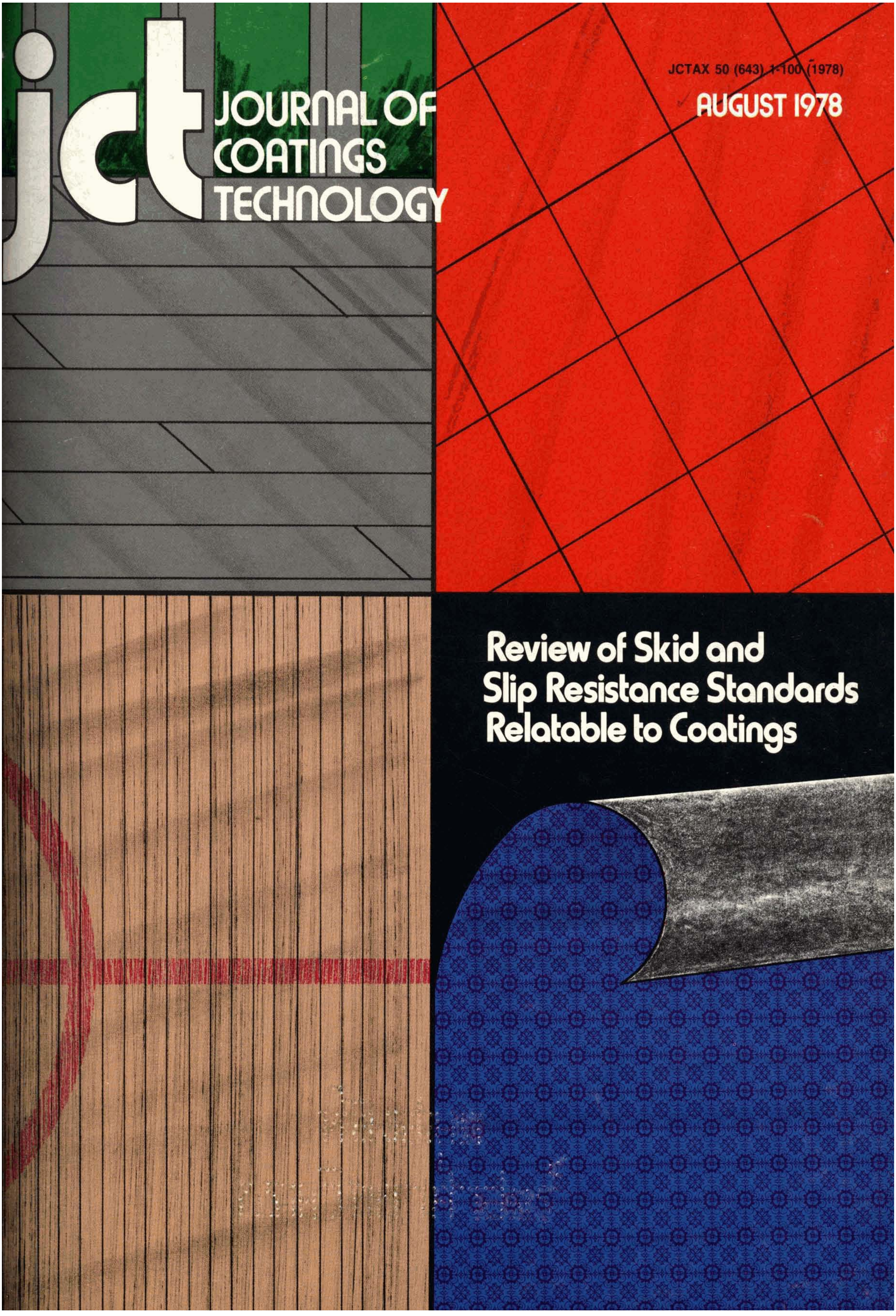
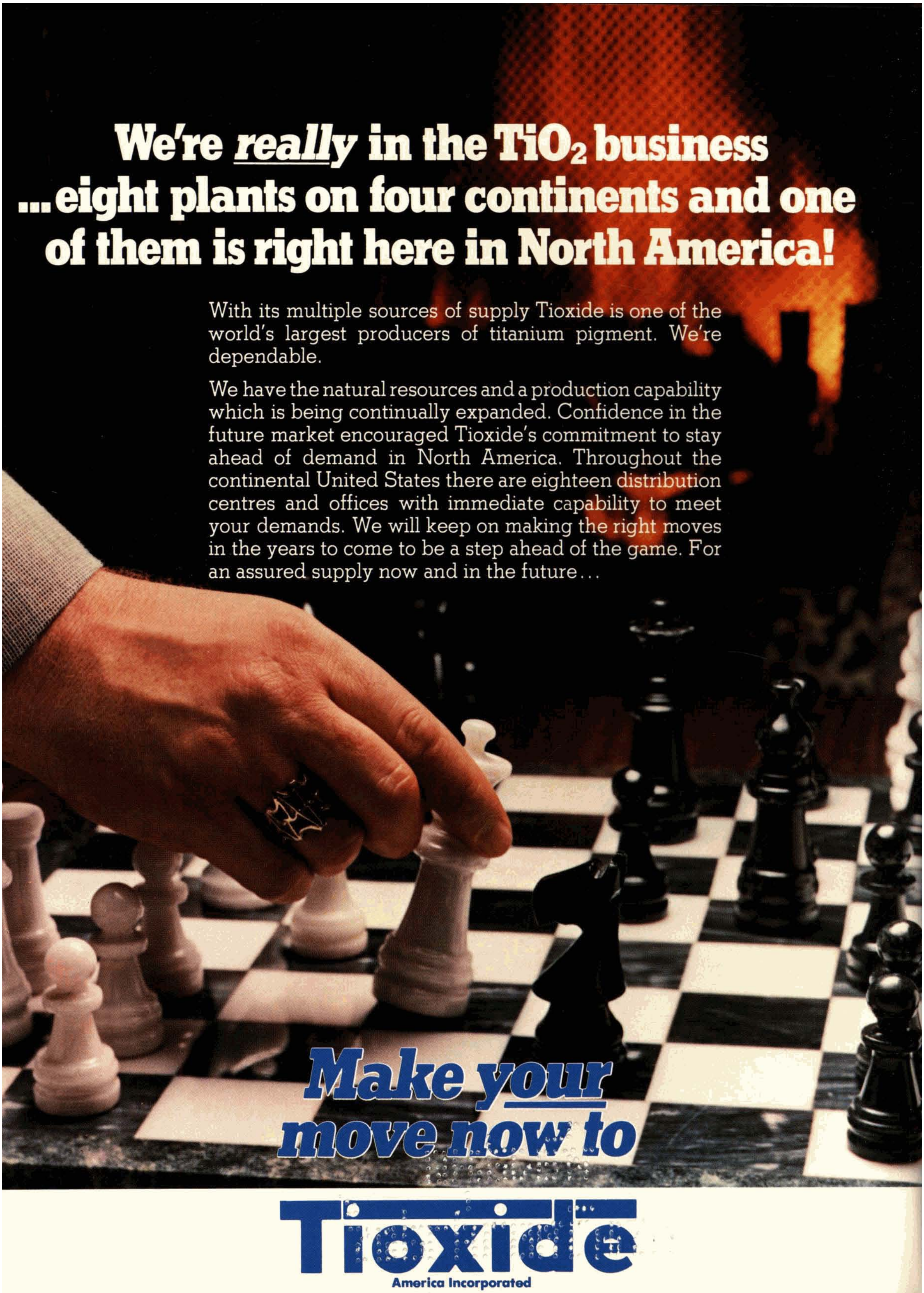


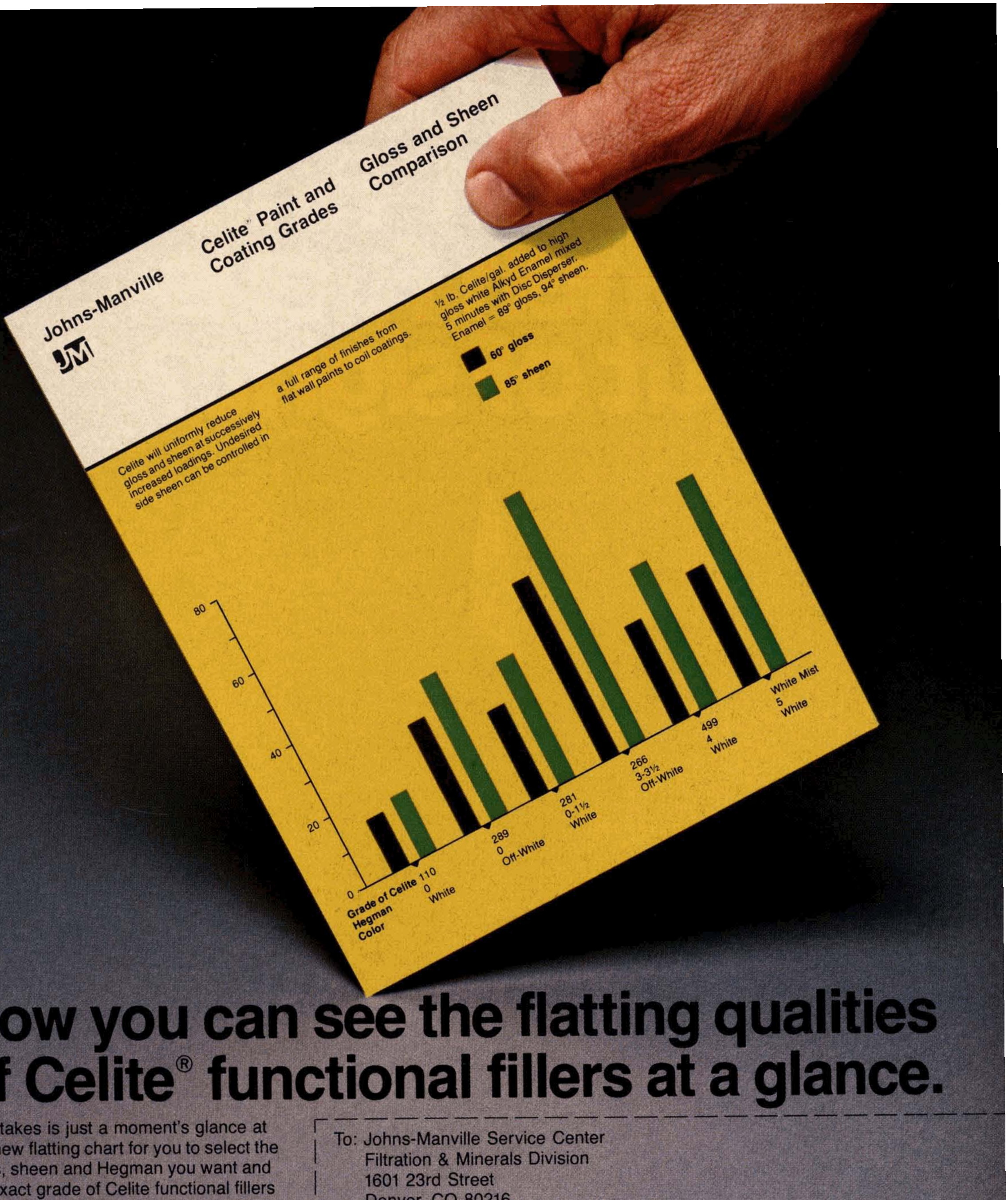
JOURNAL OF COATINGS TECHNOLOGY

JCTAX 50 (643) 1-100 (1978)

AUGUST 1978

Review of Skid and Slip Resistance Standards Relatable to Coatings





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AUGUST 1978

JOURNAL OF COATINGS TECHNOLOGY

Volume 50 Number 643

Features

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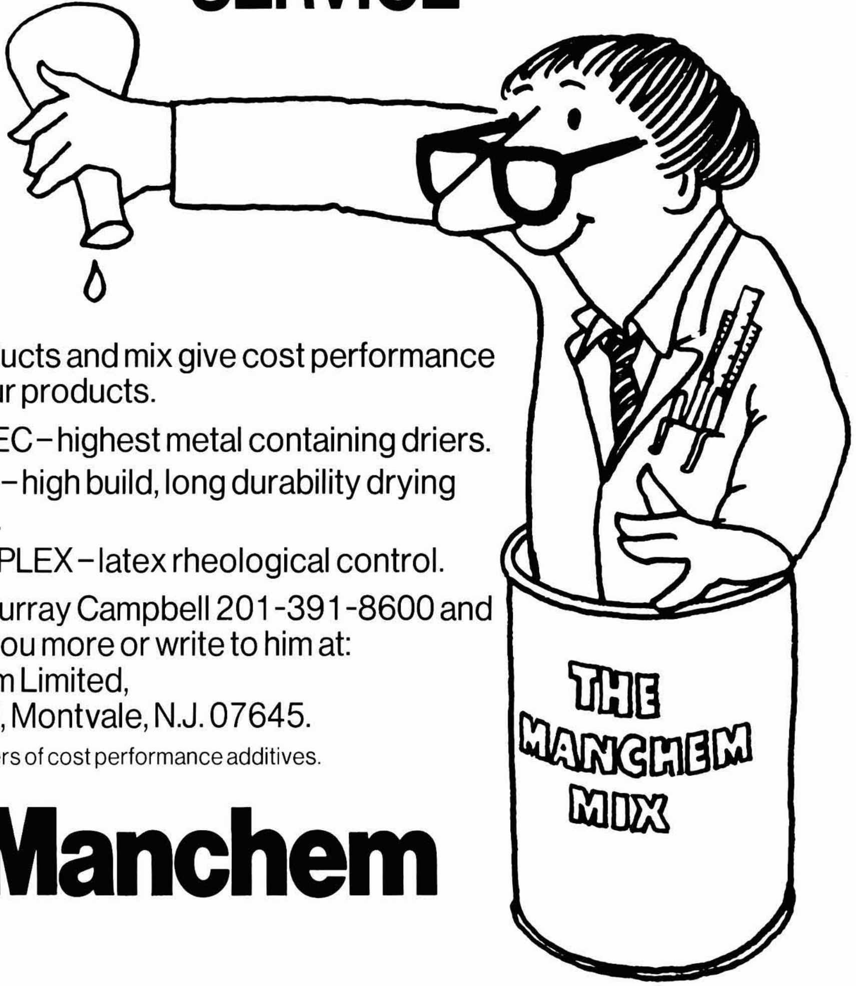
ond class postage paid at Philadel- Pa. and at additional mailing of-

bscriptions U.S. and Canada—1 $20; 2 years $37; 3 years, $52. pe (Air Mail)—1 year, $30; 2 years; 3 years, $72. Other Countries—1 $26; 2 years, $48; 3 years, $68.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Annual dues for members of the Federation of Societies for Coatings Technology, $15.00, includes a subscription to this publication. Membership in the Federation is obtained through prior affiliation with, and payment of dues to, one of its 26 Constituent Societies. Non-member subscription rates are:

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When available, single copies of back issues of the JOURNAL OF COATINGS TECHNOLOGY are priced as follows: $3.00 each for current calendar year issues; $4.00 each for all other issues.

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A Guide for Authors is published in each January issue.

The JOURNAL OF COATINGS TECHNOLOGY is available on microfilm from University Microfilms, a Xerox Co., Ann Arbor, Mich. 48106.

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Comment

The FATIPEC Congress

In June, President John Oates and the writer attended the XIVth Congress of FATIPEC, a European coatings industry organization whose objectives parallel those of the Federation.

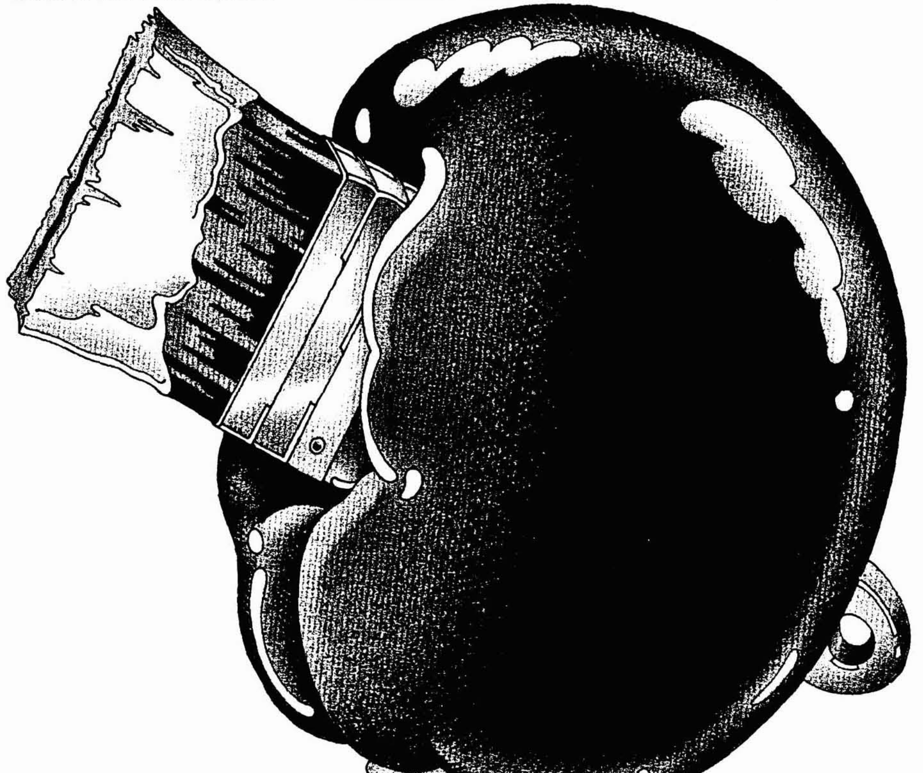
The Congress was held in the beautiful city of Budapest which straddles the Danube River in Hungary. In keeping with the FATIPEC custom, the Congress was a full five days of daytime technical papers and evening social activities. (See the story elsewhere in this issue).

Coatings industry scientific personnel from 23 countries attended, attesting to the well-known fact that the search for knowledge knows no boundaries. If you'll check the titles of the papers, you'll find the gamut of subjects that could also be presented at coatings conventions in the U.S., Canada, and United Kingdom, Scandinavia, Japan, or anywhere.

One of the encouraging events of the Congress was the luncheon meeting of executives of four paint industry organizations: FATIPEC, the Federation, the Oil and Colour Chemists' Association, and the Scandinavian Federation of Paint and Varnish Technologists. A friendly spirit prevailed and all recognized the need to maintain close cooperation and to communicate news of key developments and events to each other.

No matter how big the world and dissimilar its people may appear to be, the interests and objectives are pretty much the same.

As a result, the world is smaller than we realize and its inhabitants are not so different after all. — FJB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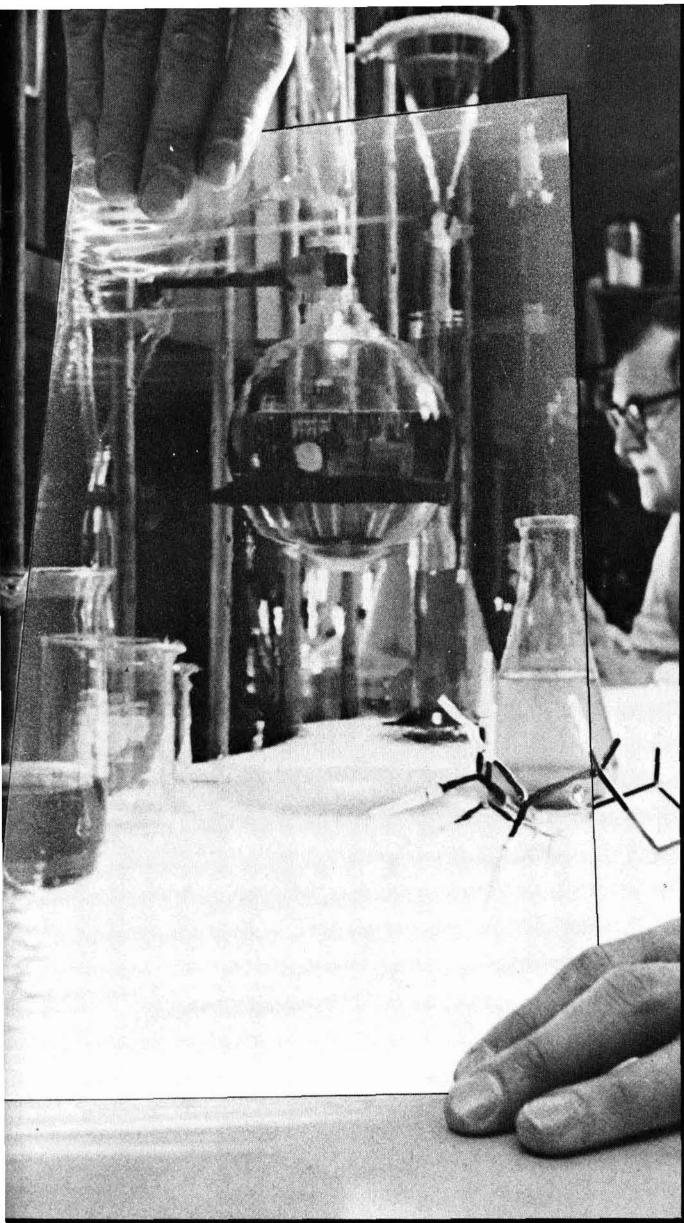

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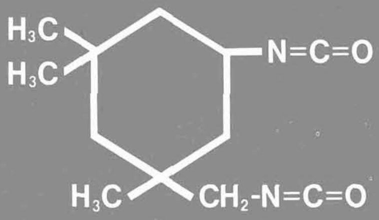

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Abstracts of Papers in This Issue

REVIEW OF SKID AND SLIP RESISTANCE STANDARDS RELATABLE TO COATINGS—P.R. Guevin

Journal of Coatings Technology, 50, No. 643, 33 (Aug. 1978)

Slips and falls result in unnecessary deaths and disabling injuries each year. In many cases, an organic polymeric surface is involved in the accident. ASTM has at least eight committees working on methods of measuring the coefficient of friction and establishing numerical standards. The National Bureau of Standards and Liberty Mutual Insurance Company are studying human perambulation in an attempt to develop better equipment to measure the coefficient of friction and are attempting to establish standards. The microstructure of organic coatings is shown to have a definite influence on the coefficient of friction of coatings, sometimes in unexpected ways.

APPROXIMATE RHEOLOGICAL CHARACTERIZATION OF CASSON FLUIDS: TEMPLATE METHOD FOR THE BROOKFIELD SYNCHRO-LECTRIC VISCOMETERS—M.R. Rosen and W.W. Foster

Journal of Coatings Technology, 50, No. 643, 39 (Aug. 1978)

The non-Newtonian characteristics of a wide variety of industrial products can be characterized by the empirical Casson flow equation. This paper presents a method for obtaining the two Casson parameters using computer generated graphs specifically designed for use with all models of the Brookfield Synchro-Lectric Viscometer. When used with cylindrical spindles and a special template, the Casson parameters are easily obtained. These empirical analogs for Casson viscosity and yield stress are often quite useful for describing and controlling product behavior.

β-HYDROXYALKYLAMIDES, LOW POLLUTING CROSSLINKERS FOR CARBOXYL CONTAINING POLYMERS—J. Lomax and G. Swift

Journal of Coatings Technology, 50, No. 643, 49 (Aug. 1978)

β-Hydroxyalkylamide crosslinkers make possible the preparation of water-borne thermosetting coatings which evolve no noxious reaction by-products and which have energy saving potential relative to other water-borne baking enamels because they require only very low levels of organic cosolvent in formulation.

β-Hydroxyalkylamides crosslink the carboxyl groups of water-soluble polymers. Synthesis and characterization of several of these crosslinkers was completed. One of them, bis [N,N-di (β-hydroxypropyl)] a dipamide, has been extensively tested as a crosslinker for acrylic colloidal dispersions in formulations for industrial baking applications. These easily formulated enamels provide a cure response comparable to conventional amino-resin crosslinked water-borne coatings and film properties useful for appliance coatings and general product finishing applications.

STYLUS MEASUREMENT OF THE MICROGEOMETRY OF A COATED SURFACE—M.J. King and T.R. Thomas

Journal of Coating Technology, 50, No. 643, 56 (Aug. 1978)

The feasibility of using a stylus instrument for looking at a section through a coated surface is discussed. Experimental measurements of the deformation of paint by a stylus are presented, and it is shown that deformation has no significant effect on roughness measurements. Changes in the statistical microgeometry of a profile after successive coats of paint are examined by relocation profilometry. Most of the changes are found to occur after the first coat: the surface becomes smoother, its slopes become gentler, and its peaks more rounded. However, the shortest surface wavelengths are found to be relatively unaffected by painting.

COMPARISON OF ELECTROSTATIC SPRAY-APPLIED POWDER COATINGS—J.P. Blackwell, D.G. Brady, and H.W. Hill, Jr.

