNSON, Cook Paint & Varnish Co., P.O. Box 389, Kansas City, MO 64141.

LOS ANGELES (Second Wednesday—Steven's Steak House, Commerce, CA). MELINDA RUTLEDGE, Allo Chemical Co., P.O. Box 443, Ontario, CA 91761.

LOUISVILLE (Third Wednesday—Breckinridge Inn, Louisville, KY). LARRY F. PITCHFORD, Reynolds Metals Co., P.O. Box 3530, Plant III, Louisville, KY 40232.

MEXICO (Fourth Thursday-meeting sites vary).

MONTREAL (First Wednesday-Bill Wong's Restaurant).

NEW ENGLAND (Third Thursday—Hillcrest Function Facilities, Waltham, MA). GAIL POLLANO, Polyvinyl Chemical Industries, Inc., 730 Main St., Wilmington, MA 01887.

NEW YORK (Second Tuesday—Landmark II, East Rutherford, NJ). John W. Burlage, Pacific Anchor Chemical, 14 Ridgedale Ave., Cedar Knolls, NJ 07927.

NORTHWESTERN (Tuesday after first Monday—Jax Cafe, Minneapolis, MN). JOAN B. LAMBERG, Horton-Earl Co., 750 S. Plaza Dr., St. Paul, MN 55120.

PACIFIC NORTHWEST (Portland Section—Tuesday following second Wednesday; Seattle Section—the day after Portland; British Columbia Section—the day after Seattle). YVON POITRAS, General Paint Co., 950 Raymur Ave., Vancouver, B.C., V6A 3L5, Canada.

PHILADELPHIA (Second Thursday—Dugan's Restaurant, Philadelphia, PA). THOMAS L. PETA, J.C. Osborne Chemicals, Inc., P.O. Box 1310, Merchantville, NJ 08109.

PIEDMONT (Third Wednesday—Howard Johnson's, Brentwood Exit of I-85, High Point, NC).

PITTSBURGH (First Monday—meeting sites vary). MARK TROUTMAN, Bradley Paint Co., 608 W. Crawford St., Connellsville, PA 15425.

ROCKY MOUNTAIN (Monday following first Wednesday-Bernard's, Arvada, CO). MARCY S. BAUGH, Hutson Industries, 60 Tejon St., Denver, CO 80223.

ST. LOUIS (Third Tuesday—Salad Bowl Restaurant). JAMES N. MCDERBY, F.R. Hall & Co., 6300 Bartmer Ind. Dr., St. Louis, MO 63130.

SOUTHERN (Gulf Coast Section—Third Thursday; Central Florida Section— Third Thursday after first Monday; Atlanta Section—Third Thursday; Memphis Section—bi-monthly on Second Tuesday; Miami Section—Tuesday prior to Central Florida Section). C. LEWIS DAVIS, Ambrosia International, 802 Black Duck Dr., Port Orange, FL 32019.

TORONTO (Second Monday-Cambridge Motor Hotel).

WESTERN NEW YORK (Third Tuesday—meeting sites vary). JEAN L. LUCK, Pratt & Lambert Inc., Powder Coatings Div., P.O. Box 22, Buffalo, NY 14240. dedicated service to the Society. Mr. Johnson also mentioned that a 25-year pin for Al Yerby, recently deceased, would be sent to his wife.

RAY DIMAIO, Secretary

## PIEDMONT......MAY "Influence of Pigments on Properties

"Influence of Pigments on Properties of Paint Films"

The Executive Committee nominated Jim Albright to be the next Society Representative. Mr. Albright succeeds Gary Marshall, who served in the position for six years.

Educational Committee Chairman, Bob Matejka, reported that several students have been employed by member companies for summer and full-time employment. He added that the Society is being recognized as a legitimate employer of chemists by the local universities.

Rolf H. Odenthal, of Mobay Chemical Corp., spoke on "The INFLUENCE OF INAC-TIVE AND ACTIVE PIGMENTS ON THE CORRO-SION INHIBITING PROPERTIES OF PAINT FILMS."

Using a slide presentation, Dr. Odenthal discussed the inactive pigments or synthetic iron oxides. Pigment particle size determines the shade of the pigment and can significantly influence the performance of iron oxide pigments in primer systems, explained Dr. Odenthal.

He stated that particle distance greatly influences the corrosion inhibiting properties of a primer using synthesis oxides. Test results indicate that formulating with coarser iron oxides at higher PVC levels yields better corrosion resistance results. When particle distance was 0.5 microns or less, corrosion resistance improved markedly. He added that complete melting of pigments and a degree of flocculation improve the corrosion inhibiting capabilities of a primer.

A discussion of the mechanisms used by active pigments to retard corrosion concluded his presentation. Dr. Odenthal elaborated on two such mechanisms: the formation of metal soaps to prevent saponification of the binder and passivating irons. Dr. Odenthal discussed why the effectiveness of active pigments is dependent upon the PVC of the system. At lower PVC levels, he explained, an inactive pigment such as red iron oxide outperforms an active pigment, zinc chromate, because of the low opacity of the zinc chromate. This low opacity allows a faster UV degradation of the binder. At higher PVC levels, the zinc chromate performs as expected. Therefore, the active zinc chromate pigment exhibits better corrosion resistance than the inactive iron oxide pigment.

STEVE LASINE, Secretary

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## Elections

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JOHNSTON, JEFF-Sperex Corporation, Gardena, CA.

TAMURA, JOE—Imperial Coatings Co., Ontario, CA.



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WARHANK, JOHN D. — Industrial Process, Lomita, CA.

#### Associate

MENEES, JAMES-Petro-Diamond Inc., Irvine, CA.

Retired

HERSHEY, ALAN U.-Long Beach, CA.

#### KAZMI, SYED QAMAR—New York Bronze Powder Co., Elizabeth, NJ.

- PERSON, CARL F.-International Paint, Union, NJ.
- SIRENO, ROBERT C. --- Reliance Universal, Somerset, NJ.
- WRIGHT, WAYNE E.—Interstab Chemicals, New Brunswick, NJ.

#### Associate

AARONSON, PHILLIP D. — Polymer Applications, Tonawanda, NY.

Ballin, Harold—Excel Products, Bronx, NY. FALSONE, JOHN J.—Spencer Kellogg, Newark, NJ.

## PACIFIC NORTHWEST

Active

HAYWOOD, BRADLEY C.—United Paint Mfg. Co., Greenacres, WA.

## PHILADELPHIA

Associate

JORDAN, DENNIS P.—L. V. Lomas Chemical Co., Williamsville, NY. MARKS, ALAN D.—Paint Manufacturer's Group, Lansdale, PA.

## PITTSBURGH

#### Active

CHRISTENSON, JAMES R. — PPG Industries, Inc., Springdale, PA.

## TORONTO

## Active

CLARK, DALE-Madison Chemical Ind., Milton, Ont.

- HARRISON, LEONARD—Rust-Oleum (Canada) Ltd., Downsville, Ont.
- PATEL, RAJENDRA D. Denalt Paints Ltd., Downsview.
- SHAYBAN, FOUAD—Chemcraft Sadolin, Port Hope, Ont.
- YOUNG, DOUGLAS K. Arterial, Toronto, Ont.

#### Associate

CORCORAN, BRIAN M.—Berolkemi Inc., Mississauga, Ont.

SHILLUM, ROBERT L. — Trebor Industries Ltd., Toronto, Ont.

NEW YORK

Ardsley, NY.