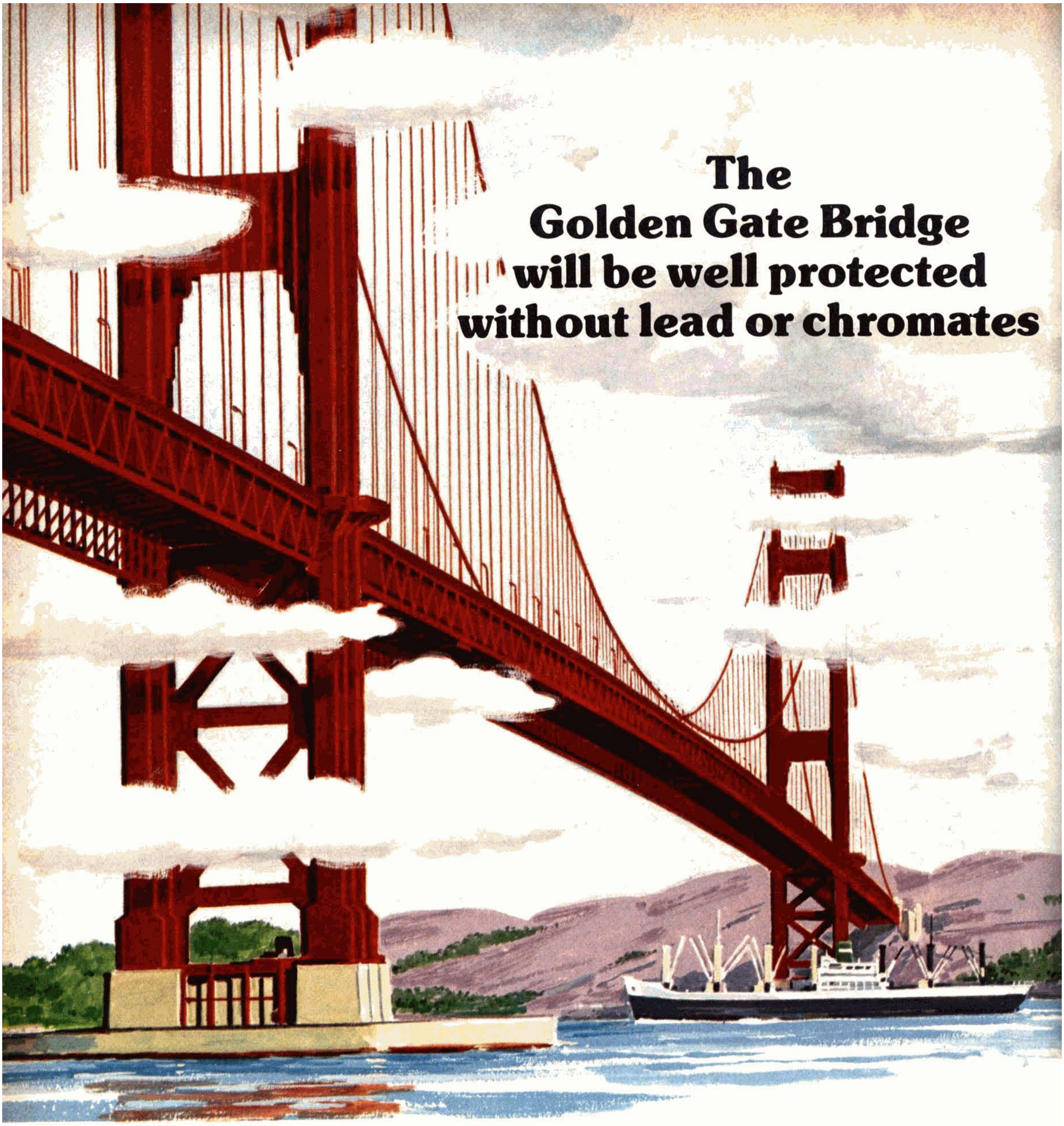
Journal of Coatings Technology, 50, No. 643, 62 (Aug. 1978)

A comparison of the thermal and chemical resistance of several electrostatically-applied coatings including phenolic, phenolic-epoxy, polyester, epoxies, polyphenylene sulfide (PPS), and ethylene-chlorotrifluoroethylene (E-CTFE) resins was made, along with two coatings, polytetrafluoroethylene (PTFE) and polyimide, applied from liquids.

Coatings were evaluated for thermal stability by monitoring property changes during aging at elevated temperatures. Chemical resistance was determined by exposure to 21 different environments for 24 hr at 90°C.

At 100 and 150°C all coatings, except the phenolic and phenolic-epoxy, survived with little deterioration evident after 5,000 hr in air. At 200°C, only the polytetrafluoroethylene coating remained unchanged after 2,700 hr. Polyimide, PPS, and E-CTFE survived 2,500 hr, while others deteriorated in 1,000 hr or less.

Outstanding chemical resistance was shown by PTFE, PPS, and E-CTFE coatings. Other coatings suffered some attack under test conditions.



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Abstracts of Papers in This Issue (Continued)

SURVEY OF THE APPLICATIONS, PROPERTIES, AND TECHNOLOGY OF CROSSLINKING EMULSIONS—J.R. Grawe and B.G. Bufkin

Journal of Coatings Technology, 50, No. 643, 67 (Aug. 1978)

This review summarizes most of the principal concepts which have been employed over the last ten years to develop thermosetting or crosslinking emulsions. The conceptual elements which have formed the foundation for this new generation of coatings systems are discussed in terms of the science, technology, properties, and applications of the emulsion systems which have been reported in the literature. This survey presents the advantages and limitations afforded by each of the functional monomers or polymer classes which have been investigated for the development of crosslinking emulsions. Part I of this review presented a state of the art introduction into crosslinking emulsions and discussed the development of conventional emulsions which produce homogeneously crosslinked coatings through the utilization of N-methylolacrylamide. *(See June 1978 JCT—Ed.)* Part II extends the discussion of conventional emulsions which produce homogeneously crosslinked coatings to include systems containing N-(alkoxymethyl)acrylamide, hydroxymethylated diacetone acrylamide, allyl N-methylolcarbamate, N-formyl-N′-acryloylmethylenediamine, vinyl and acryloxysilanes, and autooxidizable monomers. This six-part review, all of which will be published in the JOURNAL OF COATINGS TECHNOLOGY, serves as an introduction to a series of publications from these laboratories describing the research and development of crosslinking emulsions which are new and novel, or which have not been adequately described in the present literature.

Papers to be Published in Future Issues

"Zinc Oxide/Nonmercurial Biocide Combinations in Alkyd House Paints"—S. Mark, of Gulf and Western Industries.

"From Treating Solutions to Filler Surface and Beyond: The Life History of a Silane Coupling Agent"—M.R. Rosen, of Union Carbide Corp.

"Practical Technique for the CPVC Determination of Titanium Dioxide Containing Latex Paint Systems"—K.K. Hesler, of DeSoto, Inc.

"New Test Method for Resistance to Water Immersion: Some Results Obtained with Two-Part Polysulfide Sealant"—K.K. Karpati, of National Research Council of Canada.

"Survey of the Applications, Properties, and Technology of Crosslinking Emulsions: Part III"—B.G. Bufkin and J.R. Grawe, of the University of Southern Mississippi.

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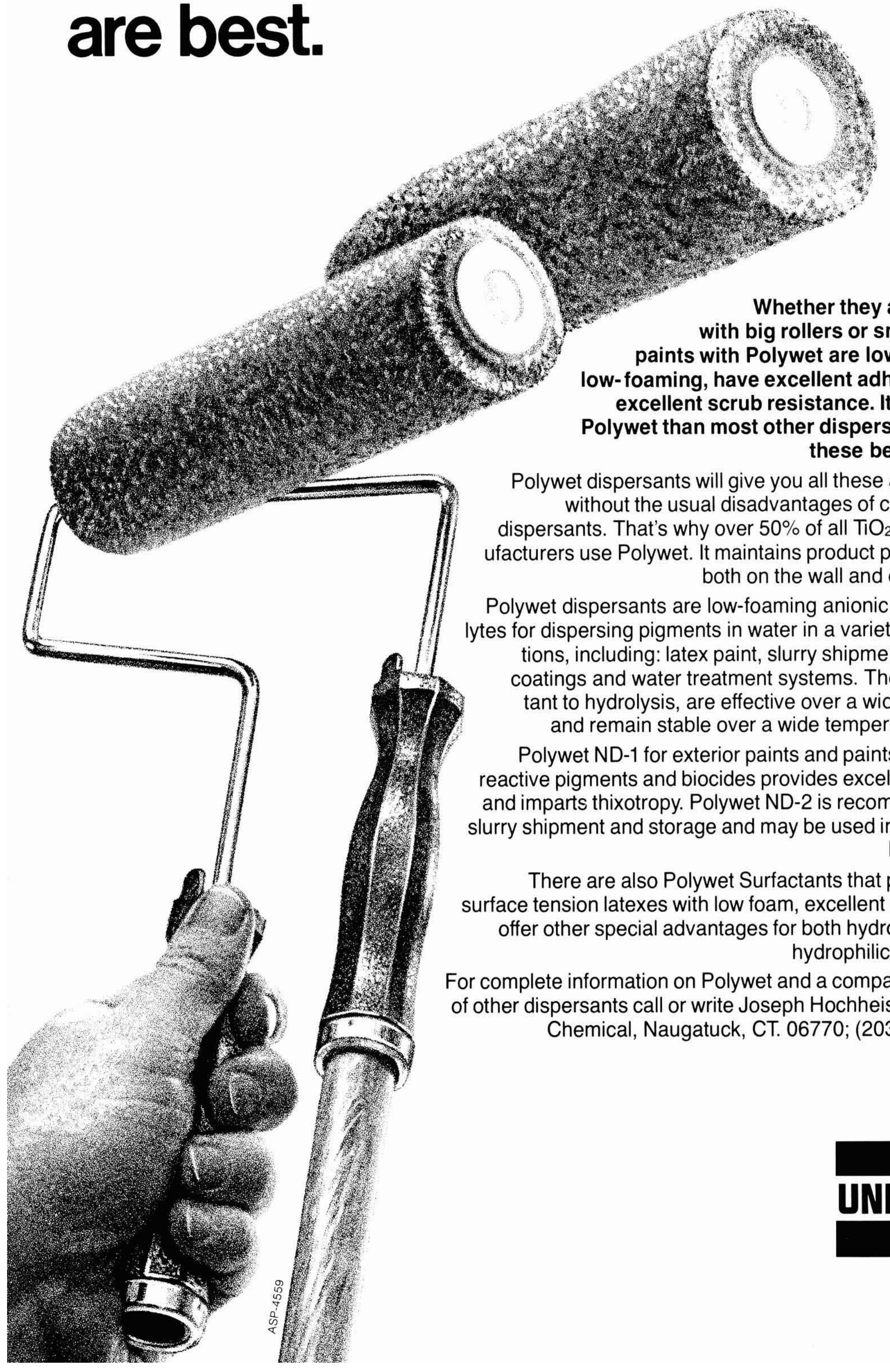

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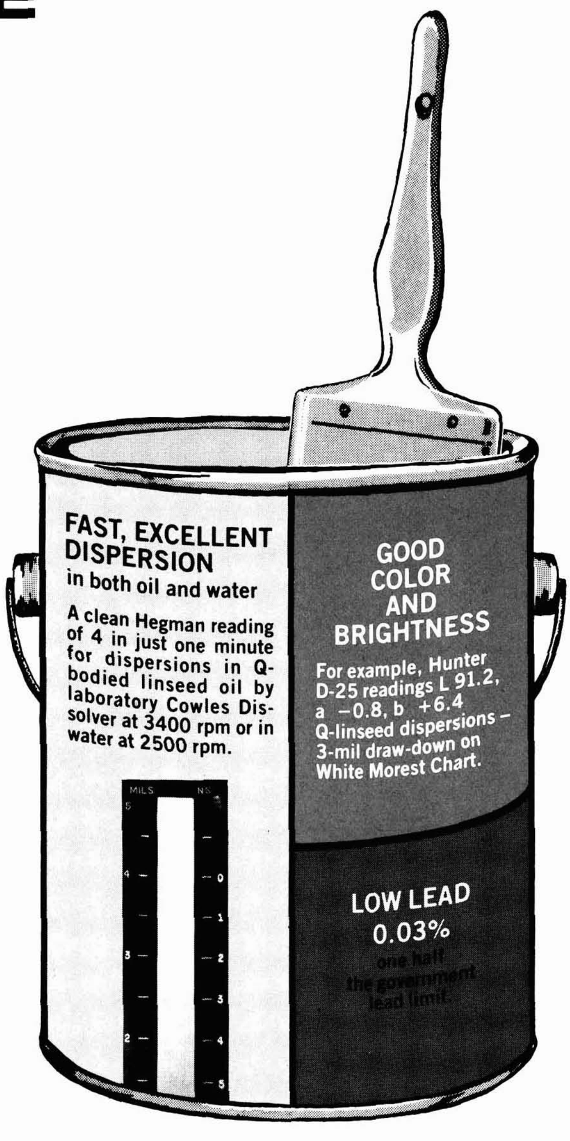
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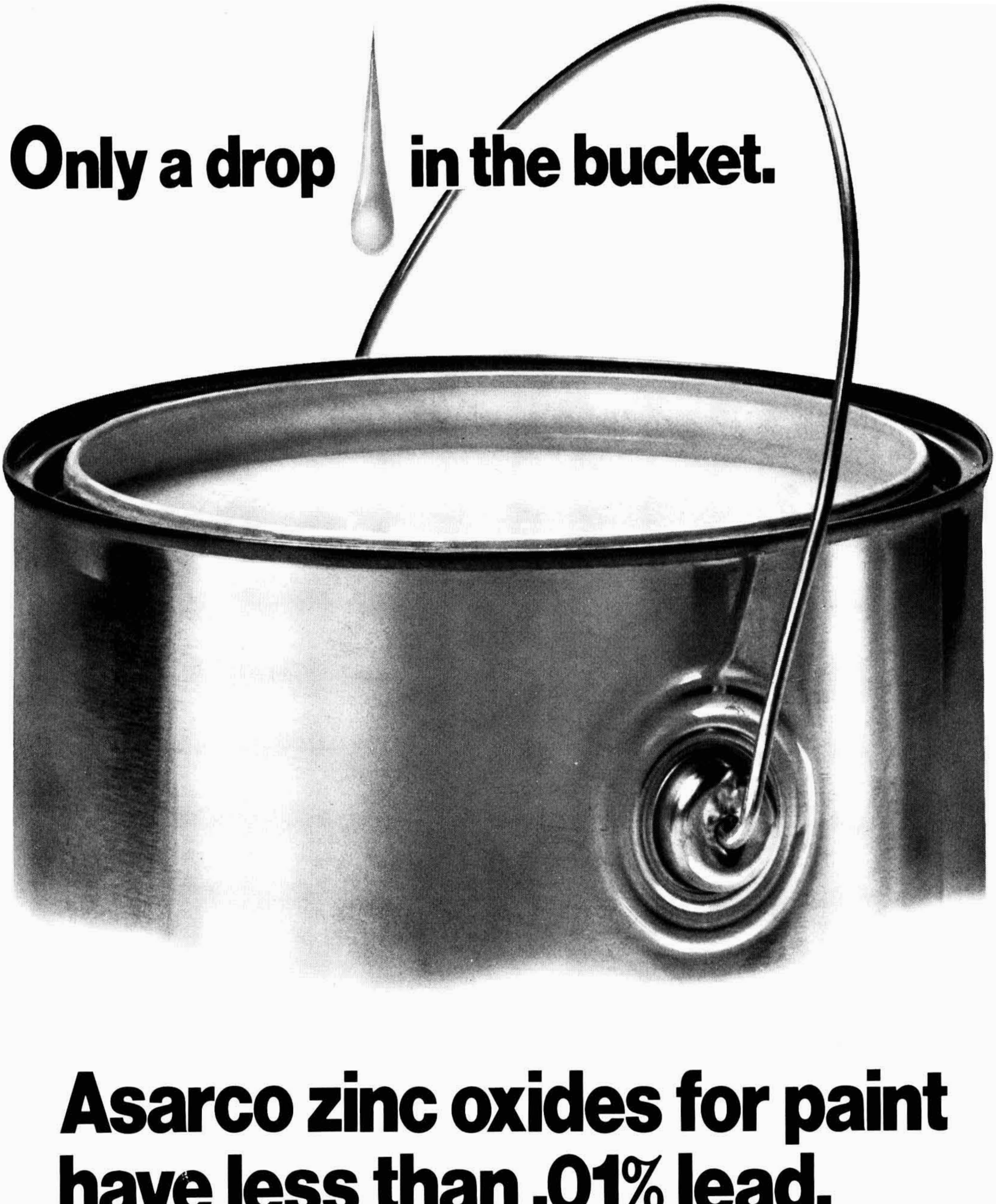
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Preliminary Program

WEDNESDAY, NOVEMBER 1

OPENING SESSION
(10:30—11:00)

Fifty-sixth Annual Meeting of the Federation of Societies for Coatings Technology opened by President John J. Oates.

Invocation and In Memoriam

Welcome: Walter J. Krason, Jr., President of Chicago Society for Coatings Technology

Thomas Nevins, Chairman of the Host Committee

Fred Schwab, Chairman of the Program Committee

Colin D. Penny, Chairman of the Paint Industries' Show Committee

Introduction of Federation Officers

Introduction of Distinguished Guests

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

TECHNOLOGY, ETHICS, AND SOCIETY — Mario G. Salvadori, of Weidlinger Associates, New York, N.Y.

GENERAL INTEREST SESSION
(2:00—5:00)

CEMENTITIOUS COATINGS — Joseph Lavelle, of Rohm and Haas Co., Spring House, Pa.

APS — A NEW COATINGS SYSTEM — Edward G. Bozzi, of CIBA-GEIGY Corp., Ardsley, N.Y.

NEW CONCEPTS IN THE FORMULATION OF GLOSS LATEX PAINTS — John Bax, of Scott Bader Co. Limited, Northamptonshire, England. (Presented on behalf of OCCA: Oil & Colour Chemists' Association.)

LATEX-BASED UNIVERSAL PRIMERS — Andrew Mercurio, of Rohm and Haas Co., Spring House, Pa.

NEW COATINGS FROM MONOMERS THAT CURE WITHOUT SHRINKING — William J. Bailey, Department of Chemistry, University of Maryland, College Park, Md.

POLYESTERAMIDE MODIFIED WATER-DISPERSIBLE RESINS — Wilma J. Schneider, of Northern Regional Research Center, U.S. Department of Agriculture, Peoria, Ill.

THEME PAPERS
(2:00—5:00)

SOLAR COLLECTOR PERFORMANCE: A DEPENDENCE UPON COATINGS — Richard E. Wolf, of DeSoto, Inc., Des Plaines, Ill.

TODAY'S COATINGS AND OUR NEEDS IN THE FUTURE — Panel Discussion by members of Painting and Decorating Contractors of America.

OPPORTUNITIES IN AIR FORCE COATINGS RESEARCH AND DEVELOPMENT — Daniel E. Prince, of Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

COATINGS AS VAPOR BARRIERS IN HOUSE INSULATION AND THEIR EFFECT ON HEAT TRANSMISSION — Douglas M. Burch, of National Bureau of Standards, Washington, D.C.

STATE-OF-THE-ART OF FIRE RETARDANT COATINGS — Joseph R. Klementowicz, of New Jersey Port Authority, Jersey City, N.J.

COATINGS OPPORTUNITIES — AN OVERVIEW — Sid Lauren, of Coatings Research Group, Inc., Cleveland, Ohio.

Program at a Glance

Tuesday, Oct. 31	**Registration (9:00 am to 5:00 pm)**
Wednesday, Nov. 1	**Registration (8:00 am to 5:00 pm)** **Opening Session (10:30 am)** **Keynote Address (11:00 am)** **Technical Sessions (2:00 to 5:00 pm)** **Paint Industries' Show (12:30 to 6:00 pm)**
Thursday, Nov. 2	**Registration (8:00 am to 5:00 pm)** **Technical Sessions (9:00 am to Noon) (2:00 to 5:30 pm)** **Paint Industries' Show (10:00 am to 6:00 pm)**
Friday, Nov. 3	**Registration (8:00 am to 3:30 pm)** **Technical Sessions (9:00 to 10:30)** **Mattiello Lecture (10:30)** **Technical Session (2:00 to 4:00 pm)** **Annual Business Meeting (4:00 pm)** **Paint Industries' Show (10:00 am to 4:00 pm)**

Awards Luncheon will be held at 11:45 am on Friday, Nov. 3, at the Conrad Hilton Hotel. Note: There will not be a dinner/dance this year.

Spouses' activities will begin with a Wine Tasting Party on Wednesday afternoon, Nov. 1, at the Conrad Hilton. Registration for Spouses' program will begin on Tuesday.

THURSDAY, NOVEMBER 2

PAINT RESEARCH INSTITUTE SEMINAR
(9:00—12:00)

SOLUBILITY — E.B. Bagley, of Northern Regional Research Center, U.S. Department of Agriculture, Peoria, Ill.

RHEOLOGY — Raymond R. Myers, Research Director of the Paint Research Institute and Chairman of the Chemistry Department, Kent State University, Kent, Ohio

COLOR AND HIDING — Fred W. Billmeyer, Jr., of Rensselaer Polytechnic Institute, Troy, N.Y.

CORROSION CONTROL — Dean M. Berger, of Gilbert Commonwealth Companies, Reading, Pa.

MILDEW DEFACEMENT — Donald J. Siehr, of Department of Chemistry, University of Missouri—Rolla, Rolla, Mo.

INDUSTRIAL HYGIENE PAPER
(9:00—9:30)

Presentation by F.S. Cooper, of DeSoto, Inc., Des Plaines, Ill.

GENERAL INTEREST SESSION
(9:30-10:30)

FORECASTING AVAILABILITY AND PRICE OF COATINGS RAW MATERIALS — Robert A. Bergfeld, of The Muralo Co., Inc., Bayonne, N.J.

HOW TO TESTIFY — Louis A. Lehr, Jr., of Arnstein, Gluck, Weitzenfeld & Minow, Chicago, Ill.

MANUFACTURING COMMITTEE SEMINAR
(10:30—12:00)

PANEL DISCUSSION ON DISPOSAL OF WATER-THINNED SOLIDS IN PAINT MANUFACTURE
Moderator — Gabriel Malkin, Coatings Consultant, Westfield, N.J.

Douglas Andrews, of Andrews Engineering Co., Springfield, Ill.

Walter W. Kovalick, Jr., of Office of Solid Waste, Environmental Protection Agency, Washington, D.C.

INFORMATION MANAGEMENT
(10:00—12:00)

INFORMATION RETRIEVAL FROM THE MANAGEMENT PERSPECTIVE — Howard L. Gerhart, of Carnegie-Mellon University, Pittsburgh, Pa.

TECHNICAL INFORMATION SOURCES FOR A SMALL COMPANY — Helen Skowronska, of Sherwin-Williams Co., Cleveland, Ohio

SOPHISTICATED RESOURCES FOR INFORMATION RETRIEVAL — Jocelyn Stephens, of DeSoto, Inc., Des Plaines, Ill.

INFORMATION — VITAL PAINT RAW MATERIAL — D. Dasgupta, of Paint Research Association, Middlesex, England

SOCIETY PAPERS
(2:00—5:30)

AN INFRARED SPECTROSCOPY ATLAS FOR THE COATINGS INDUSTRY — Chicago Society for Coatings Technology

STUDY OF ORGANIC TITANATES AS ADHESION PROMOTERS — Cleveland Society for Coatings Technology

DO WE NEED DEIONIZED WATER FOR AMINE DISPERSED VEHICLE SYSTEMS? — Detroit Society for Coatings Technology

COMPARISON OF METHODS TO DETERMINE WATER CONTENT OF EMULSION PAINTS — Golden Gate Society for Coatings Technology

FACTORS INFLUENCING DRY PROPERTIES OF A WATER SOLUBLE ALKYD — Kansas City Society for Coatings Technology

USE OF AIR PADS AS PALLET TRUCKS IN MOVING MATERIALS IN A PAINT PLANT — Toronto Society for Coatings Technology

CAUSTIC CLEANING APPARATUS FOR USE IN SMALL PAINT PLANTS — Toronto Society for Coatings Technology

ROON PAPERS
(2:00—4:30)

PHYSICAL MODEL FOR MUDCRACKING IN LATEX PAINT FILMS — Gordon Bierwagen, of The Sherwin-Williams Co., Chicago, Ill.

RHEOLOGICAL PROPERTIES OF STYRENE ACRYLIC POLYMERS — H.P. Schreiber and G. Thibault, of Ecole Polytechnique, Montreal, Quebec, Canada

SINGLE-GRADE RUTILE TiO_2 CONCEPT FOR INTERIOR LATEX TRADE SALES SYSTEMS — Calvin C. Tatman, of Glidden Pigments, Div. of SCM Corp.

EFFECT OF HUMIDITY AND OTHER AMBIENT CONDITIONS ON EVAPORATION OF TERNARY AQUEOUS SOLVENT BLENDS — Albert L. Rocklin, of Shell Development Co., Houston, Texas

CATHODE REACTIONS AND METAL DISSOLUTION IN CATIONIC ELECTRODEPOSITION — D.G. Anderson, E.J. Murphy, and John Tucci III, of DeSoto, Inc., Des Plaines, Ill.

FRIDAY, NOVEMBER 3

FEDERATION EDUCATIONAL PROGRAM
(9:00—10:00)

Papers will be presented by students from University of Southern Mississippi and North Dakota State University.

OVERSEAS PAPER
(10:00—10:30)

PART OF DIFFUSION IN THE ELECTROCHEMICAL PASSIVATION OF A PAINTED METAL — Dr. Marcel Piens, of Laboratoire de la Profession, Limelette, Belgium. (Presented on behalf of FATIPEC: Federation of Associations of Technicians in the Paint, Varnish, Lacquer, and Printing Ink Industries of Continental Europe.)

GENERAL INTEREST SESSION
(9:00—10:30)

AN UPDATE OF ANALYTICAL TECHNIQUES — Richard M. Holsworth, of Glidden-Durkee Div., SCM Corp., Strongsville, Ohio

FOAM RELATED ASPECTS OF WATER-REDUCIBLE ALKYDS — Ada C. Nielsen, of Nalco Chemical Co., Oak Brook, Ill.

ROLE OF SCANDINAVIAN INSTITUTE'S RESEARCH IN SEEKING NEW COATINGS OPPORTUNITIES — Charles Hansen, of Scandinavian Paint and Printing Ink Research Institute, Copenhagen, Denmark. (Presented on behalf of SLF: Scandinavian Federation of Paint and Varnish Technologists.)

MATTIELLO LECTURE
(10:30—11:45)

EFFECT OF REACTION PATHWAY ON EMULSION POLYMER STRUCTURE — Dr. Kenneth L. Hoy, Corporate Research Fellow, Union Carbide Corp., South Charleston, W. Va.