Active AGOSTINO, MARCIA G.—Ciba-Geigy Corp.,



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## People

Joseph G. O'Grady has been appointed President of ASTM, Philadelphia, PA. Mr. O'Grady joined the staff of ASTM as Executive Vice-President in 1984. As a volunteer, he served for eight years on the ASTM Board of Directors and was Chairman of the Board from 1978-79.

In his new capacity, Mr. O'Grady will direct the ASTM headquarters staff in Philadelphia and the Washington D.C. office which serve the needs of 140 technical committees and 30,000 members worldwide who produce voluntary consensus standards, special technical publications, and other documents sold internationally.

Prior to joining ASTM, Mr. O'Grady was Vice-President of the PSE&G Research Corporation and directed the activities of the corporate Research and Testing Laboratory in Maplewood, NJ.

Toyo Soda Mfg. Co., Ltd., Tokyo, Japan, has appointed **Robert G. Short**, President of their American subsidiary, AZS Corporation, Atlanta, GA. With the promotion, Mr. Short becomes a member of the AZS Board of Directors.

During the Chemical Manufacturers Association's Annual Meeting held in White Sulfur Springs, WV, George J. Sella, Jr., Chairman of the Board, President, and Chief Executive Officer of American Cyanamid Co., was elected Chairman of the CMA Board. He succeeds Edwin C. Holmer, President of Exxon Chemical Co.

Elected to other positions on the CMA Board were: Vice-Chairman—Harold A. Sorgenti, ARCO Chemical Co., Div. of Atlantic Richfield Co.; and Executive Committee Chairman—W.H. Clark, Jr., Nalco Chemical Co. Robert A. Roland was reelected President of the association.

Tioxide America, Inc. has appointed L. Gerard Skinner a U.S. Marketing Manager for titanium pigments and TIL organo metallic chemicals. A member of the New England and Southern Societies, Mr. Skinner will also serve as a Director of the company.

A.D. Roos, Vice-President and Managing Director of Glidden Co. Ltd., has been elected Chairman of the Board of the Canadian Paint and Coatings Association, Montreal, Que., Canada. He succeeds Ken G.W. Smith, of DuPont Canada, Inc. Mr. Roos will serve a two-year term.





J.G. O'Grady

R.G. Short



J.D. Wolf



A. Errico

J. David Wolf, formerly General Manager of the Chemical Div. of Goodyear, Akron, OH, has been appointed to Division Vice-President and General Manager. Mr. Wolf began his Goodyear career as a Research Chemist in 1961.

Stanley T. Haduch has accepted the position of Senior Sales Representative with Ball Chemical Co., Glenshaw, PA. Mr. Haduch will be responsible for industrial finishes and pipe coating sales in Pennsylvania, West Virginia, and Ohio. He is a member of the National Association of Corrosion Engineers.

To more effectively serve the numerous industry markets utilizing coated coil, the National Coil Coaters Association has restructured its Technical Section. The Technical Section Steering Committee will include: Chairman—G.R.K. Lye, Baycoat Ltd.; Operating Assistant Chairman— Timothy Kelley, Roll Coater, Inc.; Support Assistant Chairman—Robert Moorman, Glidden Coatings & Resins Div.; Marketing Section Chairman—Ronald A. Sistrunk, Inland Steel Co.; and Marketing Section Assistant Chairman—Richard Brand, Enamel Products & Plating Co.

Ferro Corp., Cleveland, OH, has promoted **Edward J. Duda** to International Product Manager for the Coating Division's powder coatings group. Previously the Western Regional Sales Manager, Mr. Duda joined Ferro in 1978.

Jan A. Juchnowski has been named a Sales/Service Representative for Ferro's powder coatings group. Mr. Juchnowski will provide sales and technical service to Indiana and western Michigan. Anthony Errico, Chemicals Advertising Supervisor with Eastman Chemical Products, Inc. since 1967, has retired effective July 1. Mr. Errico began his professional career as a coatings research chemist with Centro Research Labs in Briarcliff, NY. Prior to joining Eastman, he served as Editor of Organic Finishing and Paint & Varnish Production (now Modern Paint & Coatings).

Alan M. Sandler has been named Director of Marketing for Duron, Inc., Beltsville, MD. He will direct the expanding marketing operations of the company, which serves an eight-state region.

Richard Chodnicki has replaced Arnold G. Mix as the Eastern Regional Manager for the Pigment Division of Kerr-McGee Chemical Corp., Oklahoma City, OK. Mr. Chodnicki, a member of the Philadelphia Society, will relocate to the eastern regional office in Parsippany, NJ.

Henkel Corporation, Minneapolis, MN, has named **Frank Precopio** Chief Operating Officer. Previously Executive Vice-President, Dr. Precopio will continue to serve as General Manager of the Amchem Products Div. of Henkel located in Ambler, PA.

Harold P. Wulff has accepted the position of Vice-President and General Manager of the Organic Products Div. of Henkel. Dr. Wulff will serve in the Maywood, NJ, administrative offices.

The position of Vice-President of Henkel's Chemical Services Div., has been filled by **William K. Viel.** Mr. Viel assumes his duties at the Burlington, IA, facilities.

## FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY Announces the



896 Pages Over 1,400 Spectra Over 1.500 References

This revised and expanded 896-page book (by the Chicago Society for Coatings Technology) contains a compilation of 1433 spectra, fully indexed, of materials commonly used in the coatings

industry. Spectra of many recently marketed materials are included, as well as examples of Fourier transform infrared spectra.

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PPG Industries, Inc., Allison Park, PA, has named **Leonard W. Magnusson** an Engineering Associate of its Coatings and Resins facilities. Mr. Magnusson is a member of the Pittsburgh Society.

Andrew J. Lauer, of PPG's Coatings and Resins Group, has been promoted to Senior Development Chemist. Mr. Lauer is also a member of the Pittsburgh Society. During the American Oil Chemists' Society's 76th Annual Meeting held in Philadelphia, PA, Joyce L. Beare-Rogers was installed as the 1985-86 President. Dr. Beare-Rogers, Chief of Nutrition Research for the Health Protection Branch, Dept. of Health and Welfare, Ottawa, Canada, has been an active member of the society for the past 25 years. She succeeds Nicholas Pelick, of Supelco Inc. as President. Frank B. Redman, of Crown Decorative Products Ltd., Darwen, Lancs., England, was elected President of the Oil and Colour Chemists Association for 1985-87. Installation was held during the June Biennial Conference of OCCA in Edinburgh, Scotland. Mr. Redman succeeds Cecil N. Finlay, of Durham Chemicals Ltd.

W. Ronald Hoopes has been named National Sales Manager for specialty chemicals for W. R. Grace & Co.'s Organic Chemicals Div., Lexington, MA. Previously, he served as Commercial Manager for the firm's Productos Darex S.A. in Mexico and Central America.



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## Obituary

Alfred L. Hendry, President of A.L. Hendry & Co., Tampa, FL, died June 16, 1985. Mr. Hendry, a Past-President of the Southern Society

(1979-80), was 60.

While attending the Georgia Institute of Technology, Mr. Hendry worked at Tampa Paint Co. during summers. His professional career included employment at Harris Paint, Weatherproofing



Paint, Tung Oil Products, and Protective Coatings, before founding A.L. Hendry & Co.

He was a member of the Central Section of the Southern Society, the Florida Paint & Coatings Association, and the Society of Gallows Birds.

Mr. Hendry is survived by his wife, Colene; a daughter, Laurie; and a sister.