AWARDS LUNCHEON
11:45 am—Conrad Hilton Hotel

COPING WITH CHANGING AIR POLLUTION REGULATIONS
(2:00—4:00)

Moderator — Roy W. Tess, of Shell Chemical Co., Houston, Texas

CURRENT DEVELOPMENTS IN LEGISLATION AND REGULATIONS — Raymond Connor, of National Paint and Coatings Association, Washington, D.C.

CHOOSING COATINGS TO MEET REGULATIONS — George H. Wilhelm, of Ashland Chemical Co., Columbus, Ohio

A PAINT MANUFACTURER RESPONDS TO REGULATIONS — Neil H. Frick, of PPG Industries, Inc., Pittsburgh, Pa.

AN INDUSTRIAL PAINT USER RESPONDS TO REGULATIONS — George O. Payne, Continental Can Co., Chicago, Ill.

ANNUAL BUSINESS MEETING
(4:00—5:00)

Annual Business Meeting of the Federation.

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

Installation of Officers, 1978-79.

City of Chicago will again host Federation Annual Meeting and Paint Industries' Show. Conrad Hilton Hotel will be site of the 1978 event, November 1-3. Shown here is view of Chicago River, looking toward the near North Side; Marina Towers apartment complex is in foreground.

OTHER CONVENTION INFORMATION

PAINT INDUSTRIES' SHOW

The 43rd Annual Paint Industries' Show will run concurrently with the Annual Meeting in the Conrad Hilton. The only national exposition of materials and equipment used in the formulation, testing, and manufacture of coatings, the Show will be open from 12:30 to 6:00 p.m. on Wednesday, November 1; 10:00 am to 6:00 pm on Thursday, November 2; and 10:00 am to 4:00 pm on Friday, November 3.

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

REGISTRATION

Registration fees for the Annual Meeting and Paint Show are on a member and non-member basis:

	Member	Non-Member
Advance*	$35	$50
In Chicago	$40	$55
One-Day	$20	$30

*Special for retired members only: Advance registration will be $15.

Registration form is included in this issue (see pages 27 and 28) and has also been mailed to all members.

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

LUNCHEON

An Awards Luncheon will be held on Friday, November 3, at the Conrad Hilton.

Presentations will be made to recipients of the George Baugh Heckel Award (outstanding individual who has contributed to the advancement of the Federation) and the Flynn Awards (firms judged to have the best exhibit booths in the 1978 Paint Industries' Show).

Mark Russell, nationally-known political satirist who appeared at the Federation Annual Meeting banquet in 1976, will be the guest speaker at the luncheon.

The luncheon, which will be open to all registrants, is being held in place of the traditional banquet this year. Tickets are $10 each. Spouses' registration includes the price of the luncheon ticket.

SPOUSES' PROGRAM

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

A get-acquainted Wine Tasting Party is scheduled for Wednesday afternoon.

On Thursday, a continental breakfast will precede an all-day tour of the city, which will include Chicago landmarks, luncheon at the Ritz-Carlton Hotel, fashion show, and a shopping spree at the Water Tower Place.

Continental breakfast will be available again on Friday morning, followed by the Awards luncheon.

Registration fees for the Spouses' activities are $20 in advance and $25 on-site.

ROOM RESERVATIONS

All requests for rooms and suites must be on the official housing form and sent to the Conrad Hilton Hotel. These forms have been mailed to Federation members and additional copies are available from Federation headquarters.

NPCA MEETS SAME WEEK

The National Paint and Coatings Association will hold its Annual Meeting on October 30, 31, and November 1 at the Palmer House in Chicago.

NPCA registration badges will be honored for admission to the Federation Annual Meeting and Paint Show on Wednesday, November 1.

COUNCIL MEETING

The Fall Council Meeting of the Federation will be held Tuesday, October 31, at the Conrad Hilton Hotel.

SPEAKERS' BREAKFASTS

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

PUBLICATION OF PAPERS

The Journal of Coatings Technology has prior rights to the publication of all papers presented at the Annual Meeting.

PROGRAM STEERING COMMITTEE

Chairman — Fred Schwab of Coatings Research Group, Inc., Cleveland, Ohio; *Vice-Chairman* — Morris Coffino, of D.H. Litter Co., Inc., New York; Harvey Beeferman, of DeSoto Coatings Ltd., Toronto, Canada; Gordon Bierwagen, of Sherwin-Williams Co., Chicago, Ill.; Hugh Lowrey, of Indurall Coatings, Inc., Birmingham, Ala.; and Thomas J. Miranda, of Whirlpool Corp., Benton Harbor, Mich.

MEETINGS COMMITTEE

Members of the Chicago Society for Coatings Technology chair the various subcommittees of the Meetings (Host) Committee under General Chairman Thomas E. Nevins, of U.S. Gypsum Co. They are: Program Operations — Rudy Albrecht, of Enterprise Co.; Information Services — Roy Beversdorf, of Dow Chemical Co.; Luncheon — Richard M. Hille, of United Coatings, Inc.; Publicity — Daryl G. Luxmore, of Drew Chemical Corp.

Mrs. Thomas (Winnie) Nevins and Mrs. Richard (Claudine) Hille are in charge of the Spouses' Program.

1978 Paint Industries' Show Exhibitors

Conrad Hilton Hotel • November 1-3

Aceto Chemical Co., Laporte Industries Ltd.
Air Products & Chemicals, Inc.
Alcan Metal Powders
Allen-Stevens Drum Access. Corp.
Alpine American Corp.
Aluminum Co. of America
C.M. Ambrose Co.
American Felt and Filter Co.
American Hoechst Corp.
American Nepheline Corp.
AMF Cuno Div.
Amoco Chemicals Corp.
Applied Color Systems, Inc.
Ashland Chemical Co.
Atlas Electric Devices Co.

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

Cabot Corp., Cab-O-Sil Div.
Cargill, Inc.
CDI Dispersions
Celanese Chemical Co.
Celanese Polymer Specialties Co.
Chemetron Pigments
Chicago Boiler Co.
Cities Service Co., Colombian Chems. Div.
Color Corp. of America
Cosan Chemical Corp.

Daniel Products Co.
Davies Can Co.
Degussa Corp.
Diamond Shamrock, Process Chems. Div.
Diano Corp.
D/L Laboratories
Draiswerke, Inc.
Drew Chemical Co.

Eastman Chemical Products, Inc.
Ebonex Corp.
Engelhard Minerals & Chemicals Corp.
Epworth Mfg. Co., Inc.

Filter Specialists, Inc.

Gardner Laboratory, Inc.
General Mills Chemicals, Inc.
Georgia Kaolin Co.
Goodyear Chemicals
W.R. Grace & Co., Davison Chem. Div.
Graco, Inc.

Halox Pigments
Harmon Colors Corp.
Harshaw Chemical Co.
Hercules Incorporated
Hilton-Davis Chemical Co. Div.
Hockmeyer Equipment Corp.
Hooker Chemicals & Plastics Corp.
J.M. Huber Corp.
Hunter Associates Laboratory, Inc.

IBM Instrument Systems
Impandex, Inc.
International Mins. & Chems. Corp.
Interstab Chemical Co.

Johns-Manville

Kelco, Div. Merck & Co.
Kenrich Petrochemicals, Inc.
Kohnstamm & Co., Inc.

Macbeth Div., Kollmorgen Corp.
Madison Industries, Inc.
Manchem Limited
Mateer-Burt Co.
Meadowbrook Corp.
Merck Chemical Div.
Mini Fibers, Inc.
Modern Paint & Coatings
Montedison USA, Inc.
Morehouse Industries, Inc.
Myers Engineering, Inc.

Nalco Chemical Co.
Netzsch Bros., Inc.
Neville Chemical Co.
N L Industries, Inc.

O'Brien Industrial Equip. Co.

Penn Color, Inc.
Pennsylvania Glass Sand Corp.
Pfaudler Co.
Pfizer, Inc., MPM Div.
Photomarker Corp.
Polyvinyl Chemical Industries
PPG Industries, Inc.
Premier Mill Corp.
Productos de Zinc y Plomo, S.A.
PVO International, Inc.

Quackenbush Co.
Q-Panel Co.

Reichard Coulston, Inc.
Reichhold Chemicals, Inc.
Rohm and Haas Co.
Russell Finex, Inc.

Sales y Oxidos, S.A.
SCA Chemical Waste Services, Inc.
Schold Machine Corp.
Shamrock Chemicals Corp.
Shell Chemical Co.
Silberline Mfg. Co., Inc.
Spencer Kellogg Div., Textron, Inc.
Sun Chemical Corp.
Synres Chemical Co.

TAMMSCO, Inc.
Tenneco Chemicals, Inc.
Testing Machines, Inc.
Thiele Engineering Co.
Tokheim Corp.
Torco Engineered Prods. Div.
Troy Chemical Corp.

Union Camp Corp.
Union Carbide Corp.
Union Chemicals Div., Petrochem. Grp
Union Process, Inc.
Universal Color Dispersions, Inc.

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

Warren Rupp Co.
Waters Associates, Inc.
Weathering Research Service Co.
Wellco Chemical/Itasco Ind.
Wilden Pump & Engineering Co.

SHOW HOURS

Wednesday, Nov. 1 — 12:30—6:00 pm
Thursday, Nov. 2 — 10:00 am—6:00 pm
Friday, Nov. 3 — 10:00 am—4:00 pm

FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

1978 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW
CONRAD HILTON HOTEL, CHICAGO, ILLINOIS
NOVEMBER 1, 2, 3, 1978

**"COATINGS:
SEARCH
FOR
OPPORTUNITIES"**

**MAIL TO: Coatings Federation Housing Bureau
Conrad Hilton Hotel
720 South Michigan Ave.
Chicago, Ill. 60605**

APPLICATION FOR ACCOMMODATIONS

RESERVATION: Please reserve the following accommodations: See reverse side for room rates. Room reservations cannot be guaranteed unless this form is received by Oct. 3, 1978. Reservations accepted only on official housing form.

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

CHOICE OF HOTELS:

1st choice ____________________

2nd choice ____________________

ARRIVAL AND DEPARTURE:

Arrival: Date ____________________ Hour ____________________

Departure: Date ____________________ Hour ____________________

Reservations held only until 6 p.m. unless later arrival is indicated.

NAMES AND ADDRESS OF ALL OCCUPANTS OF ROOMS (Please "bracket" those rooming together). Incomplete information **WILL DELAY** assignment of room. Type additional names on a separate sheet, or on reverse side.

NAME(S) ADDRESS(ES)

SEND CONFIRMATION TO:

PLEASE PRINT OR TYPE

NAME ____________________

FIRM ____________________

ADDRESS ____________________

CITY ____________________ STATE ____________________ ZIP ____________________

LIST ADDITIONAL NAMES BELOW

HOTEL INFORMATION AND RATES

The Conrad Hilton and Pick-Congress Hotels have reserved blocks of rooms specifically for the 1978 Annual Meeting and Paint Industries' Show of the Federation of Societies for Coatings Technology, November 1, 2, 3, 1978, at the Conrad Hilton.

The Pick-Congress is located two blocks from the Conrad Hilton.

All room rates are subject to additional 6.1% charge to cover Illinois Hotel Operators' Occupation Tax and the Chicago Hotel Operators' Tax, as well as a 2% Chicago Hotel Accommodations Tax imposed by the City of Chicago.

You will receive confirmation from the hotel to which you have been assigned. If you wish to cancel or change the date of your reservation, contact that hotel directly. If the rate requested is not available, the next available rate will be confirmed.

	Conrad Hilton Hotel 720 S. Michigan Ave. Chicago, Ill. 60605 312-922-4400	**Conrad Hilton Towers**	**Pick-Congress Hotel** 520 S. Michigan Ave. Chicago, Ill. 60605 312-431-1102
Singles	$35 41 44 47 62*	$44 67*	$29 34 39
Doubles	$47 53 56 59	$56	$41 46 51
Twins	$47 53 56 59 62*	$47 56 67*	$41 46 51
Suite (P&1)	From $67 to $285	From $70 to $255	From $65 to $150
Suite (P&2)	From $185 to $650	From $190 to $400	From $105 to $250

*Deluxe Twins

SAVE TIME AND $$$, TOO!!

REGISTER NOW

Annual Meeting and Paint Show

November 1-3, 1978

Chicago, Illinois

USE ADVANCE REGISTRATION FORM ON REVERSE SIDE OF THIS PAGE

You can pre-register for the 1978 Annual Meeting and Paint Show by completing this advance registration form and mail with your check to FSCT headquarters. Your registration badge can be picked up at the Advance Registration desk in the Conrad Hilton Hotel, North Hall.

REGISTRATION FEES

	Member	Non-Member	Ladies' Activities
Advance*	$35	$50	$20
On-Site (Full-Time)	$40	$55	$25
On-Site (One-Day)	$20	$30	—

Note: You save $5.00 by registering in advance.

***Special registration for Retired Federation Members $15.00 in advance only.**

SAVE TIME AND $$$, TOO!!

To register in advance for the 1978 Annual Meeting and Paint Show, please fill in the adjacent form and return with your remittance, to:

Federation of Societies for Coatings Technology
Suite 832
1315 Walnut Street
Philadelphia, Pa. 19107

Forms must be received by October 13.

Federation Awards Luncheon
Friday, November 3
Conrad Hilton Hotel

Purchase of Luncheon tickets is optional, and is not part of Advance Registration fee. If you wish to attend, please check square below and include additional $10 with your registration payment.

Luncheon
($10 per person)

Note: Luncheon is open to all registrants, and will be held in place of the traditional banquet. Political humorist Mark Russell will be guest speaker.
Price of Luncheon is included in Spouses' Registration fee.

Spouses Activities $20.00

ADVANCE REGISTRATION

PLEASE PRINT EXACTLY as you wish registration badge prepared

NAME

ADDRESS

CITY STATE

ZIP (U.S. or CANADA) COUNTRY (OTHER THAN U.S.)

PLEASE PRINT EXACTLY as you wish registration badge prepared

FIRST NAME LAST NAME

BUSINESS AFFILIATION

STREET

CITY STATE *(U.S. only)*

ZIP (U.S. or CANADA) COUNTRY (OTHER THAN U.S.)

Your title or position ______

Name of the Federation Society
Of which you are a paid member ______

Hotel at which you are staying ______

ADVANCE REGISTRATION

CONRAD HILTON HOTEL
CHICAGO, ILLINOIS
NOVEMBER 1, 2, 3

Federation Member	NON-Member
$35.00	**$50.00**

YOUR COMPANY *(Check One)*:

- A ☐ Manufacturers of Paints, Varnishes, Lacquers, Printing Inks, Sealants, etc.
- B ☐ Manufacturers of Raw Materials
- C ☐ Manufacturers of Equipment and Containers
- D ☐ Sales Agent for Raw Materials and Equipment
- E ☐ Government Agency
- F ☐ Research/Testing/Consulting
- G ☐ Educational Institution/Library
- H ☐ Paint Consumer
- J ☐ Other

YOUR POSITION *(Check One)*:

- A ☐ Management/Administration
- B ☐ Manufacturing and Engineering
- C ☐ Quality Control
- D ☐ Research and Development
- E ☐ Technical Sales Service
- F ☐ Sales and Marketing
- G ☐ Consultant
- H ☐ Educator/Student/Librarian
- J ☐ Other

FSCT Annual Meeting and Paint Show

Mail to: FSCT, 1315 Walnut St., Philadelphia, Pa. 19107

CPSC Announces Policy, Procedure On Carcinogens in Consumer Products

A general policy and procedure for classifying, evaluating and regulating carcinogens in consumer products has been established by the Consumer Product Safety Commission. As published in the June 13 issue of the *Federal Register,* the policy statement presents guidelines concerning the standards CPSC plans to use to classify suspected carcinogens and to evaluate those substances when they are contained in consumer products. The policy also suggests what regulatory actions CPSC will take following classification of substances as carcinogens.

CPSC stated that it developed its policy because it has a public responsibility and statutory jurisdiction to regulate products containing carcinogens to which consumers may be exposed. In general, CPSC's policy is to prohibit carcinogens from being intentionally added to consumer products if they can be absorbed, inhaled, or ingested into the human body; to require the use of substitute products when available; or to reduce the carcinogenic substance to the lowest attainable level until a substitute is identified.

The CPSC policy has not been proposed as a rule and therefore has no binding legal effect. The determination to take regulatory action against particular products or classes of products will continue to be made in individual proceedings, in accordance with the applicable statutory provisions and the terms of the policy statement.

Specifically, CPSC's guidelines include the following four steps:

(1) CPSC will determine whether a suspected carcinogen is contained in any consumer product.

(2) CPSC will classify the substance based on the type and quality of the available test data and studies. Category A will contain substances for which there is strong evidence of carcinogenicity. Category B will contain substances for which the evidence of carcinogenicity is suggestive. Category C will contain substances for which there is some question of carcinogenicity or substances that are members of families of chemicals whose members are carcinogenic. Category D will contain substances which were previously classified A, B, or C but which no longer indicate carcinogenic potential. Classifications in Categories B and C will be subject to change based on further testing and additional data.

(3) CPSC will evaluate products containing classified substances and determine what regulatory action should be taken. CPSC will consider the extent to which those products containing classified substances are used and by whom; the potency of the substance; the potential for harmful exposure to humans; the probable effect of the regulation on the hazard; and the social and economic impact of the regulation.

(4) CPSC will select the appropriate regulatory action for classified substances, CPSC will identify the proper statutory authority and determine the solution to the hazard. For Category B substances, CPSC will make every effort to do further testing to warn consumers about possible risks in using products containing those substances. Manufacturers may be required to make health and safety information available to consumers who use products containing products in Categories B and C. For Category D substances, CPSC will maintain and update records on test data and studies.

CPSC has requested public comment on its interim statement of policy and procedures. Written comments (five copies) should be sent to the Secretary, Consumer Product Safety Commission, Washington, D.C. 20207 by October 11, 1978.

Supreme Court Holds Warrantless OSHA Inspection Unconstitutional

The Supreme Court, in a decision handed down on May 23, in the case of Marshall vs. Barlow's, Inc., has held that warrantless inspections of business premises under Section 8(a) of the Occupational Safety and Health Act of 1970 violate the Fourth Amendment's prohibition against unreasonable searches.

The decision means that in the future OSHA inspectors must obtain a search warrant if refused permission to enter and inspect a company's work area for safety hazards and violation of OSHA regulations.

The Supreme Court concluded that the administrative burden imposed on OSHA and the potential loss of surprise inspections are outweighted by the right of the employer to demand a search warrant. The Court noted that a warrant provides assurances from a neutral officer that the inspection is reasonable under the Constitution, is authorized by statute, and is pursuant to an administrative plan containing specific neutral criteria. The Court further noted that the warrant would advise the employer of the scope and objects of the search beyond which limits the inspector would not be expected to proceed.

With respect to what evidence will be required to secure a warrant, the Supreme Court made it clear that such evidence will not be contingent upon OSHA demonstrating probable cause to believe that violation exists on the premises. It appears that a showing that a specific business has been chosen for an inspection on the basis of a general administrative plan for the enforcement of the OSHA statute derived from neutral sources and that the desired frequency of such inspections has not been abused would be sufficient to permit a warrant to be issued.

Although many of the federal environmental laws contain similar inspection provisions, the Supreme Court expressly limited its decision to warrantless inspections under Section 8(a) of OSHA.

Pollution Control Efforts Add to Energy Use

Efforts by some industries to reduce energy consumption may be offset by additional energy requirements for air and water pollution control, according to a study done by Monsanto Research Corp. for the Power Technology and Conservation Branch, U.S. Environmental Protection Agency, Cincinnati, Ohio.

The organic chemical manufacturing and petroleum refining industries are expected to be hit especially hard by anti-pollution energy requirements, the study says. However, alternative methods to those currently used for water pollution abatement could reduce energy requirements.

The study is based on requirements in 1972 amendments to the Federal Water Pollution Control Act, administered by the EPA. These amendments require the application of "best practicable control technology currently available" by 1977, and application of "best available technology economically achievable" by 1983.

New amendments in 1977 to the water pollution law extended compliance dates by one to four years, depending on the pollutant and whether the treatment facilities are operated by an industry or by a municipality. The effect of the 1977 extensions was not taken into account by the study.

The report says that 2.2% of total U.S. energy requirements in 1977 would be needed for pollution control, and that the percentage would increase to 2.9% in 1983. About two-thirds of the energy requirement would be expended by industry, with the remaining one-third divided nearly equally between electric power plants and municipal treatment and disposal facilities.

There are discrepancies among reports ranking industries according to energy requirements for water pollution control, according to the study. However, the study notes, all reports agree that a major portion of the projected energy burden for wastewater treatment will fall on the chemical industry, and that the organic chemicals segment will account for about 95% of the chemical industry requirement.

The study predicts that about 9% of the energy used in the manufacture of organic chemicals will be expended to meet 1983 water pollution control regulations.

When air pollution is taken into account, the primary metals industry will account for the major portion of the energy requirement. Within the metals industry, most of the burden will fall on iron and steel manufacturing operations.

The study says its estimates are based primarily on current methods of treating water pollutants after they have occurred — that is, as they come out of a pipe at the end of the manufacturing process. By using alternative treatment techniques, particularly changes inside plants to reduce pollutants in wastewater, the energy requirements needed to combat pollution could be reduced, according to the report.

The study gives alternate, in-plant water treatment techniques reported in the literature for several industries, including organic chemicals, petroleum refining, pulp and paper, and iron and steel.

OSHA Regs Topic of "Update" By MCA on September 14

The Manufacturing Chemists Association will sponsor a one-day "OSHA Update" symposium September 14 at the Cherry Hill Inn, Cherry Hill, N.J.

Organized as an activity of MCA's Occupational Safety and Health Committee, the symposium will focus on various aspects of the U.S. Occupational Safety and Health Administration's existing and proposed regulations. The meeting is intended to help large and small chemical companies, and any others which use or handle chemicals, in meeting their obligation to provide safe and healthful working conditions for their employees.

The morning session will include speakers on toxicology, OSHA's proposed carcinogen standard, and new and proposed safety standards.

The afternoon segment will offer presentations and discussions on administrative rules and regulations, OSHA's proposal for chemical substances identification in the workplace, safe utilization of the handicapped in industry, and access to records.

Carl DeMartino, of the duPont Co. and Chairman of MCA's committee, will make the luncheon address. Other speakers at the workshop will be corporate safety and health experts from MCA member companies.

The $20 registration fee covers the luncheon and coffee breaks. Further information and registration forms are available from Milton Freifeld, Manufacturing Chemists Association, 1825 Connecticut Ave., N.W., Washington, D.C. 20009.

OIL & COLOUR CHEMISTS' ASSOCIATION

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Friday	6 April 09.30 — 16.00 hrs

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FOR APPLICATION TO EXHIBIT CONTACT:

The Director & Secretary,
OIL & COLOUR CHEMISTS' ASSOCIATION
Priory House, 967 Harrow Road, Wembley, Middx, HAO 2SF, England
(Telephone 01 908-1086 Telex 922670 (OCCA Wembley))

Review of Skid and Slip Resistance Standards Relatable to Coatings

Paul R. Guevin
Hughson Chemicals Division of Lord Corporation*

Slips and falls result in unnecessary deaths and disabling injuries each year. In many cases, an organic polymeric surface is involved in the accident. ASTM has at least eight committees working on methods of measuring the coefficient of friction and establishing numerical standards. The National Bureau of Standards and Liberty Mutual Insurance Company are studying human perambulation in an attempt to develop better equipment to measure the coefficient of friction and are attempting to establish standards. The microstructure of organic coatings is shown to have a definite influence on the coefficient of friction of coatings, sometimes in unexpected ways.

INTRODUCTION

Slipperiness is the tendency or liability to cause something to slide suddenly or involuntarily. Slips and falls in the home, office, public buildings, etc. resulted in 9,600 deaths and 1,600,000 disabling injuries according to 1974 National Safety Council data. In many cases, an organic polymeric surface (coating) is involved in the accident - be it an alkyd porch-and-deck enamel, a vinyl resilient floor covering, a pour-inplace urethane athletic gymnasium floor or a "waxed" vinyl asbestos floor tile. With the advent of the OSHA and other government regulatory agencies, the importance of measuring a coating's coefficient of friction is becoming more apparent to the manufacturer of protective coatings. The purpose of this paper is to investigate the state-of-the-art and to determine a course of action for the coatings industry.

A literature search revealed some interesting results. Using the New England Research Application Center (NERAC) and their facilities for a computer literature search, given the key words almost all of the abstracts "hit" concerned the metal alloy industry or polymers in general. None of the "hits" pertained to the coatings industry.

Definitions of terms are variant. One dictionary defined a skid as an act of sliding without rotation or as a failure to grip the roadway. This term seems more applicable to the tire industry. Slip can be defined as a slide which occurs suddenly or involuntarily and appears to describe the phenomenom satisfactorily. Skid and slip resistance can be considered as two aspects of slipperiness.

A standard is a rule established by an authority for measure of quantity, weight, extent, value, or quality. This definition encompasses most of the points that relate to this paper. The authority can be the American Society for Testing and Materials (ASTM), the Chemical Specialty Manufacturing Association (CSMA), or the Federal government who may establish coefficient of friction values for our products at some time. As yet, there are relatively few coefficient of friction standards in the coatings industry. The floor polish industry has stated that a coefficient of friction value of less than 0.5 constitutes a slippery surface and one that is greater than 0.5 is not slippery. The opposite term of slippery is tack.

COEFFICIENT OF FRICTION

Coefficient of Friction (μ) is the ratio of the force, F, required to move one surface over another surface to the total force, W, pressing the two together, expressed as:

$$\mu = \frac{F}{W} \tag{1}$$

A simple experiment (*Figure* 1) illustrates a method for obtaining the coefficient of friction.[1] The coefficient of friction can be viewed in either a static or a dynamic condition. In the above experiment, the force needed to start the object to slide (static friction) is greater than the equilibrium sliding force (dynamic friction).