James E. Heckel, an Honorary Member of the Federation and an Honorary Member of the Baltimore and Philadelphia Societies, died on February 3, 1985. He was 90. Associated with R. T. Vanderbilt Co. for many years, Mr. Heckel was a son of one of the founders of the Federation (George B. Heckel, Sr.) Mr. Heckel was a Past-President of the Philadelphia Paint and Coatings Association.

Another son of Mr. Heckel, Sr., George B. Heckel, Jr., died in June 1985. He was 89 years old. He was associated with his father in the paint industry publishing business and served as Secretary of the Philadelphia PCA.



# COME TO SUN TECH BOOTH 1105 At the paint show

OSHA's newest workplace standard will begin to take effect in November. Are you really ready for it?

We'll be at the 50th Annual Paint Industries' Show, Oct. 7–9, to help you understand this complex new regulation that impacts directly on paint and coatings manufacturers, their suppliers, and their customers.

As a service to the coatings industry, a major part of our booth at the show will be set aside as a classroom. That's Sun Tech. And in about 10 minutes of your time, our staff will get you up to speed on everything

> essential you have to know about the new OSHA standard.

You'll get a chance to ask your own questions, of course. And we'll provide useful supplementary material to take back with you...plus a certificate

indicating that you completed the OSHA course at Sun Tech. Classes will be held at posted intervals at booth 1105. So stop in and enrol. After all, a little extra learning never hurts—especially where OSHA is involved.



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## **Birmingham Club Celebrates 50th Anniversary**

The Birmingham Paint, Varnish and Lacquer Club celebrated its Fiftieth Anniversary as a constituent of the Federation of Societies for Coatings Technology at a special dinner, June 21, in Birmingham, England. About 125 persons attended.

Club President Roland L. Staples presided over the ceremonies which included words of congratulations from: Joseph A. Bauer, President of the Federation; Frank Carter, the Lord Mayor of the City of Birmingham; and F.C. Graves, President of the Birmingham Chamber of Commerce; and reminisces by Mr. Staples. John R. Green, Club Social Officer, served as Toastmaster.

Also present from the Federation were: Past-President and Board Member Neil S. Estrada and Lily; Past-President and Board Member Michael W. Malaga and Margaret; Southern Society President and Federation Board Member James E. Geiger and Lynne; and EVP Frank J. Borrelle and Rose.

Club officers this year are: President— Mr. Staples, of Midland Specialty Powders Ltd.; President-Elect—Ron Jukes, of Croda Paints Ltd.; Secretary—David M. Heath, of Holden Surface Coatings Ltd.; and Treasurer—Stan V. Bretell, of Llewellyn Ryland Ltd.



At the 50th Anniversary Dinner of the Birmingham Club. Left to right: Roland Staples, Club President; Frank Carter, Lord Mayor of Birmingham; Mrs. Carter; and Joseph Bauer, President of the Federation.

## OCCA Annual Convention Held in June; Frank Redman Elected President

Frank B. Redman, Development Manager, Solvent-Based Paints, Crown Decorative Products Ltd., Darwen, Lancs., England, was elected President of the Oil and Colour Chemists Association for 1985-87. Installation took place during OCCA's Biennial Conference on June 28, in Edinburgh, Scotland. He succeeds Cecil N. Finlay, of Durham Chemicals Ltd.

About 250 persons attended the Conference which featured the presentation of 23 papers under the theme, "New Substrates, New Materials, New Problems."

The paper sponsored by the Federation was "Cationic Electrodeposition Primers Over Zinc Coated Steels. I: Effect of Voltage and Substrates on Coating Quality." It was authored by Dr. Clifford K. Schoff and Huey-Jyh Chen, of PPG Industries, Inc., Coatings and Resins Research Labs., Allison Park, PA, and presented at the Conference by Dr. Schoff.

The Federation was represented at the Conference by President Joseph A. Bauer and Dorothy and EVP Frank J. Borrelle and Rose.

All of the papers presented at the Conference will be published in the OCCA Journal.

Members of the International Coordinating Committee held their annual meeting during the Conference. Present were: OCCA—Cecil Finlay, President; Frank Redman, President-Elect; Les Brooke, Honorary Secretary; Robert Hamblin, Director Secretary; FSCT—Joseph A. Bauer, President; FATIPEC—Amleto Poluzzi, Vice-President; Jacques Roire, Past-President; and Guttorm Abelsnes, President of SLF (Scandinavian Federation).



Members of the International Coordinating Committee who met during the OCCA Conference in Edinburgh, Scotland, on June 27, 1985. From left to right: Standing—Jacques Roire, Past-President of FATIPEC; Robert Hamblin, Director Secretary of OCCA; Les Brooke, Hon. Sec. of OCCA; Frank Redman, President-Elect of OCCA. Seated—Amleto Poluzzi, Vice-President of FATIPEC; Joseph Bauer, President of FSCT; Cecil Finlay, President of OCCA; and Guttorm Abelsnes, President of SLF.

# PICTORIAL STANDARDS OF COATINGS DEFECTS

Revised and updated edition of this manual (previously titled "Exposure Standards Manual") has been compiled in conjunction with the American Society for Testing and Materials, and includes definition, description, and photographic standards for each of the following defects: Adhesion; Blistering; Chakking; Checking; Cracking; Erosion; Filiform Corrosion; Flaking; Mildew; Print; Rust; Traffic Paint Abrasion and Chipping.

Also included is reference information on supplementary standards, along with sample record sheets for compiling exposure data.

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## Federation of Societies for Coatings Technology

## Color and Appearance Technology Is Subject of HunterLab Courses

HunterLab, Reston, VA, has released its 1985-86 schedule of educational courses on color and appearance technology. The three programs available include Regional Seminars, Workshops, and Service Seminars.

"Regional Color and Appearance Seminar" is a two-day introductory course which provides a basic background of color science to those concerned with the measurement of a product's appearance. The seminar combines one day of lectures on color theory, sample handling and preparation, and instrument technology, with an applications laboratory held on the second day. The first group of seminars scheduled is as follows: Sept. 18-19—Detroit, MI; Oct. 16-17—Minneapolis, MN; Nov. 6-7—Atlanta, GA; Nov. 20-21—Newark, NJ; Dec. 4-5—Los Angeles, CA; Jan. 22-23—Philadelphia, PA; Feb. 5-6—Seattle, WA; and Feb. 19-20—Dallas, TX.



See us at Booth 612 Paint Industries Show

Additional seminars have been scheduled from March through June and will be announced at a later date.

"Workshop on the Measurement of Appearance" is a two and one half day course which provides an in-depth look at the field of color and appearance measurement. Included are lectures, discussions, and an applications session where students receive "hands-on" experience in the instrumental measurement of their product samples. These workshop sessions will be held in Reston, VA, at HunterLab headquarters on Oct. 16-18; Feb. 26-28; and May 14-16.

"Service Seminar" is designed for owners of the D25-2 and D25-9 Colorimeters. These one-day seminars are practical training sessions to assist the instrument owner in basic maintenance and adjustment procedures. A brief review of color theory is included in these seminars, which will be held in Reston, VA. D25-2 Colorimeter will be covered on Dec. 11 and D25-9 on Feb. 5 and March 20.

For additional information, contact the Marketing Services, HunterLab, Inc., 11495 Sunset Hills Rd., Reston, VA 22090.

## Fall Short Courses Announced by Kent State

Two fall short courses have been scheduled by the Coatings Division of the Chemistry Department and the Conference Bureau at Kent State University, Kent, OH.