Is it static or dynamic friction that is important to the slip and fall problem? Under what circumstances should they be measured? The seriousness of the problem resulting from floor slipperiness and the large number of people involved have fostered many investigations of flooring, floor finish, and shoe manufactur-

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Presented at the 21st Cleveland Society for Coatings Technology Symposium, "Advances in Coatings Technology," March 22, 1978.

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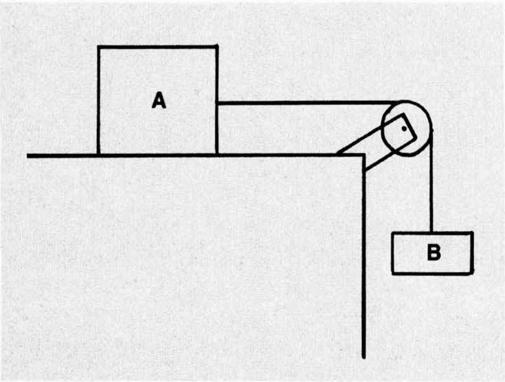

Figure 1 — Coefficient of friction experimental apparatus

ers, research laboratories, and insurance companies. Kinesiological studies made at Berkeley[2] and in Sweden[3] indicate that no slip occurs between the shoe and the floor during normal walking. It has been concluded by the National Bureau of Standards[4] that in order for slip to occur during normal walking, the static coefficient of friction must be overcome. Once slip occurs, the dynamic coefficient of friction and particularly the ratio of dynamic to static may very well become important.

The coefficient of starting friction may be calculated from the limiting angle of repose, ϕ:

$$\mu = \tan \phi \qquad (2)$$

Using the principle of equation (2), the slip-angle apparatus (*Figure* 2) was developed. The apparatus is a Fisher-Payne dip coater, a simple protractor, a hinged plywood plate, and a sled. The movable plywood plate is leveled and the pointer adjusted to 0°. With the dip coater at the lowest speed, the platform is raised. The angle at which the sled first begins to move is recorded. The tangent of the angle recorded is the static coefficient of friction. The two drawings in *Figure* 3 illustrate some resultant data. The surface on the right (where μ equals 0.36) is more slippery than the surface on the left (where μ is 0.90).

TEST EQUIPMENT AND METHODS

Most methods and equipment currently used for measuring surface slip resistance can be separated into three types: (1) drag type meter, which is based on equation (1) and the simple physics experiment previously described; (2) pendulum type meters, which measure the energy loss of the pendulum as an indirect indication of the dynamic friction; and (3) articulated strut devices, which are based on the direct and fundamental principle of the resolution of forces, equation (2). Brungraber[5] prepared an excellent review of the state-of-the-art of slip resistance studies through 1975.

Drag Type Meters

Most drag type meters (Horizontal Pull Slipmeter, TOPAKA, and Technical Products Tester) are portable for use directly on a floor. These testers are sometimes referred to as fish-scale type testers. Some laboratories have modified their Instron Tensile Testers to perform similar operations. This approach has the advantage of recording the forces graphically. The latest equipment using these principles is Instrumentors Slip/Peel Tester (*Figure* 4) developed by Robert Heinrich and can produce actual recorded traces as the coefficient of friction measurements are taken.

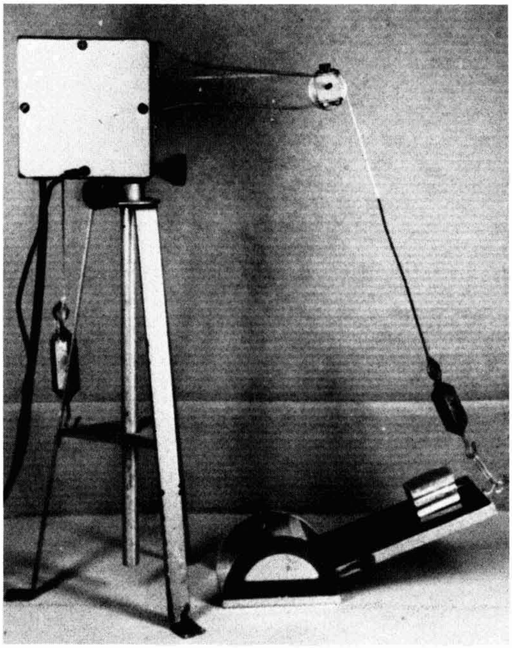

Figure 2 — Slip-angle measurement apparatus

ASTM Committee D-20 on Plastics and its Subcommittee D20.10 on Mechanical Properties developed ASTM D 1894—"Standard Test Method for Static and Kinetic Coefficients of Friction of Plastic Film and Sheeting."[6] This method involves the sliding of one surface over another surface at specified test conditions. This is one of the few standard test methods that measures static and dynamic (kinetic) friction. Although this test method is under the jurisdiction of a plastics ASTM committee, it could be applicable to the coatings industry. Liberty Mutual Insurance Co.'s

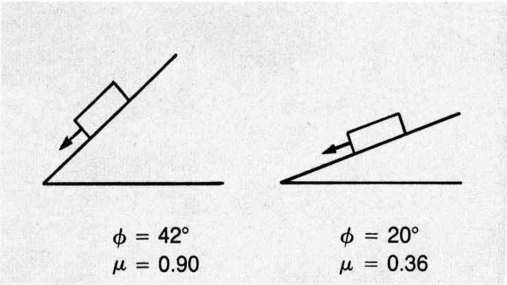

Figure 3 — Illustration of two slip angle measurements

Figure 4 — Instrumentors slip/peel tester

Charles H. Irvine has used the Horizontal Pull Slipmeter (HPS) to develop some interesting data.[7] Further work from their laboratory,[8, 9] has validated Irvine's earlier results and presents a good case to use a Horizontal Pull Slipmeter. The coatings industry can consider as *equipment and test methods* the HPS, Instrumentors Slip/Peel Tester, or similar drag type meter, and ASTM D 1894 as one approach to measure the coefficient of friction of coatings.

Pendulum Type Meters

These instruments for measuring the coefficient of friction are represented by the Sigler and the British Portable Skid Tester (BPST). They consist of a pendulum, faced with a certain shoe sole or heel material, which can be adjusted to sweep a path across a flooring surface so that the contact pressure between the facing and the floor follows a predetermined time-dependent pattern. The resultant loss of energy of the pendulum is claimed to be a measure of the dynamic friction. Slow motion films of some BPST tests conducted by the National Bureau of Standards[10] show that the contact pressure varies erratically with time. The BPST has shown good correlation with the results of automotive skid tests, particularly on wet pavements, and reveals the effects of water on the slip resistance of sole and heel materials, but some coating suppliers have not obtained a good correlation with the observed in-service results. Early editions of the Gardner-Sward *Physical and Chemical Examination of Paints, Varnishes, Lacquers and Color* describe the Sigler method for slip resistance (12th Edition). ASTM Method E 303—"Measuring Surface Friction Properties Using the British Portable Tester" was prepared by ASTM Committee E-17 on Skid Resistance.[11] Based on general results from some members of ASTM, it appears that the pendulum-type meters are not applicable for further consideration.

Articulated Strut Meters

The *articulated strut meters* are represented by the James Machine, the Hunter Tester, and the NBS/Brungraber Tester. These instruments apply a known constant vertical force to a shoe faced with a certain sole or heel material and then applied at increasing lateral (forward) force until slip occurs. The ratio of the lateral force at slip to the known vertical force is the static coefficient of friction. The vertical force is applied to the top so that the shoe is subjected to a vertical load only. As the test progresses, the articulated strut is slowly inclined so that the shoe continues to be subjected to the constant vertical load, in addition to an increasing horizontal or tangential load, until slip occurs. The tangent of the angle that the articulated strut makes with respect to the vertical at the instant of slip is taken to be the ratio of the horizontal and vertical components of the force applied to the shoe, thus the static coefficient of friction. *Figure* 5 shows the latest equipment on the market, the NBS/Brungraber Tester.

ASTM Committee D-21 on Polishes conducted some extensive work using the James Machine. Members of the Waxes and Polishes Div. of the Chemical Specialties Manufacturers Association also participated in this work. From their efforts resulted ASTM D 2047—"Test for Static Coefficient of Friction of Polish-Coated Floor Surfaces as Measured by the James Machine."[12] At present, it is the only slip resistance test method for floors that appears in an ASTM specification. This procedure appears to be a satisfactory test method for the evaluation of flooring products and treatments and shoe sole and heel materials. However, it is limited to laboratory use since the James Machine is not portable.

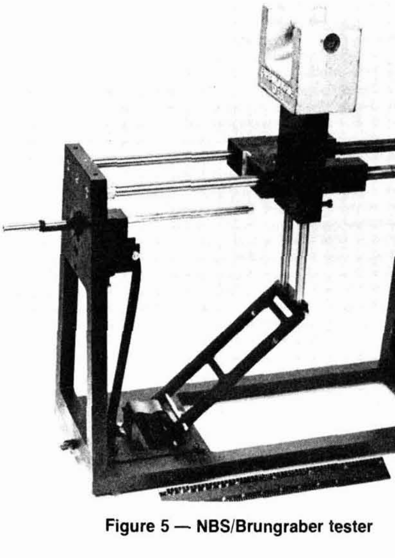

Figure 5 — NBS/Brungraber tester

Table 1—Standards for Coefficient of Friction

	Coefficient Of Static Friction			Coefficient Of Sliding Friction		
	Dry	Wet	Oily	Dry	Wet	Oily
For Leather						
Epoxy	0.75	0.75	—	0.50	0.50	—
Urethane	0.85	0.85	—	0.50	0.60	—
For Rubber						
Epoxy	1.00	0.90	0.70	0.80	0.80	0.40
Urethane	1.00	0.85	1.00	0.85	0.85	0.70

A new, portable slip resistance tester, the NBS/Brungraber Tester,[4] shown in *Figure* 5, employs the same fundamental principles of the articulated strut as the James Tester. This new slip resistance tester is self-actuated so no external power source is needed. According to the designer, it is the only commercial coefficient of friction instrument which truly simulates the mechanics of walking and reveals the reduction in walking slip resistance caused by the presence of a film of water.

ASTM ACTIVITIES

At the present time, ASTM Subcommittee D21.06 (Slip Resistance) is engaged in an extensive round robin test program which involves 12 different laboratories. Subcommittee D21.06 is comparing the following coefficient of friction test instruments using ASTM D 2047: James Tester NBS/Brungraber Tester, Sigler Machine, TOPAKA Machine (adopted by CSMA), and the CSMA under-foot test. An interim report is expected within 1978.

ASTM Committee E-17 on Skid Resistance sponsored a symposium during the ASTM meeting in Denver during June 1977. Nine technical papers were presented and are being assembled into a STP (Standard Technical Publication) which is expected to be published shortly by ASTM. The objective of the symposium was to bring together some current work being done on coefficient of friction measurements, to present a state-of-the-art discussion on related topics, and to focus further attention on this rather complex problem of pedestrian safety. The combination of the STP and the results of ASTM Committee D-21 round robin activity should shed some new light on how slip resistance test machines compare and what can be anticipated as on-going work by ASTM committees.

In addition to the slip resistance activities of ASTM Committees D-20, D-21, and E-17, ASTM Committees D-1 on Paint and Related Coatings and Materials, D-11 on Rubber, D-31 on Leather, F-6 on Resilient Floor Coverings, F-13 on Safety and Traction for Footwear, and F-15 on Consumer Product Safety have at least one group within their subcommittees actively involved or looking at some aspect of slip resistance. In late 1977, Subcommittee F06.40 (Slip Resistance) organized a group and planned a visit to the National Bureau of Standards which houses almost all coefficient of friction test instruments. The subcommittee is now determining which instrument(s) is applicable to institute a study of slip resistance measurements on resilient floor coverings.

Committee D-1 is taking a different approach. Group 15 of Subcommittee D01.23 will refine the equipment so sliding (dynamic) coefficient of friction as well as static coefficient of friction can be measured. They will develop a test method based on the slip-angle test apparatus.

STANDARDS

As previously stated, there are relatively few slip resistance standards in the coatings industry; this may appear strange to some coatings chemists since there are many painted surfaces in pedestrian walkways. The United States Navy wrote a specification, MIL-D-23002, in 1965 based on slip resistance studies some of which date back to the old Brooklyn Navy Yard. This old specification is in the process of being revised. The Canadian Government Specification Board adopted two standards for deck coatings based on this U.S. specification: nonslip epoxy (1-GP-192) and nonslip polyurethane (1-GP-200). The coefficient of friction specifications taken from these documents are combined and shown in *Table* 1. These data bring out some important aspects of slip resistance measurements. Although some of the data are being questioned as the specification is undergoing revision, values point up the differences in slipperiness of various shoe sole materials. *Table* 1 illustrates the importance of specifying the nature of sole material, i.e., leather or rubber. With some consistency leather gives a lower coefficient of friction than rubber. The same conclusion is drawn from Liberty Mutual Insurance Co. work.[8] A newer specification, MIL-D-24483-A, which covers a spray applied deck coating, was issued recently.

As more and more data on skid resistance where pedestrian traffic is involved becomes available, it appears that it is just a matter of time before OSHA becomes aware of the importance of establishing skid resistant coatings and requires standards to be set and met for floor coatings.

SLIP TESTING IN THE COATINGS INDUSTRY

There is increased concern in the coatings industry about instrumental measurements of the coefficient of friction since people walk on "painted" floors. The activity previously mentioned in ASTM Committee D-1 is only one example. A study by Consumers Union[13] indicated a need for good slipperiness measurements but evidence showed that present instrumentation is inadequate since the results did not correlate with practical observations. Their last resort was some in-use tests performed in a statistical manner.

In some early work to develop a coating for rubber that had a low coefficient of friction, Hill measured the

Table 2—Coefficient of Friction Values for SBR

Durometer	μ
40	1.66
50	1.60
60	1.15
70	0.97

slip resistance of various rubbers and coatings applied to rubber using a Slip-Angle Tester (*Figure* 2) and developed a test method which has been previously described in this paper.[14] Coefficient of friction values for rubber are shown in *Table* 2. Similar data for coatings appears in *Table* 3. A discussion of these data appears in the section below.

Other companies within the coatings industry have investigated alternate equipment to measure slip resistance. The Horizontal Pull Tester appears applicable although some companies doubt that the resultant data are meaningful. The Instron or Instrumentors Slip/Peel Tester can be used to record surface drag. These instruments conform to ASTM D1894 which can be used as a method to measure the coefficient of friction of coatings. Interest in the NBS/Brungraber Tester in the coatings industry is increasing. One coatings company has already purchased an instrument and another one is in the process of purchasing one.

As an adjunct to the development of a rubber window channel, several SBR rubber formulations were screened. Rubbers with Durometer hardness values from 70 (hard) to 40 (soft) were prepared by varying the formulation (SBR resins, oil type and length, carbon black, and curatives). These changes in the elastomer formulation are reflected in the resultant slip resistance (*Table* 2). The formulation with the best performance as an elastomer for the automotive window channel was the softest SBR (40 Durometer). Unfortunately, this elastomer formulation had the highest coefficient of friction which would not allow the glass to move freely in the channel.

Probably one of the most important features contributed by coatings on rubber is the modification of surface characteristics. In his work to develop a suitable coating, Hill investigated variables in the coatings microstructure: resins, pigments, and flatting agents. The marked reduction in elastomer coefficient of friction by coatings is shown in *Table* 3. Although these coatings were based on proprietary formulations, the results do show some general trends. Flexible resins produce coatings with more slip resistance than coatings formulated with rigid resins. The rigid clear coating had the lowest coefficient of friction but was not an acceptable coating, from a flexibility standpoint, for the intended use. Flexible coatings with flatting extender pigments offered the best route for a commercial product. The low gloss black coating was accepted by the automotive industry to coat rubber for window channels. *Figure* 6 illustrates this application which was popular when the picture was taken, circa 1964.

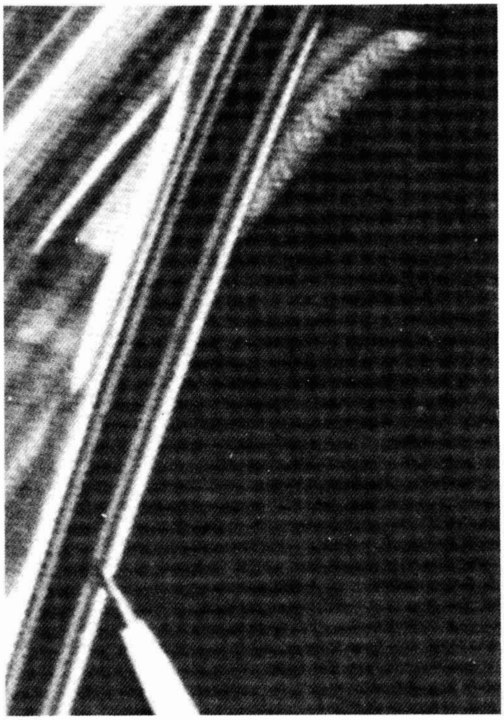
Figure 6 — Illustration of automotive window channel

Besides automotive window channels, bowling alleys require surfaces with a low coefficient of friction. *Figure* 7 illustrates a typical lane cross section. In most instances, the bowling ball rides on an oil surface rather than making direct contact with the clear wood coating. Since the oil is not permanent, bowling establishments use clear coatings which are formulated with proprietary materials to achieve a low coefficient of friction. Most likely, no instrumental measurements are made in

Table 3—Coefficient of Friction of Coated Elastomers

Material	μ
Uncoated SBR (40 Durometer type)	1.66
SBR with glossy black coating	1.10
SBR with clear, flexible coating	0.90
SBR with low gloss clear coating	0.49
SBR with low gloss black coating	0.42
SBR with clear, rigid coating	0.33

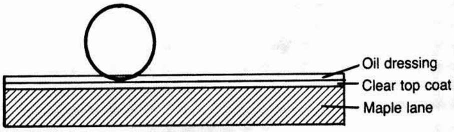

Figure 7 — Diagram of bowling lane

Table 4—Typical μ Values (Polymer over Polymer)

Polymer	μ
Cellulose acetate	0.59
Polyacrylonitrile	0.49
Nylon	0.45
Polyethylene terephthalate	0.29
Teflon	0.07

the development of these special clear bowling alley finishes. More likely, the coatings manufacturer tests coatings in a series of in-use applications on test lanes and rates the coatings accordingly. The United States and Canadian Navies have specifications for nonslip deck coatings in which oil is used as part of a coefficient of friction test. These offer possibilities for achieving quantitative results in which the coatings manufacturer can screen resins and additives to achieve an improved bowling alley finish.

Fort[15] studied the coefficient of friction of polymers and "treated" polymers in a textile application. Measurements were made using an apparatus similar to the instrument described in ASTM D3028 method.[6] Fort's data (*Table* 4) clearly indicate that various polymer backbones affect the coefficient of friction. Teflon®'s very low surface energy, combined with its relatively high yield pressure, leads to weak adhesion over a small contact area so that slip resistance is always low.[16]

Besides coatings with a low coefficient of friction, Consumers Union's report on porch-and-deck coatings indicated a need for coatings with high coefficients of friction. Their report pointed out that a greater skid resistant surface should be the objective of the manufacturers of these coatings. In their evaluation of 24 porch-and-deck coatings, of which 13 were latex-based and 11 were oil-based, they performed some in-use tests to rate the coatings since they were not always able to translate laboratory instrumental tests of resistance to slipping into actual performance on painted wooden floors. The results in *Table* 5 show that latex-based coatings were far less skid-prone than oil based coatings. However, in their judgement, none of the coatings could be considered nonskid. Without knowing the exact compositions, one cannot determine what specific variables in the coatings microstructure (formulation) effected the differences in slip resistance but it is quite apparent that the categorical difference between the latex and the oil-based resins produced notable differences in slip.

Table 5—CU Porch-and-Deck Paint Evaluation

Vehicle[a]	Rating of Slip Resistance
Acrylic latex (2)	Excellent
Latex (9)	Very good
Latex (1)	Good
Alkyd (5)	Fair
Alkyd (4)	Poor
Oil modified urethane (1)	Poor

(a) Numbers refer to species in this category.

CONCLUSIONS

The multitude of types of slip resistance test instruments and the various models within each type make it difficult to determine which instrument should be given further study. ASTM Committees D-1 and F-6 have taken the position that something should be done in their area of specific interest. Already some probing work is underway. The two ASTM methods—D1894 and D2047—appear to be adequate test methods which would be applicable in the coatings area but the selection of suitable test instruments needs to be accomplished.

Certainly the limited work reported by some laboratories points to a definite influence of a coatings formulation on its slip resistance. However, there is a need to perform more work to quantify this relationship. Since much of this work would involve proprietary information, it is doubtful that coatings manufacturers will publish their results. It is recommended that activity in ASTM and other standards organizations be monitored to keep abreast of emerging technology.

ACKNOWLEDGMENT

The author acknowledges with thanks Sanford C. Adler, of the National Bureau of Standards and Dr. Robert J. Brungraber, of Bucknell University for their technical assistance and their help in obtaining documents and pictures. The author thanks the management of Lord Corp. for permission to publish this paper.

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Approximate Rheological Characterization of Casson Fluids

Template Method For the Brookfield Synchro-Lectric Viscometers

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The non-Newtonian characteristics of a wide variety of industrial products can be characterized by the empirical Casson flow equation. This paper presents a method for obtaining the two Casson parameters using computer generated graphs specifically designed for use with all models of the Brookfield Synchro-Lectric Viscometer. When used with cylindrical spindles and a special template, the Casson parameters are easily obtained. These empirical analogs for Casson viscosity and yield stress are often quite useful for describing and controlling product behavior.

INTRODUCTION

Rheological measurements are widely used to study and characterize many industrial products such as paints, emulsions, dispersions, and polymer solutions. One of the most commonly employed instruments for such measurements is the Brookfield Synchro-Lectric Viscometer. While this instrument does not measure apparent viscosity at well defined shear rates in a non-Newtonian fluid, its low cost and simplicity of operation are compelling reasons to find ways of using it to characterize such fluids.

Characterizing Pseudoplastic Fluids With a Yield Value

The flow properties of Newtonian fluids are easily characterized by one parameter known as the viscosity. This value can be obtained by means of Newton's law which states that a plot of shear stress vs. shear rate is linear and the slope of the line is the viscosity. Unlike Newtonian fluids, non-Newtonian systems may be both shear and time dependent. In this category one of the most common types of flow is pseudoplastic or time independent shear thinning behavior. Such fluids are evidenced by nonlinearity on shear stress-shear rate coordinates. In some cases pseudoplastic fluids act like solids and will not flow until a critical shear stress is exceeded. These fluids are said to have a yield value or a gel strength. Flow behavior in such systems may be classified as Bingham or non-Bingham types.

Bingham Fluids

In Bingham fluids, the behavior seen in *Figure* 1 can easily be represented by two parameters: a yield stress (the extrapolated intercept at zero shear rate) and a viscosity (the slope of the line using the yield stress intercept as the origin). In practical terms, the former parameter is a measure of the strength of the structure, or a measure of the initial stress which must be exerted to initiate flow. For example, toothpaste does not flow out of an open tube because it has a yield stress. The higher the yield stress the harder the tube must be squeezed to initiate flow. By contrast, the latter parameter is independent of shear rate and can be thought of as the "Newtonian" viscosity of the system without the yield stress. It has variously been referred to as the plastic, structureless, or extrapolated viscosity at infinite shear rate.[1]

Non-Bingham Fluids

While Bingham fluids produce linear shear stress-shear rate relationships, other fluids produce nonlinear shear stress-shear rate curves with nonzero intercepts. For these fluids, apparent viscosity decreases with increasing shear rate (*Figure* 1). Because so many systems (e.g., paints, vinyl plastisols, emulsions, polymer solutions, formulated dispersions, etc.) fall into this category (pseudoplastic with a yield value) it is of great interest to simply characterize their flow properties.

The Casson Equation

For many pseudoplastic fluids with a yield value, flow properties can be successfully characterized by means of the Casson equation.[2]

$$\tau^{\frac{1}{2}} = K_0 + K_1 \dot{\gamma}^{\frac{1}{2}} \qquad (1)$$

where τ is the shear stress and $\dot{\gamma}$ is the shear rate. This equation was originally derived for use with pigment-oil suspensions of the printing ink type. Equation (1) has also been shown to describe the properties of most coatings[3] and molten chocolate.[4,5] Asbeck[6] emphasized

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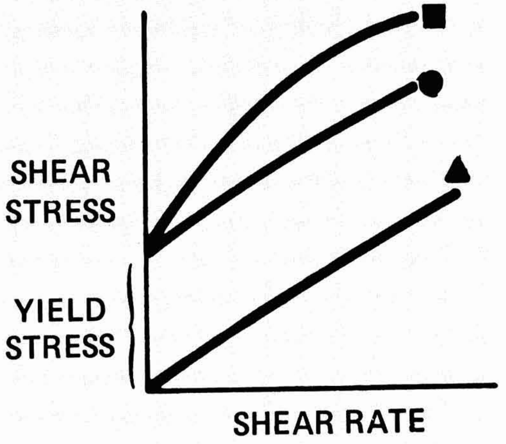


Figure 1—Three types of flow behavior: ■, Pseudoplastic with yield value; ●, Bingham; and ▲, Newtonian.

the applicability of this equation in the high shear region by employing an alternate form

$$\eta^{\frac{1}{2}} = \eta_\infty^{\frac{1}{2}} + \tau_0^{\frac{1}{2}}\dot{\gamma}^{-\frac{1}{2}} \tag{2}$$

where η_∞ is the limiting (i.e., independent of shear rate) high shear viscosity and τ_0 is the yield stress. If equation (2) is divided by $\gamma^{-\frac{1}{2}}$ one obtains

$$\tau^{\frac{1}{2}} = \eta_\infty^{\frac{1}{2}}\dot{\gamma}^{\frac{1}{2}} + \tau_0^{\frac{1}{2}} \tag{3}$$

which is a useful form for a low shear viscometer. If equation (3) applies, a straight line will result when the square root of shear stress is plotted vs. the square root of shear rate. This straight line can be interpreted by analogy with a Bingham fluid as shown in *Figure* 2. The intercept is the square root of the yield stress $\tau_0^{\frac{1}{2}}$ and the slope of the line is the square root of the infinite shear viscosity $\eta_\infty^{\frac{1}{2}}$. The latter corresponds to the plastic viscosity of a Bingham fluid.