"Painting Processes: Industrial Paint Application Technology" will be held from November 4-5. This program is intended to familiarize those in the paint, resins, pigment, solvents, and additives industries with equipment, operation, and application factors that the user experiences. It will be taught by Prof. Norman R. Roobol, of GMI Engineering and Management Institute and NR Consulting Services. Prof. Roobol will discuss the industrial coatings processes with emphasis on application; and the economy, ecology, safety, and energy requirements for numerous means of paint application and cure.

"Introduction to Coatings Technology" has been scheduled from November 18-21. Professor John A. Gordon, Jr., of Eastern Michigan University and Pacific Technical Consultants will be the lecturer. He will describe the coatings materials, formulating techniques, manufacture, testing and new trends in coatings. This program is directed towards newcomers and those wanting an overview for personnel in coatings laboratories, production units, and marketing groups.

Additional details on the courses can be obtained by the Program Manager, Carl J. Knauss, Kent State University, Chemistry Dept., Kent, OH 44242.

## New England Society Symposium May 15-16, 1986

The New England Society for Coatings Technology will sponsor a spring symposium on May 15 and 16, 1986. The theme for the symposium is "Launching the New Revolution—Compliance for the 21st Century."

The program will consist of invited lectures and contributed papers. Those interested in presenting contributed papers are invited to submit abstracts (no more than one page) on subjects related to the theme, including compliance coatings, devices, right-to-know legislation, and hazardous waste.

For additional information, contact Maureen Lein, Davidson Rubber Co., Industrial Park Dr., Dover, NH 03820.



## Coatings Inspection Training Offered by KTA-Tator, Inc.

KTA-Tator, Inc., Pittsburgh, PA, is sponsoring a series of five intensive threeday seminars for coating inspection.

Designed to train or refresh coating personnel in the fundamentals of coatings technology, application, and inspection procedures, the training includes extensive hands-on use of instruments for thickness measurement, holiday detection, adhesion, determination of ambient conditions, etc. In addition, each attendee will learn how to assess blast cleaning and spray painting equipment and techniques through actual use of production equipment.

Other sessions cover corrosion theory; surface preparation techniques and equipment; coating application techniques and equipment; types, calibration, and use of inspection instrumentation; coating material types and characteristics; problem solving; and coating failure analysis. Course dates and titles are as follows:

Sept. 24-26—"Inspection of Coatings and Linings for Immersion Service"

Oct. 15-17—"Maintenance Painting Practices"

Nov. 13-15—"Nuclear Power Coating Inspection"

Mar. 4-6—"Coating Inspection of Chemical Plants"

Mar. 26-28—"Bridges and Highway Structures Coating Inspection"

For additional information, contact Bill Corbett, KTA-Tator, Inc., 115 Technology Dr., Pittsburgh, PA 15275.



## **Market Analysis**

The results of an industry study providing detailed statistics and analysis of the size, growth, and projected growth of the overall paint and coatings industry as well as its individual market segments have been released. The 250-page report is designed to provide top-level management with the most comprehensive investigation on the U.S. paint and coatings industry. Order inquiries should be directed to Charles J. Ritchie, Reports and Publications Dept., Business Trend Analysts, Inc., 2171 Jericho Turnpike, Commach, NY 11725.



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## Porosimeter

A new porosimeter which features manual, semi-automatic, or automatic operation has been described in a technical bulletin. User friendly software guides the operator through instrument operations by means of screen prompts. Performance specifications can be obtained from Micromeritics Instrument Corp., 5680 Goshen Springs Rd., Norcross, GA 30093.

## **Micaceous Coating**

A brochure details the performance properties of micaceous iron oxide, an effective raw material for weather resistant metal coatings. The coating is used primarily in intermediate and finishing coats in association with inhibitive primers. For copies of the brochure, write to Reichard Coulston, Inc., 1421 Mauch Chunk Rd., Bethlehem, PA 18018.

## **Pultrusion Colorants**

Recently developed and described in literature is a line of color dispersions for the pultrusion market. The pigments are dispersed in several specialized polyester vehicles, insuring complete compatibility. Write: James Steever, Pigment Dispersions Inc., P.O. Box 412, 54 Kellogg Ct., Edison, NJ 08818 for detailed information.

## Vinyl Copolymer

A high solids vinyl copolymer which permits the formulation of fast air dry, low VOC industrial finishes has been introduced in a product bulletin. Resultant finishes are reported to demonstrate excellent hardness and gloss, good recoatability, and adhesion to plastics. Complete data on Styresol 13-510 is offered by Reichhold Chemicals, Inc., Resins & Binders Div., 407 S. Pace Blvd., Pensacola, FL 32596.

## **Filling Machine**

A new model filling machine is featured in literature. Designed to fill one to five gallon containers at high speed with abrasive liquids or textured paint, the machine is available in a fully automatic or semiautomatic model with a dual or single filling head. Additional details on the Spectrum I BF machine are offered by Beltron Corp., P.O. Box 893, Red Bank, NJ 07701.

## **Coupling Agent**

A polymeric coupling agent recommended for polyethylene, polypropylene and polyolefin elastomers has been developed and is described in literature. The new silane-modified polyolefin has multiple sites for reactivity with both fillers and polymers. Data sheets on the CPS 078.5 and CPS 076 are available from Petrarch Systems, Inc., 2731 Bartram Rd., Bristol, PA 19007.

## Media Hill

A new high speed small media mill provides continuous throughput in wet grinding and dispersion of suspension. The completely enclosed new model is reported to minimize atmosphere pollution and provide high efficiency in pumping either low or high viscosity liquids. For complete details on the Dyno-mill, write to Dept. RN, Chicago Boiler Co., 1965 Clybourn Ave., Chicago, IL 60614.

## **Cationic Compounds**

Cationic compounds are outlined in a free technical pamphlet. The compounds are active in water and oil-based systems and are reported to: prevent caking in acid cleaners and silicas; inhibit corrosion in metal work and petroleum products; and act as an emulsion stabilizer in agricultural chemicals and building materials. Contact Hodag Corp., 7247 N. Central Park Ave., Skokie, IL 60076 for copies.

#### **Epoxy Resin Booklet**

Recently issued is a 12-page booklet covering epoxy resins and hardeners. Epoxy resins for a wide range of applications including casting, electrical potting and encapsulation, laminating and filament winding, flooring and surfacing, coatings, adhesives, and sealants are contained in the booklet. For copies, contact Reichhold Chemicals, Inc., Resins & Binders Div., 407 S. Pace Blvd., Pensacola, FL 32596.

## Dispersions

A new series of color dispersions for reaction injection molding (RIM), microcellular urethanes, high density molded foams, and other high molecular weight polyurethanes has been introduced. The colorants are offered in standard colors and computer color matched blends. Contact James Steever, Technical Director, Pigment Dispersions Inc., 54 Kellogg Ct., P.O. Box 412, Edison, NJ 08818 for technical data on the "5000 Series" of color dispersions.



## **Spectral Chart**

A new four-color wall chart illustrates the spectral emission characteristics of several commonly used weathering devices. The chart shows energy data plotted in the wavelength region from 250 to 800 nanometers and is useful in determining how close the output of a particular artificial weathering device is to that of the sun over a given wavelength range. To receive a copy, write to Additives Dept., Ciba-Geigy Corp., Three Skyline Dr., Hawthorne, NY 10532.

## **Color Analyzer**

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## **Book Review**

## RADIO FREQUENCY/RADIATION AND PLASMA PROCESSING: INDUSTRIAL APPLICATIONS AND ADVANCES

Edited by P.N. Cheremisinoff, O.G. Farah, and R.P. Ouellette

Published by Technomic Publishing Co., Inc. Lancaster, PA \$45.00, 213 pages

Reviewed by Dr. Joseph V. Koleske Union Carbide Corp. S. Charleston, WV

This book is an interesting collection of papers from the International Conference on Electrotechnologies in Industry that was held in Montreal, Canada, in 1982. It contains valuable information about how these techniques can be applied to industrial processes. The applications areas are extremely varied and include ceramic, chemical, coating, food, metal, mineral, plastics, rubber, textile, as well as other industries. A number of processes, including dielectric, microwave, radiowave, infrared, laser, and electron beam, are discussed by leading authorities in the field of electrotechnology.

The book, which is divided into a radio frequency and radiation processing part and a plasma processes part, has specific and detailed information about uses for the technology, its economics, and its scientific background. For example, one section contains over 100 references to industrial microwave applications as they apply to food processes and preparation, to natural materials—textiles, leather, paper, and wood—to minerals, to chemicals including the curing of coatings and crosslinking of polymers, and so on. Other sections detail process economics with specific calculations and current costs.

This book should be of interest and use to those people who are involved with industrial materials and processes. This would include research and development



The book is not without faults. For example, one of the sections has reference numbers listed within the text, but no references are given at the end of the section. Another section contains a number of figures, yet there is no reference to the figures in the text. Some of the figures were difficult to read because of size reduction. Yet, the book contains valuable, current information and a reader can easily overlook these flaws.

## INDUSTRIAL SOLVENTS HANDBOOK

Edited by Ernest W. Flick

Published by Noyes Data Corp. Park Ridge, NJ \$86.00, 648 pages

Reviewed by William H. Ellis El Segundo, CA

This book is a compilation of a vast amount of trade literature in a single 648page volume. As stated in the introduction, the information was selected from manufacturers' literature and, in most cases, has been directly reproduced. This approach results in a wide variation in the format and amount of data given for different products. In most cases, physical properties are simply tabulated. Where more data were available, such properties as azeotropic compositions and temperatures, solubilities, thermal properties, effects of temperature on densities, vapor pressures, and viscosities are includedsometimes as graphs.

The book includes almost all materials that can even remotely be considered industrial solvents. The categories are hydrocarbons, halogenated hydrocarbons, nitroparaffins, organic sulfur compounds, alcohols, glycols, phenols, aldehydes, ethers, glycol ethers, ketones, acids, amines, and esters. Most products are listed under the names of individual chemical compounds. Some of the class-





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ifications, and many of the individual items, however, are seldom, if ever, used as solvents. Some are gases: some are solids. Many would be better described as chemical intermediates. Although this does not diminish the usefulness of the book, it is misleading and may also limit its appeal because potential users may not realize its broader aspects. The title could be changed to indicate the greater scope. One obvious omission is Chevron solvents. Although Chevron Corporation is the largest manufacturer and marketer of solvents west of the Rocky Mountains, none of its products are listed. Incongruously, Chevron alpha olefins are listed, but they are chemical intermediates, not solvents.

On balance, the "Industrial Solvents Handbook" would be a valuable addition to a reference library. It contains much information on commercial organic materials not otherwise as conveniently available. The user should recognize, however, that data are for commercial, not necessarily pure, compounds. Thus, the publication is not a direct substitute for a general chemistry handbook.

## WATER-SOLUBLE SYNTHETIC POLYMERS: **PROPERTIES AND BEHAVIOR** VOLUMES I and II

Written by Philip Molyneux

Published by CRC Press. Inc. Boca Raton, FL Vol. I: \$75, 240 pages Vol. II: \$83, 280 pages

Reviewed by Thomas J. Miranda Whirlpool Corp. Benton Harbor, MI

These two volumes deal with the physical chemistry of water-soluble polymers. Volume I contains an introduction and three chapters covering nonionic polymers including the (poly)oxides, ethers, and imines. In another chapter, acrylic polymers are treated and in the last chapter, vinyl polymers such as vinyl acetate are discussed.

The general structure of these volumes covers: General Features, Production and Purification, Fractionation and Characterization, Bulk Polymer Properties, Solution Behavior, Phase Equilibria, Chemical Behavior, and Analysis.

Volume II is primarily concerned with the Physical Chemistry aspects of Polyelectrolytes and their Solution Behavior, Interaction with Small Molecule Cosolutes, Interaction between Polymers, and Polymers at the Interface.

The author has assembled numerous tables of properties of water-soluble polymers and includes extensive references and an appendix of physical constants. This book is a useful reference to those involved in water-soluble polymers as used in coatings for thickeners, ultrafiltration, stabilizers, flow control agents, and in waste treatment.

## RECENT ADVANCES IN LIQUID **CRYSTALLINE POLYMERS**

Edited by L. Lawrence Chapoy

Published by Elsevier Applied Science Publishers London and New York \$60.00, 339 pages

Reviewed by D.B. Kirby Whirlpool Corp. Benton Harbor, MI

Liquid crystalline polymers are one of the most exciting research topics in the polymer chemistry and engineering fields. These materials seem poised to become the next new members in the engineering plastics classification. Many of these polymers have practical advantages of high strength and stiffness, and excellent temperature resistance in formulations which can be easily melt-processed. Other current application interests lie in the areas of information retrieval, electronics, and opto-electronics.

This volume is based on the proceedings of the 1983 European Science Foundations's Sixth Polymer Workshop on Liquid Crystal Polymer Systems. Twenty-two papers are organized into four sections which are entitled: 1. Synthesis, Structure, Property Relationships; 2. Theory; 3. Characterization; and 4. Applications. Half of the papers are in the Characterization section where rheology, rheo-optics, thermal properties, and structural characterizations are discussed in detail.

The researcher in this field will find the volume to be an important addition to his reference shelf. It is broad in scope, international in source, and has a very handy subject index for efficient use.

## Hiding Power Equation Defended by Originator; Authors Contrast Early Model and 'New Approach'

### TO THE EDITOR:

The excellently written paper by Drs. Fitzwater and Hook in your February 1985 issue ["Dependent Scattering Theory: A New Approach to Predicting Scattering in Paints," JOURNAL of COATINGS TECHNOL-OGY, pp. 39-47] has described some difficulties that these authors apparently experienced in attempting to predict the performance of titanium dioxide pigments in practical paint systems using my hiding power equation, and also the discomfort that they felt in attempting to accept my concept of an orderly distribution of pigment particles in any paint film-based upon no stated reason other than their claim that "micrographs of paints generally show a fairly random distribution."

I should probably accept the truism that the author of any pioneering concept that is more than 25 years old is fair game to a new generation of "innovators," and meekly accept my fate. I feel, however, that a few comments from me may help to identify the cause of their difficulties, and perhaps even ease their discomfort.

In contrast to a short-term research project with the preconceived goal of developing such an equation, my development of the basic hiding power equation for titanium dioxide was almost serendipitous. It came about as the result of an effort to increase the accuracy (and understanding of) the hiding power curve for rutile titanium dioxide which had appeared in the sales literature of the Titanium Pigment Corporation ever since the introduction of that pigment in the early 1940's (and which was essentially no different than the present author's Figure 5).

This effort involved the statistical analysis of hiding power data accumulated over a period of nearly ten years, during which our laboratories had developed the first accurate test method for the determination of white hiding power, and had been extremely active in this area because of the technical service work involved in the final replacement of lithopone in paint formulations over the entire country. It was therefore based upon literally thousands of individual hiding power determinations and upon hundreds of repetitive determinations at any one given PVC. As anyone familiar with the mathematics will appreciate, the number of repetitions greatly affects the confidence that may be placed upon the statistical average.