For data gathered on a Brookfield Viscometer, and processed by a direct application of the method of Krieger,[7] Pierce[3] has pointed out that the value of η_∞ calculated using the Casson model [equation (3)] may be somewhat higher than the actual high shear viscosity obtained with a high shear viscometer. Unlike the high shear extrapolated η_∞ which is shear independent, the η_∞ obtained from extrapolated low shear Brookfield data can only be approximated as shear independent. Although this limitation must be kept in mind when treating the Brookfield η_∞ as a quasi-Newtonian parameter, it should not detract from the usefulness of the "high shear" viscosity thus obtained. To distinguish between these two cases, the authors suggest the symbol η_c and a return to the term "Casson Viscosity" which was used by Duck[4] and is a contraction of "Casson Plastic Viscosity" used as early as 1958 by Steiner.[5] Thus, for use with a low shear viscometer, equation (3) may be written as

$$\tau^{\frac{1}{2}} = \eta_c^{\frac{1}{2}}\dot{\gamma}^{\frac{1}{2}} + \tau_0^{\frac{1}{2}} \tag{4}$$

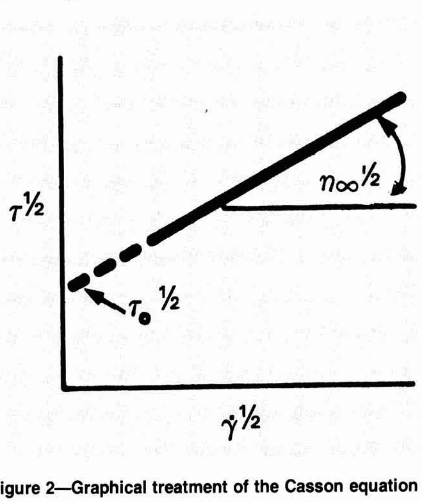


Figure 2—Graphical treatment of the Casson equation

Asbeck has noted that the Casson equation is the simplest two constant viscosity-shear rate equation with parameters that can be related to coating composition.[1,6] Pierce also found the Casson equation useful[3] for applied work on most coatings, since he too was able to relate the two parameters η_c and τ_0 to product performance and composition variables. To measure the resistance to sagging of a paint film, τ_0 can be used and is related to the maximum film thickness that a vertical wall can hold without sagging.[8] In noncoatings applications, such as emulsions or dispersions, τ_0 may be used as a measure of the resistance to separation of the dispersed phase while the value of η_c can be employed as an index of flowability at "high shear" rate.

In many industrial situations, certain non-Newtonian fluids, and in particular highly filled dispersions, require rehological characterization over a range of shear rate. This is necessary in order to obtain a broader picture of their flow properties than a one or two point measurement would provide. It is often the case that many samples need to be run to routinely assess the effects of formulation, process, or aging changes as well as to determine quality control parameters. While the method of Krieger[7] can be employed successfully in these cases to calculate accurate shear rates (which can then be used to generate Casson plots[3]), it has been found in practice that simple plotting of Brookfield dial reading vs. rpm often results in an approximate straight line when plotted on square root dial reading-square root rpm coordinates. This is not surprising in view of the fact that the dial reading is proportioned to shear stress and the shear rate can be shown to be an approximately linear function of rpm over the narrow shear rate range available with this instrument. We believe this approach to be acceptable for comparisons where the high shear viscosity varies by a factor of about 2.

In the routine cases discussed above, we have found it quite useful to prepare square root dial reading-square root rpm paper specifically for the four models of the Brookfield Viscometer. Raw data plotted on these graphs can be used to obtain a slope and extrapo-

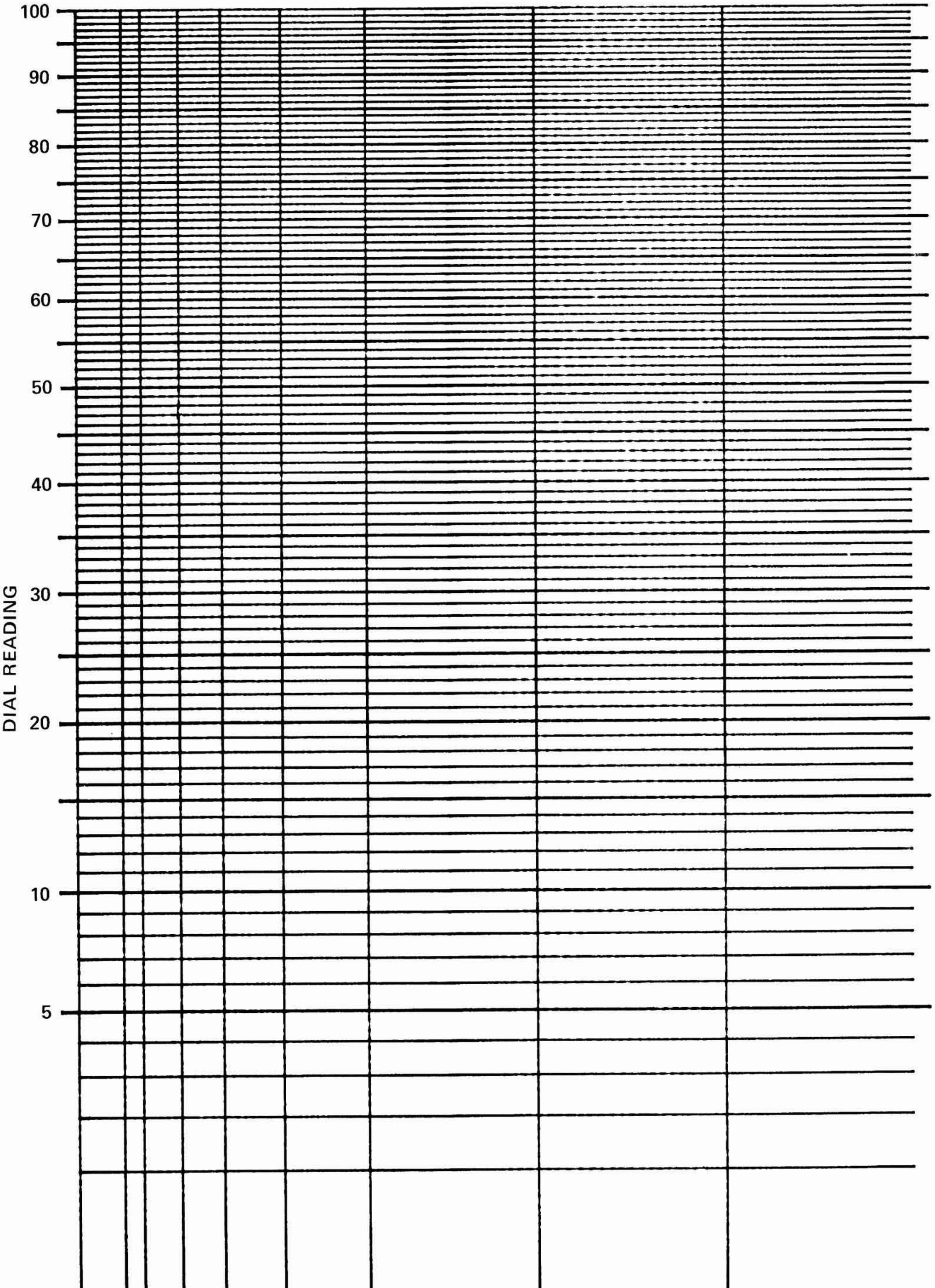


Figure 3—Casson graph paper for Brookfield Synchro-Lectric Viscometer - Model LV

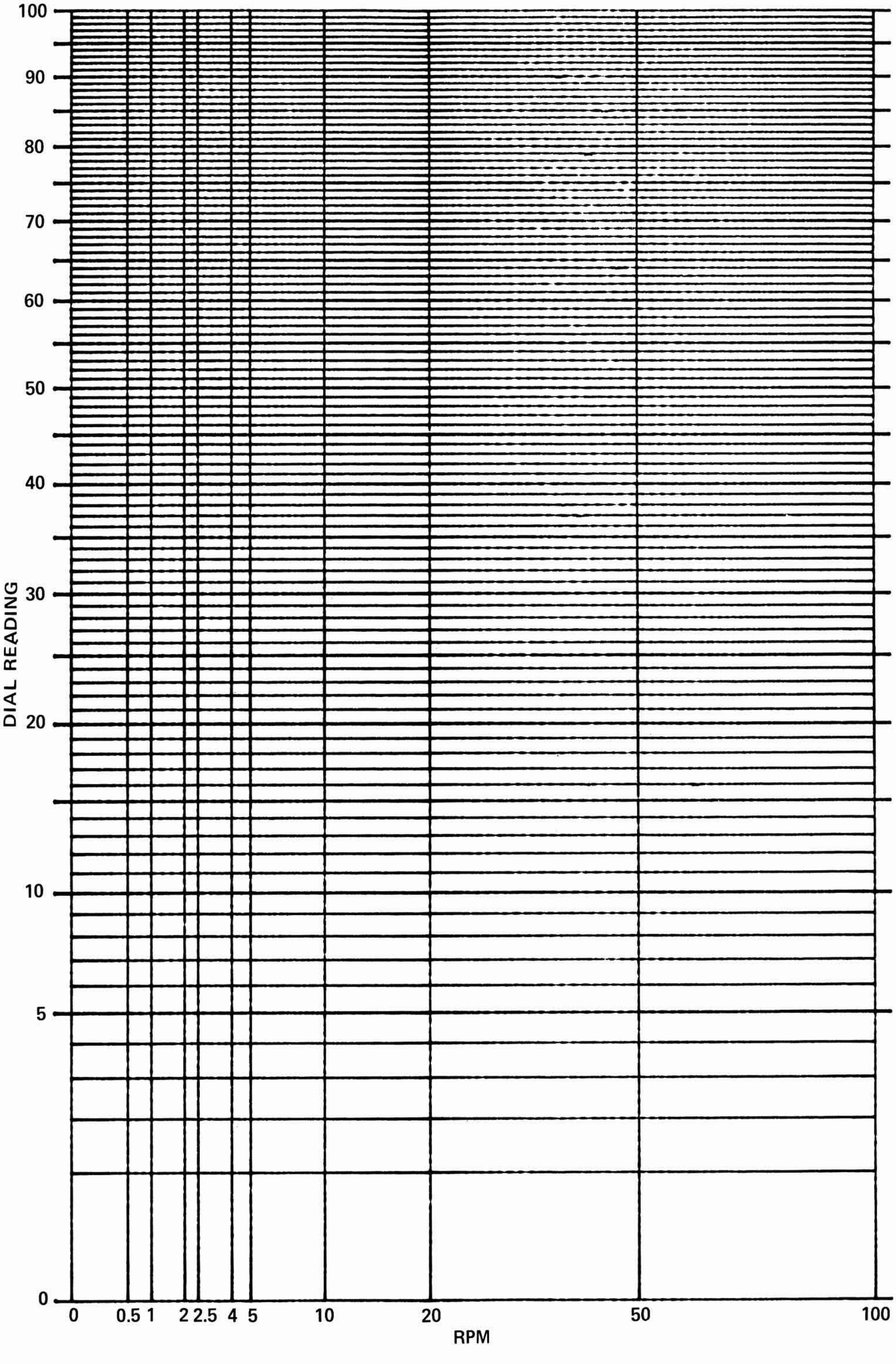


Figure 4—Casson graph paper for Brookfield Synchro-Lectric Viscometer - Models RV, HA, HB

lated intercept. Using the template method described herein, two parameters can be generated which are empirical analogs to the accurate yield stress and high shear viscosity. While the two parameters so generated are not rheologically exact, they are easy to obtain using the raw data and can be usefully interpreted according to the Casson model.

The present method is based on the use of cylindrical spindles rotating in a cup which has a radius sufficiently large that an approximation of an infinite sea of fluid may be used.[9] In practical terms, a standard 400 cc beaker is adequate. The method differs from the approach of Duck,[4] in which a nomograph was designed for use with a concentric cylinder attachment for a Brookfield HBT viscometer. Duck also points out his method can be applied to any concentric cylinder viscometer.

TEMPLATE METHOD

Special Graph Paper

Figure 3 shows the square root dial reading-square root rpm paper used for the Brookfield Model LV and *Figure* 4 is for the Models RV, HA and HB. Two graphs are required because the spindle speeds are different for the various models. These graph papers can be easily obtained by means of a computer program (*Figure* 5) which will draw the graph papers using a standard plotter attachment for a time share computer.

The Template

Having plotted the dial reading-rpm data directly onto the appropriate graph paper, a special template (*Figure* 6) is used to obtain the empirical analog to Casson viscosity. This template may be obtained by making a transparency of the figure generated using the computer program shown in *Figure* 7. The template is generated using the relationships derived below.

When a Newtonian fluid is measured on a Brookfield viscometer, a plot of dial reading (a function of shear stress) vs. rpm (a function of shear rate) produces a straight line which passes through the origin (*Figure* 8). The angle φ can be expressed as

$$\tan \varphi = \sqrt{\frac{S}{\text{rpm}}} \qquad (5)$$

or

$$(\tan \varphi)^2 = \frac{S}{\text{rpm}} \qquad (6)$$

When used with Newtonian fluids, the Brookfield Synchro-Lectric Viscometer is utilized with a set of factors which are multiplied by the dial reading to obtain the dial viscosity, η_B

$$\eta_B = \text{Factor} \cdot S \qquad (7)$$

For each spindle and speed there is a different factor. It can be shown, by plotting the log of the factor vs. the log of the rpm, that the following exact relation holds for each spindle (cylindrical or not) and all four viscometer models:

$$(\text{Factor})\ (\text{rpm}) = \text{Constant} = K^* \qquad (8)$$

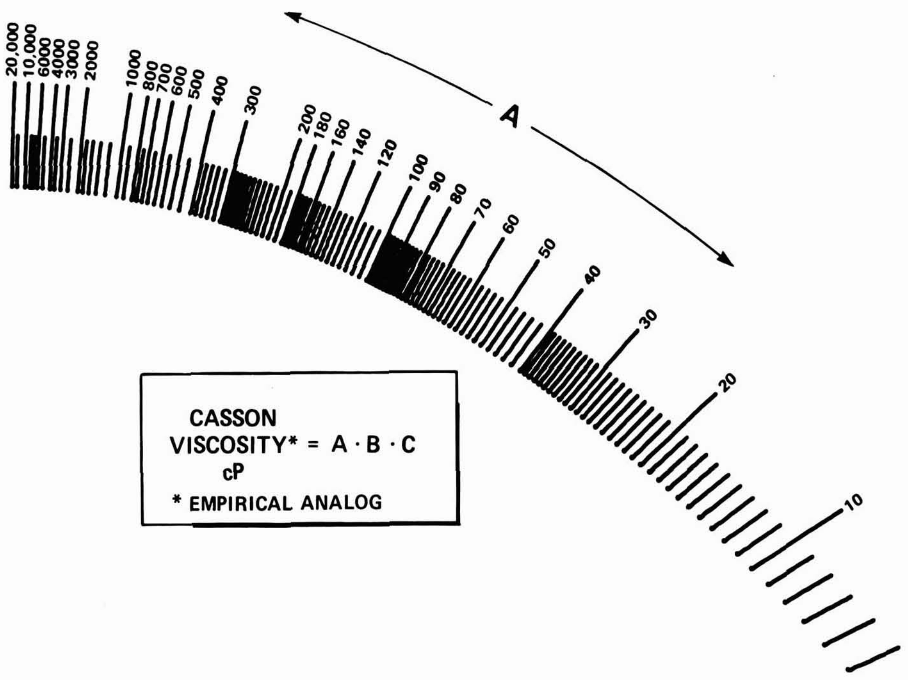
```
PLSQRT
1010C      PURPOSE: TO DRAW A SPECIAL GRAPH PAPER FOR PLOTTING DATA
1020C          FROM A BROOKFIELD VISCOMETER UTILIZING THE CASSON
1030C          EQUATION.  BOTH AXES ARE ON A SQUARE ROOT SCALE.
1040C          THE VERTICAL AXIS IS USED FOR THE BROOKFIELD DIAL
1050C          READING AND IS SCALED 0(5)100.  THE HORIZONTAL
1060C          AXIS IS MARKED FOR THE APPROPRIATE RPM VALUES.
1070C
1080C      USAGE: FORMAT IS KEYED TO A H/P PLOTTER.  FORMAT 60 TURNS
1090C          THE PLOTTER ON.  FORMAT 70 TURNS THE PLOTTER OFF.
1100C          FORMAT 80 PRINTS X-Y PLOTTING PAIRS SCALED FROM ZERO
1110C          TO THE VALUE OF SCALE.
1120C
1130C      ****************************************
1140C      *****  PLOTTER AXES MUST BE EXACTLY  *****
1150C      *****     THOSE USED WITH CASSON     *****
1160C      ****************************************
1170C
1180C      VARIABLES: IO IS THE OUTPUT DEVICE
1190C                 IR IS THE SUBSCRIPT IDENTIFYING THE SELECTED
1200C                    RPM RANGE.  1=0.3-60.0; 2=0.5-100.0
1210C                 SCALE SETS THE MAXIMUM VALUE OF THE PLOTTER
1220C                    OUTPUT
1230C                 RPM IS AN ARRAY CONTAINING TWO SETS OF RPM
1240C                    VALUES
1250C
1260       DIMENSION RPM(11,2),IP(3)
1270       DATA RPM/999.,0.,0.3,0.6,1.5,3.,6.,12.,30.,60.,100.,
1280&      0.,0.5,1.,2.,2.5,4.,5.,10.,20.,50.,100.0/
1290       IO=66
1300       IR=1
1310       SCALE=9999.
1320C
1330C          CALCULATE & PRINT HORIZONTAL LINES
1340C
1350       SC1=SCALE/10.0
1360       ISA=IFIX(0.02*SCALE+0.5)
1370       ISB=IFIX(SQRT(0.2)*SCALE+0.5)
1380       ISC=IFIX(0.98*SCALE+0.5)
1390       ISD=IFIX(0.5*SCALE+0.5)
1400       IP(2)=ISB
1410       DO 50 I=1,101
1420       KK=0
1430       IY=IFIX(SC1*SQRT(FLOAT(I-1))+.5)
1440       WRITE(IO,80)
1450       X=0.0
1460       IP(1)=ISA
1470       IP(3)=ISC
1480       IF(MOD(I,5).NE.1) GOTO 30
1490       IF(MOD(I,100).EQ.1) GOTO 20
1500    10 KK=KK+1

1510       IY=IY+((-2)**KK)
1520    20 IP(1)=0
1530       IP(3)=IFIX(SCALE)
1540       IF(KK.EQ.2) WRITE(IO,80)
1550    30 DO 40 J=1,3
1560       WRITE(IO,100) IP(J),IY
1570    40 CONTINUE
1580       WRITE(IO,90)
1590       IF(KK.EQ.1) GOTO 10
1600    50 CONTINUE
1610C
1620C          CALCULATE & PRINT VERTICAL LINES
1630C
1640       SC2=0.96*SCALE/SQRT(RPM(11,IR))
1650       IP(1)=0
1660       IP(2)=ISD
1670       IP(3)=IFIX(SCALE)
1680       DO 70 I=1,11
1690       IF(RPM(I,IR).GT.100.0) GOTO 70
1700       IF((I.EQ.11).AND.(IR.EQ.1)) GOTO 70
1710       IX=IFIX(SC2*SQRT(RPM(I,IR))+0.5)+ISA
1720       WRITE(IO,80)
1730       DO 60 J=1,3
1740       WRITE(IO,100) IX,IP(J)
1750    60 CONTINUE
1760       WRITE(IO,90)
1770    70 CONTINUE
1780       STOP
1790    80 FORMAT(4HPLTL)
1800    90 FORMAT(4HPLTT)
1810   100 FORMAT(2I5)
1820       END
```

Figure 5—Computer program to draw Casson graph paper for Brookfield Viscometer

K* is easily obtained since it is the value of the intercept at an rpm of 1.0. This equation has been used as the basis of a Viscosity Calculator Slide Rule for all Brookfield Viscometers.[10]

For purposes of this work, it is desired to generate values of K* only for the set of cylindrical LV spindles when used with each of the four viscometer models LV, RV, HA, and HB. Using the factors listed in reference (11), values of K* can be calculated for the LV spindles when attached to the model LV viscometer (*Table* 1). To calculate the K* values for the set of cylindrical LV spindles when attached to the RV, HA, and HB viscometers, a different set of factors is employed, and these constants are available from the manufacturer.[12] *Table* 1 summarizes all of the K* values. If desired, the factors may be regenerated using equation (8).



		C				
		CYLINDRICAL SPINDLE				
MODEL	B	1	2	3	4	5
LV	1	1	5	20	100	200
RV	13	1	4.295	16.539	82.051	164.103
HA	26	1	4.295	16.539	82.051	164.103
HB	104	1	4.295	16.539	82.051	164.103

Figure 6—Template for use with Casson graph paper and Brookfield Viscometer

Combining equations (7) and (8)

$$\eta_B = \frac{K^*}{\text{rpm}} \cdot S \qquad (9)$$

or

$$S = \frac{\eta_B}{K^*} \cdot \text{rpm} \qquad (10)$$

Combining (6) and (7)

$$\eta_B = K^* (\tan \varphi)^2 \qquad (11)$$

A general Casson fluid can be represented as shown in *Figure* 9. By analogy with equation (11) the shear independent Casson viscosity η_c can be expressed as

$$\eta_c = K^* (\tan \Theta)^2 \qquad (12)$$

Equation (12) allows calculation of the slope of the lines in the template. Inserting K* into equation (12) for each viscometer model one obtains

$$\text{Model LV: } \eta_c = 60 (\tan \Theta)^2 \qquad (13)$$

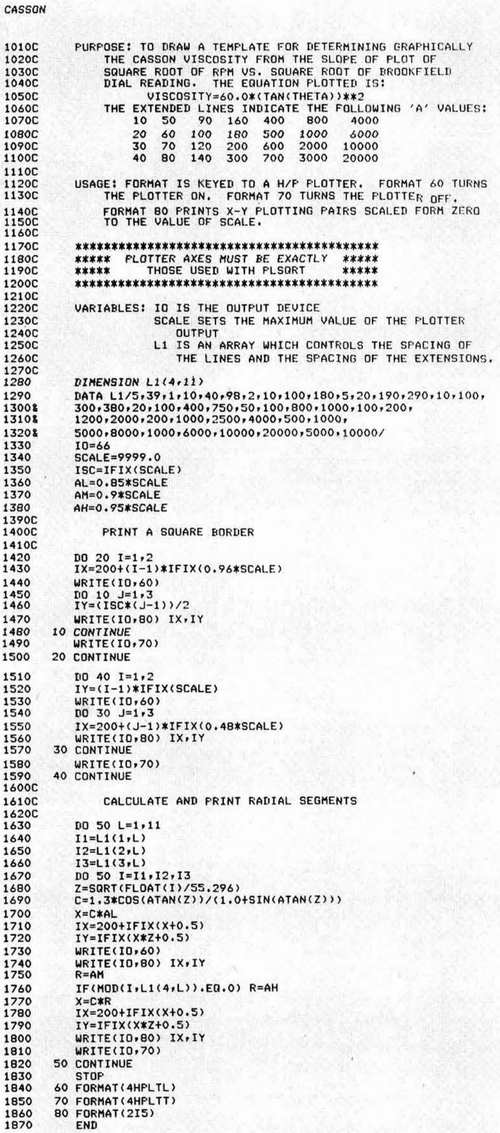
$$\text{Model RV: } \eta_c = 780 (\tan \Theta)^2 \qquad (14)$$

$$\text{Model HA: } \eta_c = 2(780) (\tan \Theta)^2 \qquad (15)$$

$$\text{Model HB: } \eta_c = 8(780) (\tan \Theta)^2 \qquad (16)$$

It will be noticed that the multiplicative constants of $(\tan \Theta)^2$ can be related to each other by normalizing these with respect to the model LV value. Dividing each by 60, the parameter B is obtained for the four models and these are summarized in *Table* 2.

To determine the A value shown in the template, equation (12) is used and the K* value is chosen to be



```
CASSON

1010C      PURPOSE: TO DRAW A TEMPLATE FOR DETERMINING GRAPHICALLY
1020C          THE CASSON VISCOSITY FROM THE SLOPE OF PLOT OF
1030C          SQUARE ROOT OF RPM VS. SQUARE ROOT OF BROOKFIELD
1040C          DIAL READING.  THE EQUATION PLOTTED IS:
1050C                VISCOSITY=60.0*(TAN(THETA))**2
1060C          THE EXTENDED LINES INDICATE THE FOLLOWING 'A' VALUES:
1070C              10  50   90  160  400   800   4000
1080C              20  60  100  180  500  1000   6000
1090C              30  70  120  200  600  2000  10000
1100C              40  80  140  300  700  3000  20000
1110C
1120C      USAGE: FORMAT IS KEYED TO A H/P PLOTTER.  FORMAT 60 TURNS
1130C          THE PLOTTER ON.  FORMAT 70 TURNS THE PLOTTER OFF.
1140C          FORMAT 80 PRINTS X-Y PLOTTING PAIRS SCALED FORM ZERO
1150C          TO THE VALUE OF SCALE.
1160C
1170C      ****************************************
1180C      *****  PLOTTER AXES MUST BE EXACTLY  *****
1190C      *****     THOSE USED WITH PLSQRT     *****
1200C      ****************************************
1210C
1220C      VARIABLES: IO IS THE OUTPUT DEVICE
1230C                 SCALE SETS THE MAXIMUM VALUE OF THE PLOTTER
1240C                    OUTPUT
1250C                 L1 IS AN ARRAY WHICH CONTROLS THE SPACING OF
1260C                    THE LINES AND THE SPACING OF THE EXTENSIONS.
1270C
1280       DIMENSION L1(4,11)
1290       DATA L1/5,39,1,10,40,98,2,10,100,180,5,20,190,290,10,100,
1300&      300,380,20,100,400,750,50,100,800,1000,100,200,
1310&      1200,2000,200,1000,2500,4000,500,1000,
1320&      5000,8000,1000,6000,10000,20000,5000,10000/
1330       IO=66
1340       SCALE=9999.0
1350       ISC=IFIX(SCALE)
1360       AL=0.85*SCALE
1370       AM=0.9*SCALE
1380       AH=0.95*SCALE
1390C
1400C          PRINT A SQUARE BORDER
1410C
1420       DO 20 I=1,2
1430       IX=200+(I-1)*IFIX(0.96*SCALE)
1440       WRITE(IO,60)
1450       DO 10 J=1,3
1460       IY=(ISC*(J-1))/2
1470       WRITE(IO,80) IX,IY
1480    10 CONTINUE
1490       WRITE(IO,70)
1500    20 CONTINUE
1510       DO 40 I=1,2
1520       IY=(I-1)*IFIX(SCALE)
1530       WRITE(IO,60)
1540       DO 30 J=1,3
1550       IX=200+(J-1)*IFIX(0.48*SCALE)
1560       WRITE(IO,80) IX,IY
1570    30 CONTINUE
1580       WRITE(IO,70)
1590    40 CONTINUE
1600C
1610C          CALCULATE AND PRINT RADIAL SEGMENTS
1620C
1630       DO 50 L=1,11
1640       I1=L1(1,L)
1650       I2=L1(2,L)
1660       I3=L1(3,L)
1670       DO 50 I=I1,I2,I3
1680       Z=SQRT(FLOAT(I)/55.296)
1690       C=1.3*COS(ATAN(Z))/(1.0+SIN(ATAN(Z)))
1700       X=C*AL
1710       IX=200+IFIX(X+0.5)
1720       IY=IFIX(X*Z+0.5)
1730       WRITE(IO,60)
1740       WRITE(IO,80) IX,IY
1750       R=AM
1760       IF(MOD(I,L1(4,L)).EQ.0) R=AH
1770       X=C*R
1780       IX=200+IFIX(X+0.5)
1790       IY=IFIX(X*Z+0.5)
1800       WRITE(IO,80) IX,IY
1810       WRITE(IO,70)
1820    50 CONTINUE
1830       STOP
1840    60 FORMAT(4HPLTL)
1850    70 FORMAT(4HPLTT)
1860    80 FORMAT(2I5)
1870       END
```

Figure 7—Computer program to draw the template

that for the cylindrical LV1 spindle when attached to the model LV viscometer. This choice is essential because it provides the means for a convenient transition to all of the other spindles and viscometer models as shown below.

For the LV1 cylindrical spindle and any of the four model viscometers (LV, RV, HA, HB) the Casson viscosity is simply obtained by multiplying the template value of A by the appropriate value of B obtained from *Table* 2.

$$\eta_c = A \cdot B \tag{17}$$

To extend the templates utility to all of the cylindrical spindles, the relationship among the spindles must be incorporated into equation (17). It should be pointed out that the model LV is routinely supplied only with cylindrical spindle LV-1 and LV-4. Cylindrical spindles LV-2, LV-3, and LV-5 are available, however, from the manufacturer. For the remaining discussion, this set of spindles will simply be referred to as cylindrical spindles #1, 2, 3, 4, and 5. If K* (see *Table* 1) is normalized with respect to the K* value of the #1 cylindrical spindle and a similar calculation is performed for the RV, HA, and HB models, it is observed that two relationships exist—one for the LV model and one for the RV, HA, and HB models. Calling the normalized value C, one obtains the values listed in *Table* 3. From the information above it can be seen that equation (17) may be expanded to:

$$\eta_c = A \cdot B \cdot C \tag{18}$$

Equation (18) is the final relationship and allows the template in *Figure* 6 to be used for any of the five cylindrical spindles when attached to any of the four viscometer models.

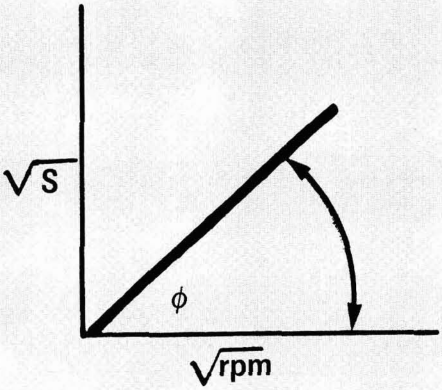


Figure 8—Casson plot for Newtonian fluid

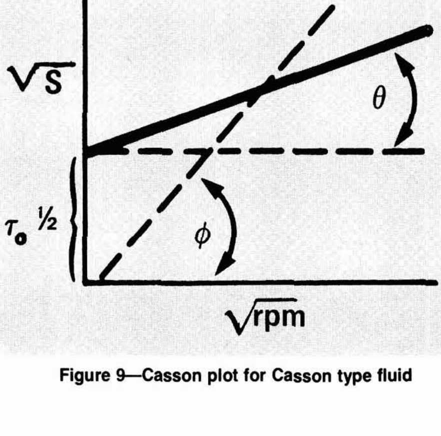


Figure 9—Casson plot for Casson type fluid

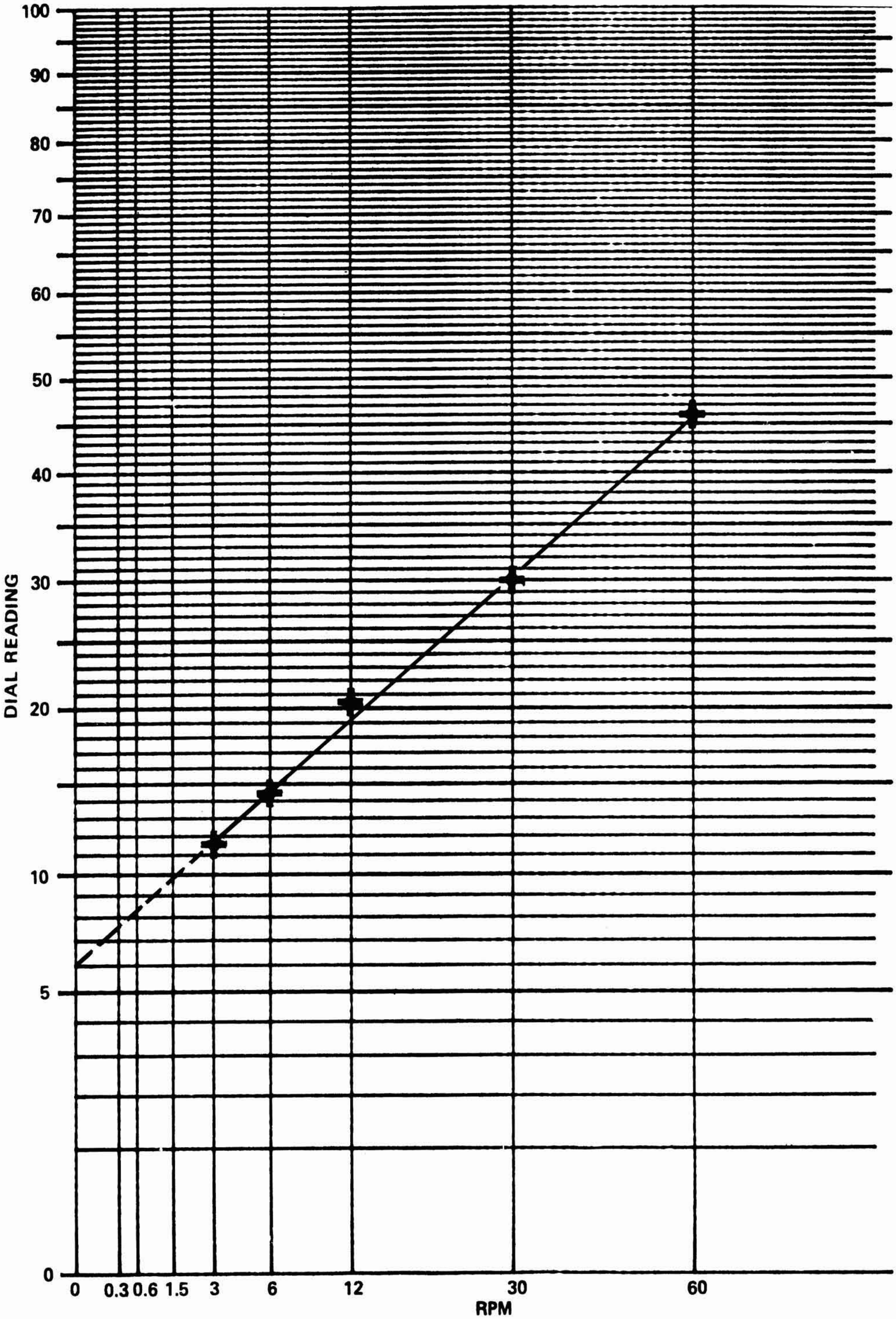


Figure 10—Example of typical Casson fluid

Determination of the Yield Stress Analog

For cylindrical spindles in a cup of large diameter, the empirical yield stress analog may be calculated from:

$$\tau_0 = \alpha \cdot So \qquad (19)$$

where So is the dial reading at 0 rpm obtained by extrapolation of the Casson plot. The constant, α, can be determined by

$$\alpha = \frac{\text{Full Scale Torque}/100}{2\pi\,(\text{Radius})^2\,(\text{Effective Length})} \qquad (20)$$

The effective length is the actual spindle length plus a correction factor for end effects. *Table* 4 lists the full scale torque for each viscometer model.[13] *Table* 5 lists the radius and effective length for each cylindrical spindle. *Table* 6 lists the calculated values of α for use with equation (19).

USING THE TEMPLATE AND GRAPH PAPER: AN EXAMPLE

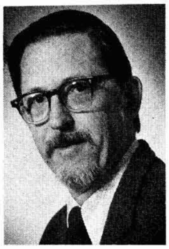
The following data were obtained on a slurry of organic polymer in a nonsolvent with cylindrical spindle 3 attached to the model LV viscometer.

RPM	3	6	12	30	60
DIAL READING	11.5	14.5	20.5	30	46

The data are plotted in *Figure* 10 and the intercept, as read directly from the figure, is 6.0. Yield stress is calculated by means of equation (19) using the appropriate constant for cylindrical spindle 3.

$$\tau_0 = (2.56)\,(6.0)$$
$$\tau_0 = 15.4 \text{ dyn/cm}^2$$

MEYER R. ROSEN received both his B.S. and M.S. Degrees in Chemical Engineering from The Polytechnic Institute of Brooklyn. In 1966 he joined Union Carbide Corp. and is currently a Project Scientist in the R&D Department. His areas of interests include the surface and physical chemistry of surfactants, foams, emulsions, and coupling agents. He is currently involved with new applications development for water soluble polymers.



WILLIAM W. FOSTER received the B.S. Degree in Engineering from Trinity College and the B.S. Degree in Chemical Engineering from Rensselaer Polytechnic Institute. He has been with Union Carbide Corp. since 1954 and is currently a Senior Research Scientist in the R&D Department. He has done work in silicone monomers and polymers and in the design and analysis of experiments. He currently heads a group responsible for applied statistics.

Table 1—Summary of K* Values

Cylindrical Spindles	Model LV	Model RV	Model HA	Model HB
1	60	780	1560.	6240.
2*	300	3,350	6700	26,800
3*	1,200	12,900	25,800	103,200
4	6,000	64,000	128,000	512,000
5*	12,000	128,000	256,000	1,024,000

---

*Not part of the standard set supplied with the LV Viscometer but available from the manufacturer.

Table 2—"B" Values

Viscometer Model	"B"
LV	1
RV	13
HA	26
HB	104

Table 3—"C" Values

Cylindrical Spindle	"C" Model LV	Models RV, HA, HB
1	1	1.000
2	5	4.295
3	20	16.539
4	100	82.051
5	200	164.103

Table 4—Full Scale Torque

Model	Full Scale Torque, dyn-cm
LV	673.7
RV	7187.0
HA	14,374.0
HB	57,496.0

Table 5—Radii and Effective Lengths of Spindles[14]

Cylindrical Spindle	Radius, cm	Effective Length, cm
1	0.942	7.49
2	0.513	6.12
3	0.294	4.85
4	0.159	3.40
5	0.159	1.68

Table 6—α Values

Cylindrical Spindle	Model LV	RV	HA	HB
1	0.16	1.72	3.44	13.78
2	0.67	7.11	14.21	56.85
3	2.56	27.30	54.60	218.39
4	12.48	133.14	266.28	1065.14
5	25.26	269.45	538.91	2155.63

The Casson viscosity analog is obtained by placing point (X) of the template (*Figure* 6) over the intercept (at zero rpm and a dial reading of 6.0). After lining the template up with the X and Y axis, obtain the constant A which corresponds with the experimental line. In this example, the constant A was 18.5. The value of B for model LV is 1.0 (*Table* 2) and the value of C for model LV and cylindrical spindle 3 is 20 (*Table* 3). Therefore,

$$\eta_c = A \cdot B \cdot C = (18.5)\,(1)\,(20) = 370 \text{ cP}$$

CONCLUSIONS

Characterization of the rheological properties of non-Newtonian fluids which follow the two parameter Casson flow equation is discussed. When used in conjunction with a low shear Brookfield Synchro-Lectric Viscometer, this rheological model is a powerful tool for many industrial problems where accurate shear rate measurements are not required.

A simple template method is presented which allows rapid determination of useful empirical analogs for Casson viscosity and yield stress. The method relies on a set of five cylindrical spindles used in conjunction with any of the four standard Brookfield viscometer models. Computer programs are given which will plot both the template and special graph papers required.

ACKNOWLEDGMENTS

The authors thank Union Carbide Corp. for permission to publish this paper.

The authors also thank the referees for their constructive criticisms and comments.

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β-Hydroxyalkylamides, Low Polluting Crosslinkers For Carboxyl Containing Polymers

James Lomax and Graham Swift
Rohm and Haas Company*

β-Hydroxyalkylamide crosslinkers make possible the preparation of water-borne thermosetting coatings which evolve no noxious reaction by-products and which have energy saving potential relative to other water-borne baking enamels because they require only very low levels of organic cosolvent in formulation.

β-Hydroxyalkylamides crosslink the carboxyl groups of water-soluble polymers. Synthesis and characterization of several of these crosslinkers was completed. One of them, bis [N,N-di (β-hydroxypropyl)] a dipamide, has been extensively tested as a crosslinker for acrylic colloidal dispersions in formulations for industrial baking applications. These easily formulated enamels provide a cure response comparable to conventional amino-resin crosslinked water-borne coatings and film properties useful for appliance coatings and general product finishing applications.

INTRODUCTION

A comparison of aqueous and solvent-borne thermoset coatings reveals that water-based systems offer several advantages, including low flammability, low toxicity and odor, reduced pollution, easy clean-up with water, and lower energy requirements.

The lower energy requirement for baking aqueous coatings is probably the least apparent of these advantages. Lower energy utilization is achieved because no afterburner is required to incinerate solvent and because safety engineers recommend that 10,000 ft^3 ($300m^3$) of make-up air be added to a typical baking oven for each gallon of organic solvent added with the coating. This volume of air maintains the atmosphere in the baking oven below the explosion limit. Since aqueous coatings contain much less solvent than their solvent-borne counterparts, much less energy is required with a very substantial saving in energy input.

Coatings formulated with novel water-soluble β-hydroxyalkylamide crosslinkers and acrylic colloidal dispersions require very low levels of organic solvent and release only water as a by-product. They are, therefore, considered to be both energy efficient and low-polluting thermosetting acrylic coatings.

EXPERIMENTAL

Synthesis of β-Hydroxyalkylamides

The general procedure outlined by the equation in *Figure* 1 was used for the synthesis of the mono- and polyfunctional β-hydroxyalkylamides described in this paper. *Table* 1 contains the assignments for R, R′, R″ and R‴ to produce the model monofunctional compounds based on 4-pentenoic acid. The polyfunctional experimental crosslinkers obtained from adipic and azelaic esters are shown in *Figure* 5.

The β-hydroxyalkylamides described were generally prepared at 75°C by the gradual addition of a stoichiometric amount of an alkyl ester to alkanolamine and basic catalyst, over two hours. By-product alcohol was continuously removed throughout the reaction by the application of a partial vacuum. A nitrogen atmosphere was maintained to avoid product discoloration. The product, 80-90% β-hydroxyalkylamide, was generally used in crosslinking reactions without purification, either as prepared or in aqueous solution. Impurities were starting materials, β-amino-alkyl ester, and esteramide, the latter being polymeric when polyfunctional β-hydroxyalkylamides were prepared.

To obtain material suitable for the rate studies described below, a two-fold excess of the alkanolamine was used in the synthesis to minimize esteramide formation. Contaminating basic compounds were then removed from the β-hydroxyalkylamide by dissolving the crude product in methanol at 50% by weight and stirring for a few minutes at 25°C with a two-fold excess of strong acid macroreticular ion exchange resin.† The calculated excess of strong acid was based on the determined (aqueous titration) basicity of the crude product. Filtration and removal of methanol at 150-200 mm Hg (200-270 kp) pressure and ≤ 50°C gave a product,

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Presented by Dr. Swift at the 55th Annual Meeting of the Federation of Societies for Coatings Technology in Houston, Tex., October 28, 1977.

†Resin used was Amberlyst® 15, Rohm and Haas Co.

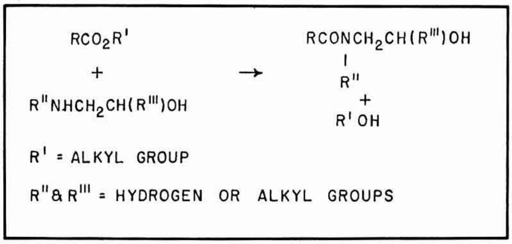

Figure 1—General synthesis of β-hydroxyalkylamides

β-hydroxyalkylamide, ≥ 99% purity based on infrared spectrum and titration for amide.[1]

Esterification Reactions of β-Hydroxyalkylamides

The esterification reactions reported in this paper were done either to compare the reactivity of hydroxy groups (can be primary or secondary) in purified model β-hydroxyalkylamides which are themselves secondary or tertiary amides, or to characterize crude polyfunctional β-hydroxyalkylamide crosslinkers.

For the comparison of reactivities, a known quantity of purified β-hydroxyalkylamide was mixed with a known weight of suitable carboxylic acid (octanoic or palmitic have been employed in this work) and heated at the designated temperature at 50% by weight solids in an aromatic solvent with azeotropic removal of water. Acidity loss, as esterification proceeded, was followed by titration of "stopped" reaction aliquots withdrawn at various time intervals. As carboxylic acid was used in excess, the time to reach a minimum acidity was taken as a measure of reactivity for that particular β-hydroxyalkylamide. Comparison of reactivities were expressed graphically as reported in this paper.

By the same procedure, the reactivity of crude β-hydroxyalkylamide crosslinkers was established to facilitate formulation into enamels. Through a knowledge of the weight of crude crosslinker, and loss in acidity of the reactant, β-hydroxyalkylamide purity is simply expressed by the relationship:

$$\%\ \text{Purity} = 100 \times \frac{\text{Determined acid loss/g of crude } \beta\text{-hydroxyalkylamide}}{\text{Calculated acid loss for pure } \beta\text{-hydroxyalkylamide}}$$

$$\text{Reactivity} = [\text{Purity of Crosslinker}] \times [\text{Theoretical Functionality}]$$

Note: It was established that free amine does not interfere with this procedure, amidation of acid is very slow compared to esterification with β-hydroxyalkylamide at the temperatures studied in this work.

Table 1—Synthesis of 4-Pentenamides

R	R'	R''	R'''	Product
$CH_2 = CHCH_2CH_2$-	C_2H_5	H	H	N (β-hydroxyethyl)-4-pentenamide
$CH_2 = CHCH_2CH_2$-	C_2H_5	CH_3	H	N-methyl-N (β-hydroxyethyl)-4-pentenamide

Table 2—Gloss White Enamel Based on QM-606 Crosslinker And Acrylic Colloidal Dispersion Resin

	Parts by Weight	
Ball Mill Grind		
TiO_2 (TiPure® R-902)	480.0	480.0
Acrylic Resin A[a] at 39.8% in water	120.6	—
Acrylic Resin B[b] at 36.5% in water	—	131.5
Dimethylaminoethanol	4.0	3.6
2-Butoxyethanol	41.4	41.4
Water	151.0	140.5
Defoamer	3.0	3.0
	800.0	800.0
Letdown		
Grind	266.7	266.7
Acrylic Resin A	460.8	—
Acrylic Resin B	—	502.5
Dimethylaminoethanol	18.5	18.4
2-Butoxyethanol	3.5	3.5
Water	170.9	129.3
QM-606 at 51.0% in water[c]	79.6	79.6
	1,000.0	1,000.0
Constants		
% Total solids	40.0	40.0
TiO_2/Binder	40/60	40/60
Acrylic Resin/QM-606, solids basis	83.1/16.9	83.1/16.9
Equivalents polymer acid/Eq crosslinker	1.0/1.0	1.0/1.0
pH	8.1	8.1
Viscosity, #4 Ford Cup, secs.	25	28
Volatiles, by weight	94% Water/ 6% Organic	94% Water/ 6% Organic

TiPure is a registered trademark of E.I. duPont de Nemours & Co., Inc.

(a) Acrylic Resin A (acid no. = 98 mg KOH/g polymer) is sold in a dimethylaminoethanol neutralized form as Acrysol WS-68.

(b) Acrylic Resin B is an experimental resin similar to Resin A except synthesized from a more hydrophobic monomer balance.

(c) QM-606 is 80% active and added as a 51% solids solution in water.

Formulations

One of the β-hydroxyalkylamide crosslinkers, bis [N,N-di (β-hydroxypropyl)] adipamide which has the product code QM-606 has been evaluated as a crosslinker for two alkali-soluble acrylic colloidal dispersion resins in white gloss enamels for spray application. The formulations are shown in *Table* 2. The acrylic resins are both acid functional with acid numbers equal to 98 mg KOH/g polymer. Acrylic Resin A is an unneutralized form of a commercially available colloidal dispersion resin.* Acrylic Resin B is an experimental resin of the same type but synthesized from a more hydrophobic monomer balance.

DISCUSSION

The relative ease of uncatalyzed esterification of carboxylic acids with β-hydroxyalkylamides has been known for some years.[2-5] We have extended this facile

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*Acrysol® WS-68, Rohm and Haas Co.

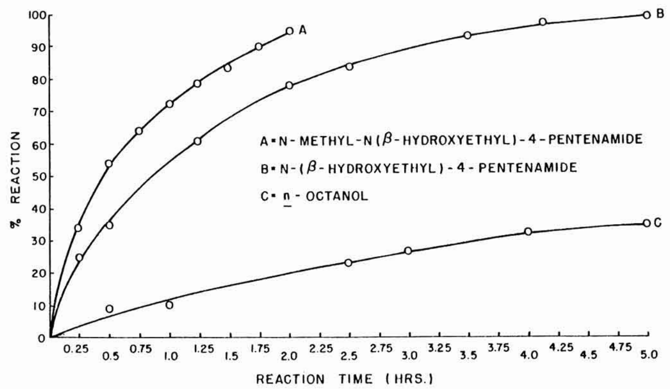

Figure 2—Esterification on *n*-octanoic acid with a series of alcohols at −140°C

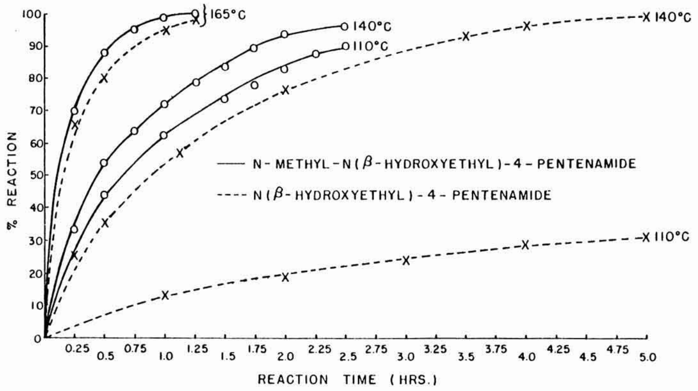

Figure 3—Effect of temperature on the rate of esterification of *n*-octanoic acid with secondary and tertiary β-hydroxyethyl amides

reaction to the crosslinking of carboxylated acrylic resins in general and to water soluble dispersions in particular.

Choice of β-Hydroxyalkylamide Functionality

As can be seen from *Figure* 1, the amide group of the β-hydroxyalkylamides under consideration can be secondary or tertiary, R″ can be hydrogen or an alkyl substituent; and the hydroxyl functionality can be primary or secondary, R‴ can be hydrogen or alkyl, respectively. The relative reactivities of primary hydroxy groups in secondary and tertiary β-hydroxyalkylamides towards esterification at 140°C are shown in *Figure* 2 using *n*-octanoic acid and 4-pentenamides as model compounds.

The tertiary β-hydroxyalkylamide is faster reacting than the secondary, and both are superior to the conventional alcohol in the absence of catalyst.

Figure 3 indicates the temperature effect on the same esterifications described above. Increasing temperature promotes the equalization of reactivity of secondary and tertiary β-hydroxyalkylamides. The reactivity difference at lower temperature is hypothesized as being due to the greater thermal stability of the substituted oxazoline esterification intermediate (*Figure* 4) obtained from secondary β-hydroxyalkylamides. Oxazolines are isolatable materials and are known to undergo facile ring opening reactions with carboxylic acids at temperatures in excess of 150°C.

Since we are interested in reducing cure temperature, tertiary β-hydroxyalkylamide is obviously the crosslinking functionality of choice (when considering primary hydroxy functionality).