When this more accurate data was plotted in the original units of the hiding power curve—spreading rate at 0.98 contrast ratio divided by pounds of titanium pigment per gallon versus PVC—its association with particle spacing came about simply from the sudden insight that this was the only physical change taking place in the system as PVC varied. Derivation of the hiding power equation then became simply a matter of determining what mathematical treatment of the calculated spacing would best fit the data.

The published equation fit the accumulated experimental data within the limits of accuracy of any single hiding power determination, which makes any argument that it can not be used to predict the performance of titanium dioxide in practical paint systems somewhat less than plausible, unless there is something radically different in how it has been employed by Drs. Fitzwater and Hook—as will be shown to be the case.

Insofar as the 0.74 "packing factor," of which much has been made in the recent article, is concerned, this is the unavoidable mathematical result of any assumption that all particles in a suspension are equidistant from all adjacent particles. Furthermore, it permits the equation to fit the data with a deviation of less than 2%, whereas the assumption of a cubic arrangement (as suggested by Drs. Fitzwater and Hook) resulted in a deviation of over 43%, as shown in my 1967 Mattiello Lecture. All particles in a cubic arrangement are, or course, not equidistant from all adjacent particles (consider the diagonals!).

I can assure the authors that I was initially no more happy than they at the necessity for assuming a completely uniform distribution of particles, but several circumstances combined to make it more acceptable to me—at least as a first approximation. Stereo micrographs (and, much later, stereo scanning electron micrographs) disclosed that the seemingly nonuniform dispersions of titanium dioxide seen in common photo-micrographs were merely artifacts created by the relative thickness of the examined film in relation to the particle size of the pigment. Particles that appear to be in contact, or nearly in contact, in either one of a stereo pair, are generally disclosed to be behind one another. Uniformity of dispersion is the rule, rather than the exception.

Furthermore, I was reassured by discussions with Dr. Peter J. DeBye, a Nobel Laureate at that time employed by the Titanium Pigment Corporation as a consultant, to the effect that only an orderly array of particles could possibly account for the observed fact that the scattering of individual particles was not additive. He was, of course, considering the scattering of particles of this radius of curvature (relative to the wavelength of light) to be the effect of diffraction, rather than refraction as suggested by Drs. Fitzwater and Hook.

Any objection that he might have had to the fact that such an orderly array of particles would produce an iridescent diffraction pattern was removed by the observed fact that all of our experimental dispersions of titanium dioxide pigment at PVC's at or above 30 PVC visually displayed such iridescence. This concentration coincides with that at which hiding power per gallon actually begins to decrease as more titanium dioxide is added to the system. This is contrary to the statement by the present authors that such iridescence is not observed in most TiO<sub>2</sub> based paints only if we assume that they have not limited their observations to practical paint systems (in which the concentration of titanium dioxide would be much lower, due to economic considerations) or to latex paints (in which the effect would be obscured by surface diffusion before such concentrations could be reached).

The authors' expressed hesitation to use my equation "on very dilute or very concentrated systems" (after having stated that it was good for most practical paints), would indicate that they have not read my 1962 paper, "The Geometry of White Hiding Power'' (which they do not reference). In that paper it was disclosed for the first time that the hiding power of titanium dioxide would become constant at PVC's below 10% (a fact that was confirmed, 10 years later, by Ross, in a paper that the present authors do reference). It was also disclosed that the hiding power of titanium dioxide would become constant, insofar as the equation was concerned, at the CPVC for the titanium dioxide pigment used. This

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will of course occur at a much lower PVC for a pigment such as high-oil-absorption "flat grade" containing only 76% titanium dioxide by volume than for an enamel grade.

It seems most probable that the authors' described difficulties in applying my equation to the substitution of one type of titanium pigment for another (as illustrated in their *Figure 2*), and for the solution of which they have proposed a brace of "fudge factors" to be determined for each pigment, are primarily due to the changes

they have made in the equation by substituting Kubelka-Munk scattering coefficients for hiding power, and pigment volume for pigment weight, although the actual mathematics of the substitutions are not completely clear.

Being a paint formulator and engineer, rather than a research scientist, I was constrained to communicate the results of my discovery in terms that would be most easily understood and applied by my fellow paint formulators. Consequently, my equation was couched in terms of hiding power



per pound of pigment, which was familiar to all paint formulators at that time, rather than scattering efficiency, which was definitely not, and it applied to an actual commercial grade (95%) of titanium pigment, rather than to a hypothetical "pure"  $TiO_2$ , as has apparently been assumed by Drs. Fitzwater and Hook. Since the term "pure" was rather commonly applied to all unextended titanium pigments at that time to distinguish them from the barium-base and calcium-base varieties, this is probably an understandable misinterpretation, but one which would seriously affect effective PVC calculations.

The hiding power per pound of pigment was calculated from actual determinations of hiding power per gallon of paints in which the film brightness was permitted to vary to conform with that which would be observed in practice. This creates a problem for anyone attempting to reproduce the same results based upon relative scattering, for the reason that hiding power involves a specific contrast ratio.

While it is often assumed that the hiding power of paints is directly proportional to Kubelka-Munk scattering coefficients, this is true only if the hiding powers are measured at the same ultimate brightness, since this affects the contrast ratio measurement.

There are therefore several reasonable explanations for the authors' observations regarding grade substitutions that failed to conform to my own experience, but no reason to suggest that the original hiding power equation is not at least as accurate, and definitely more convenient to use for a practical paint formulator, than the revised version. This is particularly true in light of the fact that the authors' observed deviations-even if we assume that their results were unaffected by the abovenoted departures from my original equation-occur only at effective PVC's far in excess of those to be found in practical paint systems.

By-and-large, practical paint formulations contain, or should contain, only that amount of titanium dioxide that will produce the desired level of hiding power at minimum total raw-material cost, which precludes the use of any effective PVC greatly in excess of 20 percent. The optimum level varies with the relative costs of pigment and binder, but not to any extent sufficient to cause the system to approach the authors' area of concern.

I realize that the primary goal of the reported research project was to find some means for adequately predicting the scattering produced by systems containing both titanium dioxide and the Rohm and Haas "opaque polymer." I question the authors" conclusion that this can be accomplished by additive methods, since my own experience has shown that the opacity of the polymer as a single "pigment" is inadequate, but increases dramatically with the addition of even relatively small amounts of titanium dioxide. This would suggest to me that the effect of the encapsulated air upon the average refractive index of the medium surrounding the titanium dioxide particles should be taken into account when calculating the term "R" in their modification of my equation.

FRED STIEG PigmenTech Consulting Jekyll Island, GA

\* \* \*

TO THE EDITOR:

As Mr. Stieg points out, his hiding model has stood the test of time very well. His basic hiding power equation has guided many formulators, including ourselves, to more efficient usage of TiO<sub>2</sub> in paints. It should be obvious from our paper that we drew heavily on his work. Mr. Stieg's insight that the loss of hiding power in TiO<sub>2</sub> based paints depends primarily on the pigment spacing has made the term "crowding" an integral part of the paint formulator's vocabulary. Without his efforts, we probably would not have attempted to model hiding directly, but would have continued to use experimental design methods like those described by Nyi (Aust. OCCA Proc. News, 19, No. 11, 4-13, 1982). With that approach, each formulation requires a large number of experiments to develop a predictive hiding model which works only for that formulation. By using a more general hiding model, such as Mr. Stieg's, we can reformulate paints with considerably more efficiency.

To answer Mr. Stieg's comments on our paper, we would first like to contrast our two approaches in very broad terms. Mr. Stieg observed that hiding power depended on the interparticle spacing of the TiO<sub>2</sub>. He developed an equation to describe how that spacing changes with the PVC of the TiO<sub>2</sub>. In order to calculate that spacing, he had to assume a specific packing fraction and found that the "rhombohedral" constant of 0.74 best fit his data. Other than his references to "refraction" and "diffraction," Mr. Stieg's model does not explain the physics of why hiding power depends on TiO<sub>2</sub> spacing, but it is reasonably successful in modeling that dependence for practical paints.

In contrast, our model is based on a more physical explanation of dependent scattering. In calculating scattering based on Mie theory, we found that light is scattered from a small scatterer in a volume which extends beyond the physical boundaries of the particle. At high concentration, these scattering volumes overlap, lowering the overall scattering efficiency of the scatterers. Because this model (equations 6 through 11 in our paper) was not very easy to apply for practical systems, we used the form of Mr. Stieg's equation to approximate the behavior predicted by our physical model for practical paint systems (equation 13c). In doing so, we found that the choice of the packing fraction was not so important as it had been in Mr. Stieg's work: in our model, the denominator under the cube root sign, d, contains a packing fraction multiplied by a constant. We also found that in practical systems, we could fit the data better by using d as a fitting parameter for a specific TiO<sub>2</sub> grade in a specific system.

We would like to respond further to Mr. Stieg's comments in three general areas: modelling Kubelka-Munk S, as opposed to hiding power; experimental parameterization of our model and its practical use; and our assumption of random dense packing of the TiO<sub>2</sub> particles.

S vs HIDING POWER: As Mr. Stieg points out, S and hiding power are different quantities: S is proportional to hiding power, but the constant of proportionality depends upon the absorption ("brightness") of the paint. We chose to model S, rather than hiding power, for both theoretical and practical reasons. First, the physical pic-



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ture behind the model is of light scattering, not a mixture of scattering and absorption; therefore, the model should be for light scattering (S), not a mixture of scattering and absorption (hiding power). Second, in practice, money spent on prime pigments like TiO<sub>2</sub> and opaque polymer is money spent on scattering, not hiding power (after all, a cost-effective way to increase hiding power is to tone the paint). "White hiding power' is basically scattering, and in our experience, S is a more precise measurement of scattering than is the contrast ratio.

We believe that S is a quantity with which most practical paint technologists are familiar. ASTM Method D 2805-70 for determining hiding power of paint films gives determination of S and the corresponding absorption parameter K as an alternative. Also, computer color matching systems, which are widely used, are based upon K and S.

PARAMETERIZATION OF THE MODEL AND ITS USE ON PRACTICAL PAINT SYSTEMS: Like Mr. Stieg, we derived parameters for our model from a large data base (replicated measurements on several hundred paints), developed over several years, which covers a wide variety of paint compositions. Our additive model for S of paints containing both TiO2 and Ropaque OP-42 was derived from this data base. The model fits the S values as accurately as they can be measured. The data show that the encapsulated air should not be included in calculating the average refractive index of the medium. Viewed in another way, however, OP-42 does enhance TiO2 scattering, since it allows the formulator to use TiO<sub>2</sub> at lower levels where it is a more effective scatterer, while maintaining a high scattering coefficient for the paint.

Unlike Mr. Stieg's finding with hiding power, we found that to fit S with the accuracy with which it can be determined, we had to derive K1 and d values for each TiO<sub>2</sub> grade. This difference may be due to the fact that S is a more precise measurement of scattering than is hiding power. Deriving the parameters needed to handle a variety of TiO2 grades required only a moderate amount of experimental work. The parameters needed for a given TiO<sub>2</sub> grade can be derived from S-measurements on a four-to-five paint TiO2 ladder, and the number of truly unique TiO2 grades used in coatings is limited. We felt that the modest investment of time to do the experiments was more than compensated for by the increased accuracy of the model predictions.

Effective TiO<sub>2</sub> PVC's in some practical paint systems could easily be in the range where the increased accuracy conferred by using grade-dependent model parameters will be important. For instance, a 55 PVC paint made with large extender and 20 PVC TiO<sub>2</sub> has an effective TiO<sub>2</sub> PVC of 31. In this formulation, a heavily coated TiO<sub>2</sub> (such as DuPont R-931) will give significantly higher S than a lightly coated TiO<sub>2</sub>, even if the two are used at the same "pure" concentration (see *Figure* 2 of our paper).

ORDERED ARRANGEMENT OF TIO2 PARTI-CLES: Before commenting upon possible arrangements for TiO<sub>2</sub> particles in a paint film, we wish to point out that, unlike Mr. Stieg's model, our work is relatively insensitive to assumptions about such arrangements. First, none of the parameters of our practical model (equation 13c) depend upon an assumed packing fraction. The "d" parameter is proportional to the packing fraction, not equal to it. Second, the physical picture behind the dependent scattering theory is insensitive to assumptions about the particle packing. Because dependent scattering is shown to be the consequence of the overlap of "scattering spheres" around adjacent particles, the phenomenon is not affected by the longrange characteristics of the particle arrangement. In comparing theory (equations 6-11, Figure 5) with experiment, a value for the packing fraction had to be assumed. Random dense packing gave the best agreement to our data, but other packing fractions, such as random and rhombohedral, gave fairly good agreement too.

Given the available evidence, a random packing arrangement of the TiO2 particles still seems the most physically reasonable assumption. The assumption of random dense packing does not imply a non-uniform distribution of particles: it simply implies that the particle arrangement has no long-range order. In recent investigations into long-range order in latex suspensions, the "acid tests" for such order have been (1) observation of such order in micrographs, and (2) iridescence, the consequence of diffraction of white light by the latex lattice. TiO<sub>2</sub> particles in paint films don't pass either test. If TiO2 particles in a paint film are in an ordered three-dimensional arrangement, some order should be evident in the two-dimensional micrographs: ordered three-dimensional arrangements collapse into ordered two-dimensional arrangements (consider the crystallographic space groups). We observed that paint films (as opposed to TiO2 dispersions) with 30-40 PVC TiO<sub>2</sub> appeared white, not iridescent. In any event, if dependent scattering is a consequence of (postulated) long-range ordering of TiO<sub>2</sub> in the paint film, then iridescence should be observed for TiO<sub>2</sub> PVC's as low as 10, where dependent scattering effects become observable.

We would like to thank Mr. Stieg for his interest in our paper, and the Federation for providing us this opportunity to respond to his comments.

SUSAN FITZWATER JOHN W. HOOK, III Rohm and Haas Co. Spring House, PA

## Coming Events

#### FEDERATION MEETINGS

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

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

#### 1986

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

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

#### SPECIAL SOCIETY MEETINGS

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

#### 1986

(Apr. 2-5)—Southern Society. Annual Meeting. DeSoto Hilton Hotel, Savannah, GA. (Ronald R. Brown, Union Chemicals Div., P.O. Box 26845, Charlotte, NC 28213).

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

(Apr. 29-May 1)—"Advances in Coatings Technology" Conference sponsored by the Cleveland Society for Coatings Technology. NASA, Lewis Research Center, Cleveland, OH. (Dr. Rosemary Loza, Standard Oil Co. (Ohio), 4440 Warrensville Center Rd., Cleveland, OH 44128).