The nonavailability of a suitable alkanolamine to prepare a monofunctional β-hydroxyalkylamide with secondary hydroxy and tertiary amide functionality precluded its evaluation at this point. However, this comparison is addressed with the polyfunctional crosslinker studies *(vide infra)*.

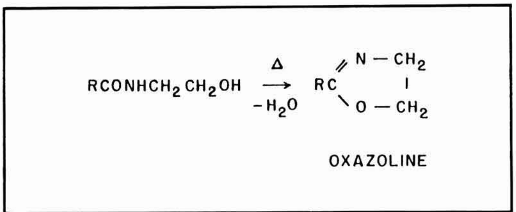

Figure 4—Oxazoline formation from secondary β-hydroxyalkylamides

Table 3—Polyfunctional β-Hydroxyalkylamides

$R(CO_2R')_2$	+ Monoalkanolamine	→	Difunctional
RCO_2R'	+ Dialkanolamine	→	Difunctional
$R(CO_2R')_2$	+ Dialkanolamine	→	Tetrafunctional

Choice of Crosslinker

A plurality of functionality is required for an efficient crosslinker. Commercially available materials and application economics limit the choice of esters and alkanolamines for the preparation of polyfunctional tertiary β-hydroxyalkylamides to those shown in *Table* 3.

Obviously, a functionality of four is the best choice available, especially as this is an ideal number and bearing in mind that only 80-90% of this can be achieved with a crude unpurified β-hydroxyalkylamide. (i.e., real functionality will be 3-3.5). *Figure* 5 shows the general synthesis and structure of tetrafunctional crosslinkers that are obtained from adipic (n=4) and azelaic (n=7) dimethyl esters and diethanolamine (R=H) and diisopropanolamine ($R=CH_3$). This latter alkanolamine produces polyfunctional β-hydroxyalkylamides with tertiary amide and secondary hydroxy groups referred to in the preceding section. *Table* 4 lists these nominally tetrafunctional crosslinkers with their acronyms, product codes, and physical forms in the pure state. Those based on an even numbered carbon skeleton, adipamides, are solids, and all are freely water-soluble.

Figure 6 indicates that of the three amides evaluated, the one based on azelaic acid is faster in esterification rate with palmitic acid than those based on adipic acid. This slight advantage is reproducible and is attributed to the better solubility of the azelamide in the reaction medium. It is noteworthy that the adipamides which compare primary and secondary hydroxy group reactivity demonstrate similar rates of esterification at this temperature.

For a crosslinker to be usable in water-soluble carboxylated coatings, it must have hydrolytic stability at alkaline pH. Results in *Figure* 7 indicate that the tertiary amides with secondary alcohol groups based on diisopropanolamine (more hydrophobic) are more stable than those obtained from diethanolamine which have primary alcohol groups. Also, the azelamides are superior to the analogous adipamide, reflecting their greater hydrophobic character.

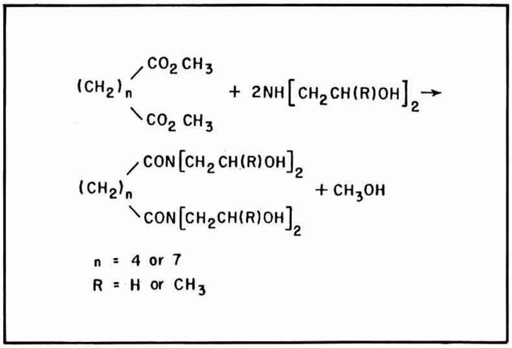

Figure 5—Tetrafunctional β-hydroxyalkylamides-synthesis

Table 4—Tetrafunctional β-Hydroxyalkylamide Crosslinkers

Compound	Code	Acronym	M.P. °C
Bis [N,N-di (β-hydroxyethyl)] adipamide	QM-552	BHEAD	120-122
Bis [N,N-di (β-hydroxypropyl)] adipamide	QM-606	BiPAD	128-130
Bis [N,N-di (β-hydroxyethyl)] azelamide	QM-591	BHEAZ	Liquid
Bis [N,N-di (β-hydroxypropyl)] azelamide	—	BiPAZ	Liquid

Based on reactivity and hydrolytic stability, the azelamide would be the crosslinker of choice for acrylic colloidal dispersions. However, as so often happens in industry, economics dictate that the adipamide, QM-606, be used. Fortunately, this is an acceptable compromise as the following application studies demonstrate.

QM-606 in Water-Borne Coatings

While the following discussion emphasizes evaluations of QM-606 as a crosslinker for acrylic colloidal dispersion resins only in coatings for spray application, its utility is not so limited. Enamels based on QM-606 have been formulated for application techniques other than spray and QM-606 has been found to be an effective crosslinker for resins other than colloidal dispersions. It can be used with acid-functional acrylic emulsion polymers and with organic solvent polymerized water-reducible resins. It might also be used as a modifier to

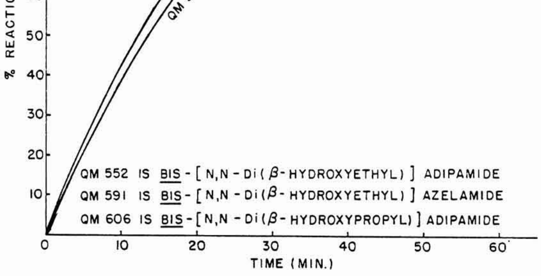

Figure 6—Esterification of palmitic acid with β-hydroxyamides

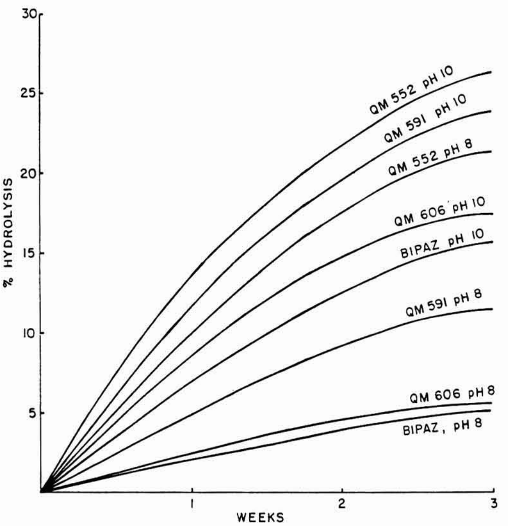

Figure 7—Hydrolysis of β-hydroxyalkylamides at 60°C

upgrade properties of resin types other than acrylic which have unreacted acid functionality to the detriment of film resistance properties. This discussion deals solely with the use of QM-606 as a crosslinker for acrylic colloidal dispersions and demonstrates the potential of this type of resin/crosslinker combination for low polluting coatings for appliances and other industrial baking finishes.

Effect of Crosslinker Level

Table 5 shows the effect of QM-606 crosslinker level on solvent swell ratios of Acrylic Resin A/QM-606 clear films. The solvent resistance maximizes at 1.0 equivalent crosslinker per equivalent polymeric acid. All subsequent evaluations were conducted at this stoichiometric ratio.

Table 5—Effect of QM-606 Crosslinker Level On Clear Film[a] Solvent Resistance

Eq. Polymer Acid/ Eq. Crosslinker[b]	Solvent Swell Ratio[c]
1.0/0.0	∞
1.0/0.5	1.91
1.0/0.8	1.67
1.0/1.0	1.54
1.0/1.2	1.55
1.0/1.4	1.64

(a) Films cast on glass and baked 300°F(149°C)/30 min. Dry film thickness equals 1.2-1.5 mils (30.5-38 μm).

(b) Acrylic Resin A/QM-606 clear blends with pH adjusted to 7.5 with dimethylaminoethanol.

(c) Film strips immersed in dimethylformamide.

$$\text{Swell ratio} = \left[\frac{\text{length of strip after immersion}}{\text{length of strip before immersion}}\right]^{3}$$

Effect of Bake Schedule

The effect of bake temperature and time on film property development is shown in *Table* 6. Full cure of the QM-606/acrylic enamel requires 300°F (149°C)/30 min but most properties develop in half that time: films baked 15 min suffer slightly reduced solvent resistance. Conventional acidic and basic catalysts do not reduce cure temperature.

Enamel Formulation and Stability

Using the hydroxyalkylamide crosslinker QM-606 and alkali-soluble acrylic colloidal dispersion resins we were able to formulate high gloss enamels with excellent sprayability, no formaldehyde emissions and with only very low levels of organic volatiles. About 6% of the volatiles of the formulations in *Table* 2 is organic solvent. The 2-butoxyethanol was added to promote metal wetting and improve spray atomization, the dimethylaminoethanol to solubilize the acrylic resin.

Solubilization with ammonia was also evaluated since the hydroxyalkylamides do not require tertiary amine neutralization for stability as do most amino resin crosslinkers. However, with the colloidal dispersion polymers ammonia neutralization resulted in inferior film formation. Microscopic mudcracks developed in the enamel films. This may have been caused by ammonia volatilization during early stages of baking which desolubilizes the resin. Adjustment of solvent balance or use of higher organic solvent levels may make ammonia neutralization possible with these colloidal dispersions but this approach has not yet been evaluated. In as yet unpublished studies with hydroxyalkylamides and certain acrylic emulsion polymers and organic solvent polymerized water-reducible resins, ammonia neutralization did not interfere with film formation and the expensive tertiary amines could be replaced with ammonia in the formulations.

QM-606/acrylic colloidal dispersion enamels have good hydrolytic stability since film properties of fresh and aged enamels are essentially identical. However,

Table 6—Effect of Bake Schedule on Enamel[a] Film Properties

	Bake Schedule			
Film Property[b]	275°F (135°C)/ 30 min	300°F (149°C)/ 15 min	300°F (149°C)/ 30 min	350°F (177°C)/ 30 min
Knoop hardness	4	13	13	14
Pencil hardness	—	H	2H	2H
Solvent resistance (% film failure after 100 MEK double rubs)[c]	100	10	0	0
Humidity resistance 1000 hr/100% RH/ 100°F (38°C)	—	No Effect	No Effect	No Effect

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(a) See *Table* 2 for enamel formulation. Binder = Acrylic Resin A/QM-606 = 83.1/16.9, TiO_2/Binder = 40/60.

(b) 1.0 - 1.4 mils (25-35 μm) on iron phosphate treated cold rolled steel.

(c) Films rubbed firmly with cheesecloth soaked in methyl ethyl ketone.

Table 7—Effect of TiO_2 Grade on Enamel[a] Viscosity Stability

TiO_2 Grade	Viscosity, #4 Ford Cup (Sec.)			pH		
(Rutile, High Gloss)	Initial	After 140°F (60°C) For 1 Week	After 22 Months At Room Temperature	Initial	After 140°F (60°C) For 1 Week	After 22 Months At Room Temperature
TiPure R-902	21	23	24	8.05	8.00	7.90
Zopaque® RC1-9	28	61	160	8.05	7.90	7.85

(a) See *Table* 2 for enamel formulation. Binder = Acrylic Resin A/QM-606 = 83.1/16.9. TiO_2/Binder = 40/60, 40% solids.
Zopaque is a registered trademark of Glidden Pigments, SCM Corp.

enamel viscosity stability was found to vary with TiO_2 grade and may be related to some interaction dependent on TiO_2 surface treatment differences. *Table* 7 compares stability at spray viscosity with two grades of high gloss rutile TiO_2. Viscosity is quite stable in one enamel but increases in the other. TiO_2 type does not significantly affect pH stability.

QM-606 vs. Melamine Crosslinking

Melamine resins are used extensively today as crosslinkers in water-borne coatings. In *Table* 8 film properties with melamine crosslinking are compared to those with QM-606 crosslinking of Acrylic Resin B. Many of the properties are identical. There is a slight gloss advantage for melamine and a slight salt spray advantage for QM-606. A major difference is the detergent resistance advantage for QM-606, which may make this crosslinker important for appliance enamel applications when used with an acrylic resin with composition optimized for maximum detergent resistance. Over primed substrates the QM-606 enamel baked 300°F (149°C)/30 min is as good as an organic solvent-borne acrylic-epoxy laundry appliance enamel which requires 350°F (177°C)/30 min for full cure. Over unprimed substrates the epoxy is superior.

Comparison of QM-606/Acrylic Enamel With Water-Reducible and Organic Solvent-Borne Coatings

Table 9 compares the properties of enamels prepared from some major types of acrylic/melamine and alkyd/melamine water-reducible and solvent-borne resins with Acrylic Resin A/QM-606 enamel.

In the comparison in Table 9, the QM-606 enamel film is harder but just as flexible and more stain resistant than the commercial acrylic/melamine. The QM-606 enamel has better adhesion to cold rolled steel than the solvent-borne alkyd, and accelerated weathering

Table 8—Film Properties with QM-606 vs. Melamine Crosslinking

Enamel Constants		ENAMEL TYPE Acrylic/Hydroxyalkylamide	Acrylic/Melamine	Acrylic/Epoxy
Binder		Acrylic Resin B/ QM-606=83.1/16.9	Acrylic Resin B/ Cymel® 303=80/20[a]	Acryloid® AT-70/ Epon® 1001=60/40
TiO_2 (TiPure R-902)/Binder		40/60	40/60	40/60
Film Property[b]	**Substrate**	**[Film Baked 300°F (149°C)/30 min]**	**[Film Baked 300°F (149°C)/30 min]**	**[Film Baked 350°F (177°C)/30 min]**
Pencil hardness	C.R.S.[c]	H	H	—
Print, 2psi/2hr/180°F (82°C)	C.R.S.	Very light	Very light	—
Gloss, %, 20°/60°	C.R.S.	75/85	83/92	—
Mandrel flex	C.R.S.	Pass ¼"	Pass ¼"	—
100 MEK double rubs, % film failure	C.R.S.	0	0	—
Ink stain, 30 min exposure	C.R.S.	Very light	Very light	—
Salt spray, 250 hr exp. rust/undercut at "X" scribe	C.R.S.	Slight/Slight	Slight/Moderate	—
Detergent resistance,[d] (exposure hours before blistering)	Electrodep. Primer C.R.S.	325 48	<24 <24	325 200

(a) Catalyzed with p-toluene sulfonic acid, 0.5% on binder.
(b) 1.1-1.4 mils (25-35μm) dry film thickness
(c) Iron phosphate treated cold rolled steel.
(d) 5% Tide laundry detergent at 165°F (74°C).
Cymel is a registered trademark of American Cyanamid; Acryloid is a registered trademark of Rohm and Haas Co.; Epon is a registered trademark of Shell Chemical Co.

Table 9—Properties of QM-606 Enamel Compared to Water-Reducible And Organic Solvent -Borne Coatings

	ENAMEL TYPE		
Enamel Constants	**Acrylic Colloidal Dispersion/ Hydroxyalkylamide**	**Water-Reducible Acrylic/Melamine**	**Solvent-Borne Alkyd/Melamine**
Binder	Acrylic Resin A/QM-606 = 83.1/16.9	Acryloid WR-97/ Uformite® MM-83 = 80/20	Duraplex® ND-77B/ Unformite MM-47 = 70/30
TiO_2 (Ti Pure R-902)/Binder	40/60	40/60	40/60
% Organic in volatiles, by volume	7	20	100
Enamel solids, %	40	40	50
Film Properties[a]			
Pencil hardness	2H	H	H
¼″ Mandrel flex	Pass	Pass	Pass
Print, 2psi/2hr/180°F(82°C)	Very light	Light	Very light
Gloss, %, 20°/60°	70/85	68/85	65/87
Color, K value	5.9	5.5	5.0
Overbake color, K, 400°F (140°C)/2hr	9.3	6.8	9.1
Adhesion (taped crosshatch), % failure	0	0	5
MEK resistance, % failure after 100 double rubs	0	0	0
Ink stain, 30 min	Trace	Light-Moderate	Light-Moderate
Boiling water, 30 min	No Effect	No Effect	No Effect
Accelerated weathering, % 20° gloss retained (2000 hr Xenon Arc)	97	97	42

(a) 1.0-1.3 mils (25-30μm) on C.R.S. (iron phosphate treated). Films baked 300°F (149°C)/30′.

Uformite and Duraplex are registered trademarks of Reichhold Chemicals, Inc.

shows that a QM-606 enamel should have the better durability which is typical of acrylics relative to alkyds.

On balance, the QM-606/acrylic enamel compares very favorably with currently used commercial products while emitting far less organic volatiles and no formaldehyde into the atmosphere.

SUMMARY

Polyfunctional tertiary β-hydroxyalkylamides are readily prepared by the amidation of polycarboxylic esters with secondary β-hydroxyalkylamines. The rapid esterification of the hydroxyl groups in these water-soluble amides with acid groups in polymers allows the formulation of low-energy and low-polluting coatings with water-borne resins. Studies with an hydroxyalkylamide crosslinker and acrylic colloidal dispersion resins demonstrate the potential for these systems as thermosetting coatings which emit no noxious cure volatiles and which can be used as coatings for appliances and other industrial baking finish applications.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the experimental support and technical contributions of many colleagues, in particular, O.C. Carter, H.J. Cenci, A. Gryn, K.A. Hughes, W.I. Kachelries, A. Kohr, A. Mercurio, C. O'Farrell, R.O. Palmer, W.H. Rice, and M.R. Yunaska.

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Stylus Measurement Of the Microgeometry of a Coated Surface

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The feasibility of using a stylus instrument for looking at a section through a coated surface is discussed. Experimental measurements of the deformation of paint by a stylus are presented, and it is shown that deformation has no significant effect on roughness measurements. Changes in the statistical microgeometry of a profile after successive coats of paint are examined by relocation profilometry. Most of the changes are found to occur after the first coat: the surface becomes smoother, its slopes become gentler, and its peaks more rounded. However, the shortest surface wavelengths are found to be relatively unaffected by painting.

INTRODUCTION

It is of some interest, both practical and theoretical, in the coating industry to establish the relationship between the microgeometry of the coated component and that of its coating. Such a measurement would show, for example, the extent to which the coating conformed to its substrate, which in turn might shed some light on the physical properties of the coating and on the mechanics of its adhesion.

For some time the measurement of the way in which a paint affects the surface finish of a material relied mainly on the use of taper-sectioning[1] and glossmeters.[2] In the first of those methods the specimen is sectioned at a very shallow angle, α, to the surface and the section is polished and examined microscopically. The result is a profile with its vertical scale exaggerated by a factor cot α, thus emphasizing the surface features. This method has the disadvantages of being destructive and time-consuming. The glossmeter, by measuring the reflecting properties of a surface, will give a relative determination of its average roughness but is incapable of measuring individual surface parameters.

The stylus instrument has for many years been used to record surface profiles of many types of engineering surfaces. In the stylus instrument, a pickup, driven by a gearbox, draws the stylus over the surface at a constant speed. The stylus is connected to a transducer which converts its vertical movement relative to some datum into an oscillating electrical signal which is electronically amplified to produce a trace on a chart recorder. This system has been modified to record the surface profile digitally on punched paper tape for subsequent computer analysis. The system has been described in detail elsewhere.[3]

The stylus instrument has previously been used by Hansen to measure profiles of painted surfaces.[4] Hansen used a Talysurf 4 instrument (Rank Taylor Hobson, Leicester) to measure a number of substrates and surface coatings. These measurements showed that identifiable differences in roughness could be detected with the stylus instrument for high gloss, semi-gloss, and flat coatings. It was also shown possible to detect the degree to which substrate irregularities are filled, and that substrate roughness can have a major influence on the appearance of the finished surface. Also vertical and horizontal distances could be measured directly from the chart recordings knowing the relevant magnifications. Thus, it was shown that the stylus instrument is a potentially applicable tool in the field of surface coatings.

In this paper Hansen's work is extended to show that the same stylus instrument will faithfully record the profile of even a soft coating. Some measurements of a painted surface made with a stylus instrument and their statistical analysis are described and discussed. All the coating measurements described were carried out on a shotblasted steel surface previously spray-coated with a two-pack reinforced edged-type primer paint (Metagard G250) and given subsequent brush-applied coats of a thixotropic anticorrosion marine paint (Silver Primocon®). Metagard 250 is for use with shotblasted surfaces. Its dry film thickness is about 20 μm. It contains 14% solids, weighs 0.95 kg/l and surface dries in three minutes. Silver Primocon has a recommended dry film thickness of 75-125 μm. It contains 39% solids, weighs 1.17 kg/l and surface dries in 6-8 hr. The microhardness of its dried film was measured by us as 50 VPN; hardness measurements on the primer were not feasible.

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Primocon is a registered trademark of International Paint Co.

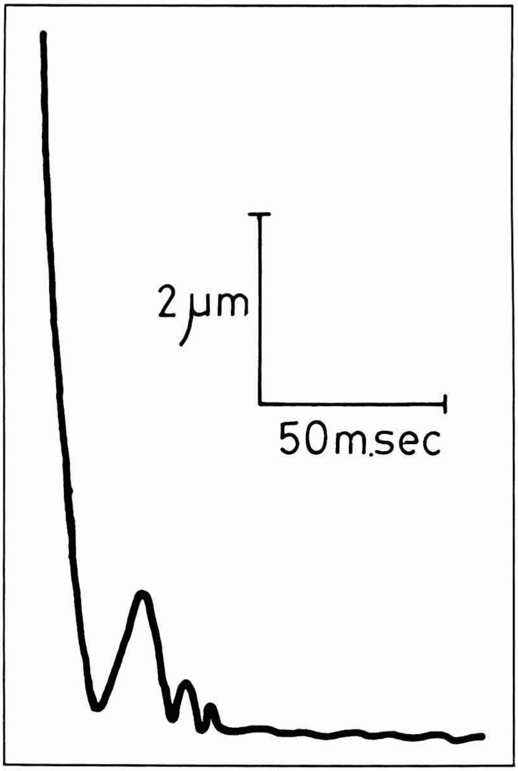

Figure 1—Oscillogram of the transducer output as the stylus falls towards and makes contact with the painted surface

SURFACE DAMAGE

Users of stylus instruments are sometimes concerned by the marks it occasionally leaves on the test surfaces, particularly if the material is soft, and the possibility that this damage can affect the accuracy of measurement. In this investigation, preliminary measurements were made to see if a stylus instrument could be used to measure a painted surface without the stylus ploughing through the surface geometry and thus misrepresenting the surface.

First a square of side equal to the traverse length of the stylus (8 mm) was scribed on a glass microscope slide painted three hours previously with a coat of the marine paint. A series of equally spaced traverses were taken within the scribed square parallel to one of its sides at intervals of time corresponding to paint drying times from three hours to eight hours. The paint was then allowed to dry for a total drying time of 24 hr. The film thickness was measured to be 13 μm.

Another series of parallel and evenly spaced traverses were then taken within the scribed squares and at right-angles to the initial traverses. Thus, the depth of ploughing of the stylus into the surface could be seen from the relative depth of the troughs on the chart recordings corresponding to the crossings of the initial traverses at various stages along their length and at different drying times. It was found from the chart recorder cross traces of the stylus that it does not produce any sign of surface damage after the paint has been allowed its manufacturer's specified drying time.

The possibility of the stylus deforming the painted surface elastically and thus misrepresenting the surface geometry was investigated further by pneumatically raising the stylus from the work surface and then letting it fall freely and observing the path of the stylus as it falls towards the surface, comes in contact, and any subsequent motion after initial contact. This was made possible with the connection of a storage oscilloscope to the amplified output of the stylus and by arranging the oscilloscope's triggering along with the storage facility to produce a photographic record of the reaction of the painted surface to the contact of the falling stylus.[5] From the results (*Figure* 1) there is no evidence of significant elastic deflection. It is concluded, on the basis of these preliminary experiments, that accurate measurement of the roughness of this particular coating material is feasible with a stylus instrument.

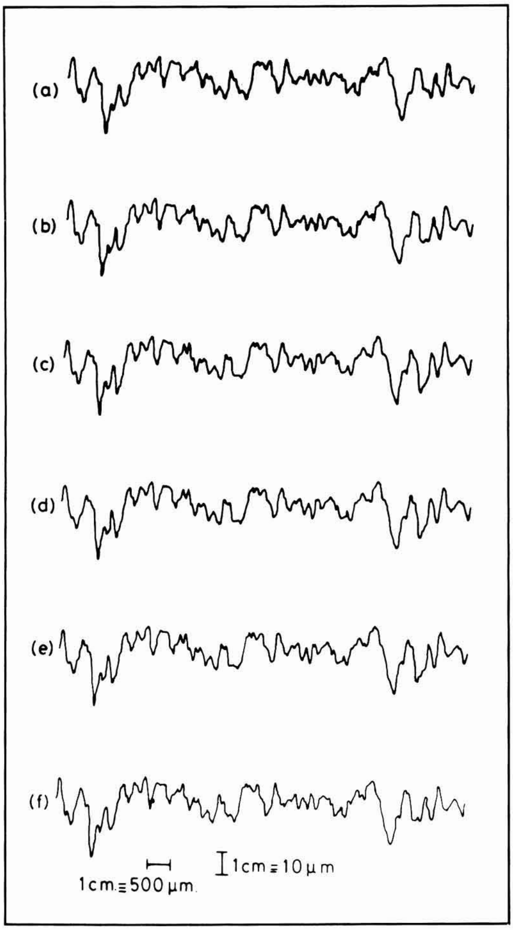

Figure 2—Profiles relocated at successive intervals of 24 hr

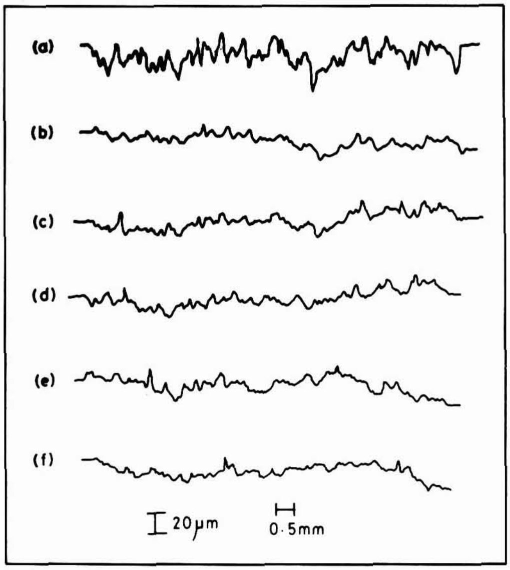

Figure 3—Relocated profiles of a shotblasted steel surface after coating with (a) primer, (b-f) successive top coats

RELOCATION

There are often situations, particularly in research, where it would be very useful to look at a particular section through a surface before and after an experiment, such as an investigation of wear, to see what changes have occurred to the surface geometry. It is clearly desirable that exactly the same section is traversed each time, otherwise the changes observed could be attributed to the displacement of the profile, and small but significant changes might not be observed at all. In general, however, the stylus, which is only 8 μm wide, will not retrace its path exactly unless constrained to do so. A jig for providing this constraint is termed a relocation table.[6]

The relocation table is bolted to the bed of the stylus instrument and the specimen stage is located kinematically and held in position at three points by three pneumatically operated pistons. The stage can be lowered and removed, an experiment of some kind performed on the specimen secured to the stage, and the stage with specimen replaced on the table. Relocation of the stylus then occurs to within the width of the original profile.[7] To preclude the possibility of lateral displacement it is necessary to raise the stylus both during the return stroke of the pickup and when the specimen stage is lowered and raised. The stylus is raised by directing two converging jets of compressed air, controlled by an electrically operated valve, onto the underside of the stylus arm.