(May 1-3)—Pacific Northwest Society, Annual Symposium. Marriott Hotel, Portland, OR. (Gerald A. McKnight, Rodda Paint Co., 6932 S.W. Macadam Ave., Portland, OR 97219).

#### 1987

(Feb. 23-25)—Western Coatings Societies' Symposium and Show, Monterey Convention Center, Monterey, CA. (Barry Adler, Royell, Inc., 1150 Hamilton Ct., Menlo Park, CA 94025).

#### OTHER ORGANIZATIONS

(Sept. 9-13)—"The Basic Composition of Coatings" Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, MO 65401).

(Sept. 16-19)—Fifth Finishing Conference and Exposition of the Association for Finishing Processes of the Society of Manufacturing Engineers. Cobo Hall, Detroit, MI. (Public Relations Dept., AFP/ SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121). (Sept. 21-24)—Canadian Paint and Coatings Assn. 73rd Annual Convention. Clevelands House, Minnett, Ont. (CPCA, 515 St. Catherine St. W., Montreal, Que., Canada H3B 1B4).

(Sept. 23-27)—"Paint Formulation" Introductory Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401).

(Sept. 24-26)—"Inspection of Coatings and Linings for Immersion Service." Seminar offered by KTA-Tator, Inc., Pittsburgh, PA. (Bill Corbett, KTA-Tator, Inc., 115 Technology Dr., Pittsburgh, PA 15275).

(Sept. 30-Oct. 2)—12th International Naval Stores Meeting. Westin Peachtree Plaza Hotel, Atlanta, GA. (Douglas E. Campbell, Executive Director, Pulp Chemicals Assn., 60 E. 42nd St., New York, NY).

(Oct. 8-11)—XVIth Congress of AFTPV (French Assn. of Paint and Varnish Technicians), Palais des Congres, Strasbourg, France. (AFTPV, 5, Rue Etex, Paris. 75018, France).

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

(Oct. 15-17)—ASTM Committee B-8 on Metallic and Inorganic Coatings. ASTM Headquarters, Philadelphia, PA. (Teri Carroll, ASTM, 1916 Race St., Philadelphia, PA 19103).

(Oct. 15-17)—"Maintenance Painting Practices." Seminar sponsored by KTA-Tator, Inc., Pittsburgh, PA. (Bill Corbett, KTA-Tator, Inc., 115 Technology Dr., Pittsburgh, PA 15275).

(Oct. 21-23)—Symposium on "New Chemistry for Product Diversification and Specialization in Coatings." Sponsored by Eastern Michigan University. Ann Arbor, MI. (Alan Green, Technology Services Center, Eastern Michigan University, 150 Sill Hall, Ypsilanti, MI 48197).

(Oct. 21-25)—"Physical Testing of Paints & Coatings" Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, ROla, MO 65401).

(Oct. 27-30)—Fire Retardant Chemicals Association Fall Conference. Pinehurst Hotel, Pinehurst, NC. (James D. Innes, FRCA, 851 New Holland Ave., P.O. Box 3535, Lancaster, PA 17604).

(Nov. 4-5)—"Painting Processes: Industrial Paint Application Technology" Short Course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Kent State University, Chemistry Dept., Kent, OH 44242).

(Nov. 4-6)—"Estimating for Painting Contractors and Maintenance Engineers" Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401).

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

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

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(Nov. 18-19)—Seminar on "Fine Particles and Fillers—Overlooked Opportunities." Sheridan Park Conference Center, Mississauga, Ont., Canada. (H.G. McAdie, Ontario Research Foundation, Sheridan Park Research Community, Mississauga, Ont., Canada, L5K 183).



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

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

(Sept. 15-17)—13th International Naval Stores Meeting. Waldorf-Astoria, New York, NY. (Douglas E. Campbell, Executive Director, Pulp Chemicals Assn., 60 E. 42nd St., New York, NY).

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## Humbug' from Hillman

Recent mail brought correspondence and the contribution below from Ray Pierrehumbert, whom many of our readers will remember as one of the industry's emulsion and latex paint experts. Ray retired last year and, like so many of us who really relish the thought of retirement, felt obliged to hang out a consultant's shingle (probably to satisfy conscience or a wife). Ray, although doing nicely, doesn't let his consultant assignments get in the way of enjoyment of the glorious Pocono Mts. and the local wit of his neighbors. Here follow some definitions developed by folks in the community.

ALPONIUM—(Chemical symbol: Ap) Initial blast of odor upon opening a can of dog food.

CABNICREP—A structural condition in which the closing of one kitchen cabinet causes another to open.

CARPERPETUATION—The act, when vacuuming, of running over a string or piece of lint at least a dozen times, reaching over and picking it up, examining it, then putting it back to give the vacuum one more chance.

FRUST—The small line of debris that refuses to be swept onto the dustpan and keeps backing a person across the room until he finally decides to give up and sweep it under the rug.

RETROCARBONIC—Any drink machine that dispenses the soda before the cup.

UFLUATION—The peculiar habit, when searching for a snack, of constantly returning to the refrigerator in hopes that something new will have materialized there.

And speaking of retirees and consultants, one of the stellar members of the crowd, Fred Stieg, wrote me not so long ago and said, in part—

Some additional evidence that "fame is fleeting" appeared in a recent issue of the *Journal* (I happened to page through it after reading your column)! I found the following item in the index of available literature published by the Golden Gate Society:

667 S Stieg, F.B.J. Partical Size as a Formulation Parameter JPT, 1967 (1968)

I have been able to figure out that the extraneous initial "J" resulted from my practice of adding "Jr." after my name while my father was still alive, but it is quite a bit more difficult to find any reason for changing "Particle" to "Partical," or "Formulating" to "Formulation"—and why two publication dates? As far as I could tell, my listing appeared to be the only one to be given special treatment!

Maybe someone out there is making a comment about my having retired to southern Georgia, where the art of proofreading never fully recovered after "The War Between the States," and strange headlines appear in our local paper like SEVENTH SEXTUPLET DIESI and DAR-IEN CELIBRATES BLESSING OF THE FLEAT.

From the April Del-Chem Bulletin, via Joe Vasta, we selected the following to brighten your today.

• A nudist colony is a place where men and women air their differences.

• *Diner:* "Look Jim, why are you always trying to impress me? I noticed that you spoke to that waiter in French. So, what good is it to know a few words in French?—Waiter, what did he tell you?"

Waiter: "Sir, he told me to give you the check."

 Never put off 'til tomorrow what you can put off for good.

Joe Vasta plucked this from a Scientific American article on garlic and onion chemistry.

"Garlic's virtues and faults are perhaps summarized by Sir John Harrington in "The Englishman's Doctor," written in 1609:

Garlic then have power to save from death

Bear with it though it maketh unsavory breath

And scorn not garlic like some that think

It only maketh men wink and drink and stink."

And from our files a number of quickies that merit your attention-

 Bath mat: a small, round, dry rug that wet children love to stand beside.

-The Lion

 Minister at funeral: "Don't think of our dearly beloved as gone forever, just think of him as having been recalled by his manufacturer."

-Metafax, quoted by Borrelle

- I like good honest engineering. I'm a nostalgia buff.
- Bee oh double ess is double ess oh bee spelled backwards.
- Ignorance and superstition always triumph over science and skill.
- They taught me how to delegate, but there isn't anyone to.

-Bob Ahlf

- A boss is a person who is late when you're early and early when you're late.
- Here, have a piece of candy. The ones with the teeth marks in them have hard centers.
- Fresh air is when you open the window yourself. A draft is when someone else opens it.

-National Safety Council via Maureen Lein

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

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