The relocation table up to the present time had been used successfully over a maximum period of 24 hr only. Due to the drying time of the paint and the several coats required, it is necessary in the present investigation to rely on the relocation technqiue over a period of one week. This enhanced performance was achieved by increasing in steps the air pressure to the three location pistons until the distance between the specimen stage and table as measured with slip gauges stops decreasing. The pressure required to fully relocate the stage plus specimen was thus established and was then used to check the relocation up to seven days.

Table 1—Changes in the Microgeometry of a Shotblasted Steel Surface

Coating[a]	RMS Roughness (mm)	CLA Roughness (μm)	Mean Slope (degrees)	Mean Peak Curvature (μm)	Mean Valley Curvature (μm)
Primer only	10.4	8.4	9.3	12	–11
Primer +1 overcoat	7.0	5.9	6.4	20	–20
Primer +2 overcoats	6.2	5.0	5.8	19	–20

(a) Initial coat was a two-pack reinforced edged-type primer. Second coat was a thixotropic anti-corrosive marine paint. The surface was then air-dried for 24 hr at 17°C and 60% R.H.

In the second part of these measurements, chart traces were taken of traverses at 24 hr intervals over a period of seven days on a section of the primer-coated specimen surfaces. The specimen and stage were lowered after each traverse and removed from the relocation table as would be the case in the actual investigation. Prior to each traverse the stage with specimen was replaced and relocated with the previously determined relocation pressure; the resulting traces were compared and were found to show acceptable relocation of the specimen over the period of one week (*Figure* 2).

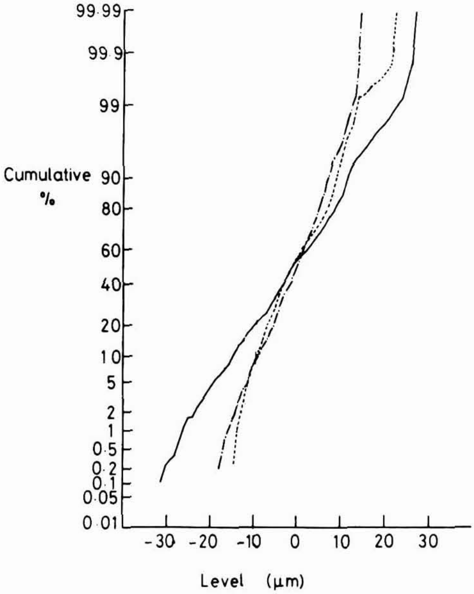

Figure 4—Cumulative height distributions: solid line, primer only; broken line, one top coat; chained line, two top coats

COATING MEASUREMENTS

A piece of ½ in. thick steel plate approximately 7 in. square was securely bolted to the top of the relocation specimen stage. The relocation housing was itself bolted to the bed of the stylus instrument. The initial specimen working surface was coated with a layer of primer paint to prevent corrosion. The surface was positioned so that traverses 8 mm in length could be taken approximately in the middle of the plate.

The raised stylus was brought to the start of its traverse, lowered, and the chart recorder was started. The traversing mechanism was put into gear and a traverse was taken. At the end of the traverse the gearbox was put into neutral, the stylus raised, and the arm was returned to its starting position. The specimen stage plus plate could now be lowered, removed from the rig, and then part of the working surface given an evenly brush-applied coat of marine paint.

After allowing the paint to dry for 24 hr, the specimen stage was replaced and relocated on the relocation table. The stylus was lowered and a traverse taken. This procedure was repeated to produce chart recordings of profiles of the initial primered surface and of the subsequently applied five coats of the marine paint. Any change in the surface profile produced by painting could then be seen by aligning the traces by their predominant surface features (*Figure* 3). The output from the stylus transducer on passing through the instrument's amplifier circuits represents the measured surface profile but with its horizontal and vertical scales distorted in relation to the actual surface. Thus, when looking at the traces it must be remembered that the vertical magnification is exaggerated relative to the horizontal by a factor of ×50.

The whole process of taking traverses and painting was repeated on another part of the working surface and this time the stylus transducer output was digitized and recorded on paper tape for computer processing. For this the sampling rate of the system had to be substantially increased to sample at a sufficiently large number of points on the profile for a statistical analysis to be made possible. The data logging system in use[3] was already in its maximum sampling mode and to achieve this desired greater sampling rate the sampling per unit length was increased by decreasing the stylus speed of traverse. To do this the gearbox was switched to neutral and a special gearbox connected to the stylus arm drive mechanism. Thus the ×20 horizontal magnification setting on the stylus instrument for the chart recorder traces was increased to ×1000 to produce the increased number of samples required for computer analysis. The vertical magnification was unaltered.

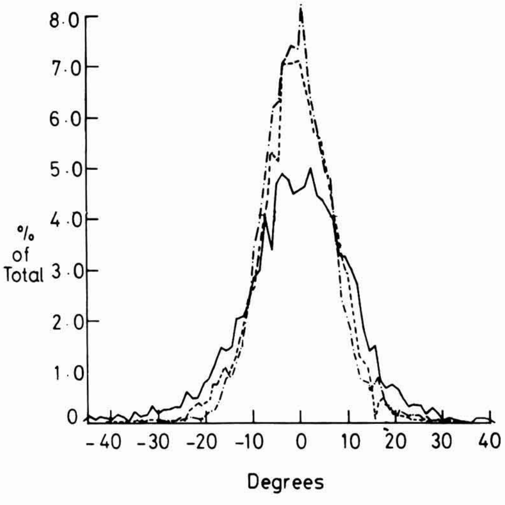

Figure 5—Slope distributions: legend as for Figure 4

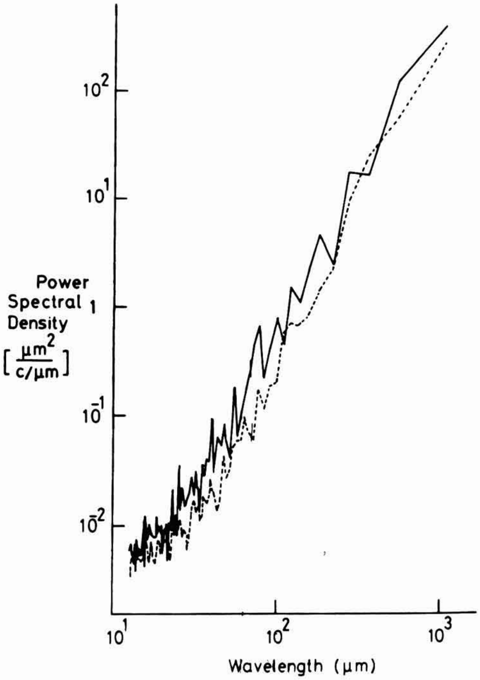

Figure 6—Power spectrum: legend as for Figure 4

From inspection of *Figure* 3 it appeared that few significant changes took place in the profile after the second applied coat. On the strength of these observations it was decided to take the repeat traverses, using the data logging system, on only two applied overcoats. This was thought adequate, in the time available, for a statistical analysis of the painted surface.

First, a general-purpose program written in Fortran IV for an ICL 1905E computer was used to analyze the profile data. This program computes root-mean-square roughness, center-line average roughness, peak and valley distributions, mean line crossings, peak and valley curvatures and their distributions, and profile slopes and curvatures and their distributions.[3] Height readings are input as voltages positive and negative with respect to some arbitrary reference. They are first reinterpreted as heights and a mean line fitted, thus correcting for any slight mechanical misalignment in the measuring instrument. Second, a program, again written in Fortran IV, was used to compute the power spectral density functions of the data.

RESULTS AND DISCUSSION

A visual inspection of the profile chart recorder traces (*Figure* 3) gives a general indication of the surface changes. The traces show that on the application of the initial coat of paint the roughness of the surface is substantially decreased whereas on further applications of paint the surface roughness does not seem to change any further. This is confirmed by the computed results collected in *Table* 1.

One useful way of examining surface changes is to plot the cumulative height distributions on a normal probability scale (*Figure* 4). This is a scale so distorted that a Gaussian height distribution appears as a straight line, whose slope is inversely proportional to the roughness. The height distribution of the primered surface is quite closely Gaussian except for its topmost fraction, which is considerably smoother. This is a characteristic "transitional" topography^8 and suggests that the original surface peaks had been removed by some finishing process. The same surface after one overcoat had been applied is very much smoother but less closely Gaussian. The effect of a further coat is confined to the top 1% of the height distribution. The roughness of the top 0.1% of the surface remains the same throughout as shown by the local slopes of the three curves. A possible explanation is that the paint flows off the highest peaks and leaves them as untouched "islands" of the original surface.

The distribution of slopes also changes (*Figure* 5). Not far from Gaussian for the primered surface, it becomes markedly more leptokurtic for the two coated surfaces, i.e., the fraction of smaller slopes is higher than a Gaussian distribution would predict. This no doubt reflects the inability of the paint to subtend a very steep angle as it dries. The absolute mean slope in fact decreased by 38% after the second coat, while the mean peak radius of curvature increased by 58% (*Table* 1). Numerical values of peak and valley radii of curvature are about the same, possibly a consequence of the limiting effect of surface tension.

When the power spectra of the surfaces are compared (*Figure* 6) it appears that the process of painting has reduced the height of surface irregularities whose horizontal dimensions range from about 20 μm to about 200 μm. It is interesting to speculate on the mechanism responsible for the lower limit. One possible limiting factor is the particle size of the aluminum filler. It was not possible to obtain a distribution of particle sizes from the manufacturer but the information is available that 0.7% of filler particles will not pass a 44 μm sieve, suggesting that a substantial fraction may be as large as 20 μm.

The inference is that due to the presence of a filler it will be impossible to obtain a painted surface smoother than a certain limiting roughness corresponding to wavelengths of less than about 20 μm, no matter how smooth the substrate. To test this a glass microscope slide of negligible roughness was painted; the roughness of the painted surface was measured as 2.0 μm CLA. The discrepancy between vertical and horizontal dimensions suggests that the larger flakes of filler, if lying flat, are about 10 times as long as they are thick.

The roughness of painted surfaces can be regarded as the sum of three components: one due to the composition of the paint, one due to the method of its application, and one due to the irregularities of the original substrate. The second of these components was effectively absent in the present investigation; no evidence of anisotropy was found in the surface measurements, and were any application effect present it is unlikely that *Figure* 3 would present its actual striking similarity between successive coats. The final microgeometry is clearly influenced heavily by the original substrate roughness. The composition of the paint must also play a part, however, otherwise it is difficult to see why power was not removed evenly across the whole spectrum of surface wavelengths.

CONCLUSIONS

It is important to know how painting changes roughness. This has been measured qualitatively in the past. With a stylus instrument, however, quantitative measurements are possible. Interesting results have already been obtained with a stylus instrument on a painted surface. But does the stylus damage the paint and so give false readings?

Our measurements were made on a shotblasted steel surface sprayed with primer and then brush-coated with successive layers of marine paint. To examine stylus damage, glass was painted and the surface was scratched with the stylus at intervals as it dried. Scratches made after the recommended drying time were imperceptible. The stylus was then dropped onto the surface to look for elastic deflection of the paint, but none could be found.

To ensure that the same section was examined before and after drying, the testpiece was mounted on a relocation table, which made sure that the testpiece was replaced in exactly the same position under the stylus. The table was modified so that it would work for a week and the testpiece, measured daily, was relocated perfectly.

Profiles 8 mm long were measured in the middle of the primer-coated testpiece. The testpiece was removed for each overcoat, replaced, and measured again. This was done for five overcoats, allowing the paint to dry for 24 hr before each measurement. The experiment was repeated and this time the profiles were recorded digitally. As no changes were observed visually in the first experiment after the second overcoat, the repeat was carried out for two overcoats only. The results were analyzed by computer.

The first overcoat made the surface about two-thirds as rough, but the second overcoat made it only 11% smoother. The roughness of the top 0.1% of the surface did not change, suggesting that paint may not cover the highest peaks. Slopes and peak radii decreased by 48% and 58%, respectively, and there were fewer high slopes, probably due to the rheology of the paint. Wavelengths of between 20 μm and 200 μm were reduced in height, but the surface was just as rough below 20 μm, a dimension corresponding to the size of the larger particles of filler. This suggests that a smooth surface could actually be made rougher by painting, an effect which we confirmed by experiment.

ACKNOWLEDGMENTS

We are grateful to the British Ship Research Association, Wallsend, for supplying the marine paint, and to Smith's Dock Co. Ltd., Middlesbrough, for supplying the specimen surface. This work was supported by the Science Research Council.

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APPENDIX

DATA LOGGING SYSTEM AND DATA ANALYSIS

The data logging system (*Figure* 7) consists of a demodulator, digital voltmeter (DVM), punch encoder, and paper-tape punch. The demodulator filters the 3KHz carrier waveform from the signal on its way to the DVM. The DVM is an analogue-to-digital converter (ADC) which for each sampling sequence integrates the signal over a length of time (equivalent to horizontal distance) which is finite but small compared with the constant sampling interval.

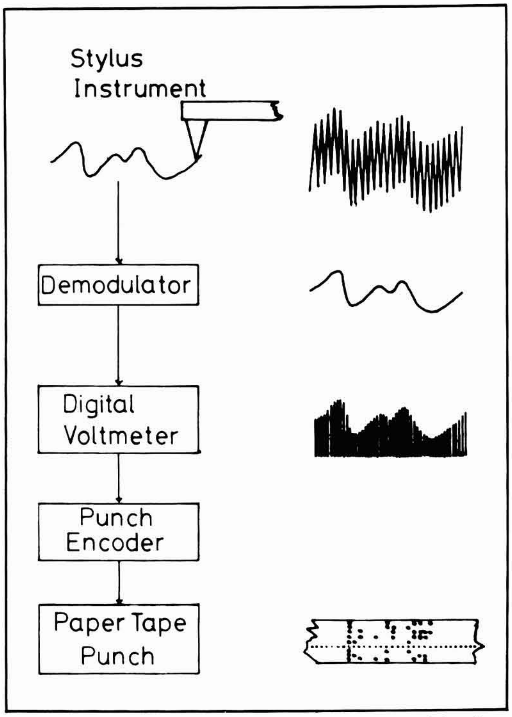

Figure 7—Block diagram of surface measurement and data logging system

The voltage readings are then output as a string of digital pulses to the punch encoder, the DVM output being displayed on a digital display. The punch encoder converts the pulses into a form which will be accepted by the paper-tape punch. The punch records the height readings as a sequence of six-character words comprising four digits, a polarity sign, and an end-of-word character. Four digits gives a height resolution to 1 part in 9999 which is theoretically more than adequate.[3]

The system is very slow, sampling at a maximum rate of three readings/sec, equivalent to 1.85 μm in horizontal distance. The length of stylus traverse was 8 mm.

Comparison of Electrostatic Spray-Applied Powder Coatings

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Phillips Petroleum Company*

A comparison of the thermal and chemical resistance of several electrostatically-applied coatings including phenolic, phenolic-epoxy, polyester, epoxies, polyphenylene sulfide (PPS), and ethylene-chlorotrifluoroethylene (E-CTFE) resins was made, along with two coatings, polytetrafluoroethylene (PTFE) and polyimide, applied from liquids.

Coatings were evaluated for thermal stability by monitoring property changes during aging at elevated temperatures. Chemical resistance was determined by exposure to 21 different environments for 24 hr at 90°C.

At 100 and 150°C all coatings, except the phenolic and phenolic-epoxy, survived with little deterioration evident after 5,000 hr in air. At 200°C, only the polytetrafluoroethylene coating remained unchanged after 2,700 hr. Polyimide, PPS, and E-CTFE survived 2,500 hr, while others deteriorated in 1,000 hr or less.

Outstanding chemical resistance was shown by PTFE, PPS, and E-CTFE coatings. Other coatings suffered some attack under test conditions.

INTRODUCTION

The application of coatings by electrostatic powder spraying has increased in popularity in recent years because it offers many advantages for coatings users, such as the absence of solvents, high efficiency, and excellent coverage of complicated parts. With the increased popularity of this method of application, a wide range of plastic coating materials has become available for electrostatic spraying to give coatings with a wide variety of properties and capabilities.

Recent reviews [1,2] compare high temperature coatings and the corrosion resistance of various plastics, but direct comparisons of a spectrum of coatings tested under the same conditions are not available. The purpose of this study was to determine the advantages and limitations of a wide range of coatings, particularly in regard to their cost, cure requirements, performance at elevated temperatures, and chemical resistance. Properties of six different types of powder coating resins were compared, with two coatings applied from liquid systems (slurry, varnish) being included for comparison. These materials are described with regard to type, cure conditions, hardness ratings and general appearances in *Table* 1.

EXPERIMENTAL

The coating materials used in this study were commercial materials, and are listed in *Table* 1. The manufacturer's specifications for application and curing were followed as closely as possible. Usually a range of curing conditions was initially employed to allow for variations in curing conditions, oven temperatures, and heat-up time. The coatings so cured were then examined and tested to pinpoint optimum curing conditions.

Test coupons of 20 gauge carbon steel, about 40 mils (1 mm) thick, 3 × 6 in. (76 × 152 mm), were solvent degreased, then grit blasted before coating. The dry powder coating materials were applied electrostatically using a GEMA HP720 Electrostatic Spray Gun following manufacturer's instructions to obtain optimum coverage for each type of powder.

Test Methods

AGING: Test coupons were aged in electrically heated, forced draft ovens regulated at 100°, 150°, and 200°C. Coupons were removed periodically, examined for effects of aging on appearance, and then tested by the following tests:

Thickness—determined with calibrated Mark 1 Type A Elcometer gauge (Gardner Laboratory, Inc.), according to ASTM E-376.

Pinholes—checked with a K-D Bird Dog Paint Flaw Detector (Gardner Laboratory, Inc.).

Mandrel bend—according to ASTM D-1737, using mandrels of 3/16 through 1-in. diameter.

Reverse impact—determined with a Gardner Heavy-Duty Variable Impact Tester, according to ASTM D-2794.

Adhesion—judged by cutting an X mark through the coating to the metal, then probing, under 20X magnification, with a scalpel to determine ease of separation of coating from the metal along the cut.

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Portions of this paper appeared in the March 1978 Preprint booklet of the Organic Coatings and Plastics Chemistry Div. of the American Chemical Society and are reprinted by permission of the ACS, the copyright owner.

Table 1—Comparison of Coatings and Curing

Coating Material	Coating Type	Curing Conditions (min/°C)	Pencil Hardness[a]	Average Thickness (mil)	Appearance
Phenolic	Powder	5/177	9H+	2	Green, glossy, slight roughness
Phenolic-epoxy	Powder	10/177	5H	1.5	Transparent, glossy, smooth
Polyester	Powder	45/177	6H	3	Black, very smooth and glossy
Epoxy A (pipe coating)	Powder	10/232	5H	5	Brown, glossy, slight roughness
Epoxy B (electrical)	Powder	12/177	9H	3	Green, glossy, slight roughness
Epoxy C (general)	Powder	15/177	3H	4	White, very smooth and glossy
Polyphenylene sulfide (PPS)	Powder	40/370	H	3	Black, glossy, smooth, some craters
Ethylene-chlorotrifluoroethylene (E-CTFE)	Powder	10/270	HB	2	Transparent, glossy, smooth
Polytetrafluoroethylene (PTFE)	Slurry	15/400	HB	1.5	Gray, smooth, some mud cracking
Polyimide	Varnish	5/250	9H	2	Brown, glossy, smooth

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[a] HB is softest; 9H is hardest.

CHEMICAL RESISTANCE: Small aluminum coupons (13 × 102 mm) were coated by the same procedure used for coating the larger steel coupons. A coupon of each type was then immersed in the chemical contained in an 8 oz bottle. The bottle was sealed and kept immersed in a heated water bath for 24 hr. The bars were removed from the chemical and washed thoroughly in running water. The coatings were examined, under magnification where necessary, to determine the extent of attack by the chemical. For tests with sodium hydroxide and sodium hypochlorite solutions, small steel strips were coated and used instead of aluminum.

DISCUSSIONS AND RESULTS

Thermal Aging

APPEARANCE: The effect of aging at elevated temperatures on the appearance of the coatings is summarized in *Table* 2. Aging at 100°C had little effect on the

Table 2—Effect of Thermal Aging on Appearance[a]

Coating	Effect of Aging at: 100°C	150°C	200°C
Phenolic	Gradually darkened (4-30 wk)	Darkened (1 wk)	Blackened (1 wk) Cracks (4 wk)
Phenolic-epoxy	Gradually darkened (4-30 wk)	Gradually darkened and less gloss (1-16 wk)	Darkened and lost gloss (1 wk)
Polyester	No change (30 wk)	No change (16 wk)	Very dull, slightly rough (10 wk)
Epoxy A	No change (30 wk)	Sl. darkened and less gloss (16 wk)	Darkened, less gloss (1 wk) Dull and rough (10 wk)
Epoxy B	No change (30 wk)	Gradually darkened (1-16 wk) Sl. less gloss (16 wk)	Darkened, less gloss (1 wk) Dull and rough (10 wk)
Epoxy C	Gradually yellowed (4-30 wk)	Gradually yellowed (1-16 wk)	Browned (1 wk) Visible pinholes (3 wk)
PPS	No change (30 wk)	Sl. less gloss (12-16 wk)	Dull surface haze (1 wk) Mottled (3 wk)
E-CTFE	No change (30 wk)	Sl. darkened (4-16 wk)	Darkened (2 wk) Dulled and deteriorated (16 wk)
PTFE	No change (30 wk)	No change (16 wk)	Sl. less gloss (1 wk) No other change (16 wk)
Polyimide	No change (30 wk)	No change (16 wk)	Sl. darkened (1 wk) No other change (16 wk)

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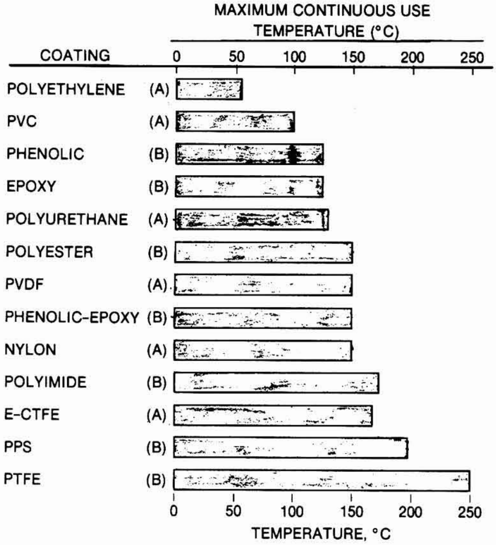
[a] Slightly is abbreviated in this table.



(A) VALUE FROM LITERATURE (1,2)
(B) VALUE BASED ON OVERALL DATA OF PRESENT WORK.

Figure 1—Comparison of maximum continuous use temperature

various coatings other than slight darkening of the phenolic, phenolic-epoxy and the white epoxy. Aging at 150°C had a more pronounced effect with gradual darkening of the phenolic, phenolic-epoxy and epoxy coatings. At 200°C, aging dramatically increased the deterioration in appearance of most of the coatings. The phenolic and phenolic-epoxy coatings deteriorated in one week, and the epoxies and polyester in 3 to 10 weeks. The PPS coating became dull and mottled in three weeks. Although the E-CTFE coatings appeared darkened in three weeks, this was probably due to darkening of the metal surface of the coupon which could be seen through the transparent coating. Only the PTFE and polyimide coatings showed little change in appearance in the 200°C aging, after a total of 16 weeks.

Table 3—Pinhole Formation During Thermal Aging

Coating	Time (wk) to Form Pinholes at 100°C	150°C	200°C
Phenolic	4-8	3-4	1-2
Phenolic-epoxy	>30	>20	1-2
Polyester	>30	>20	3-4
Epoxy A	>30	>20	3-4
Epoxy B	>30	>20	6-10
Epoxy C	>30	>20	3-4
PPS	>30	>20	>16
E-CTFE	>30	>20	12-16
PTFE	>30	>20	>16
Polyimide	>30	>20	16

Based on these and literature data, typical maximum continuous use temperatures for a variety of organic coating resins are given in *Figure* 1.

PINHOLE FORMATION: Formation of pinholes induced by aging at elevated temperatures is shown in *Table* 3.

Coating continuity was determined by electrical conductivity measurements taken on the coated coupon. Aging at 100-150°C affected only the phenolic coating. At 200°C, continuity of phenolic, phenolic-epoxy, epoxy and polyester coatings was affected as evidenced by the rapid formation of pinholes. Polyimide and E-CTFE coatings slowly deteriorated, developing pinholes in 12-16 weeks. Continuities of PPS and PTFE coatings were not affected during 16 weeks exposure to 200°C.

ELONGATION: Comparison of elongations, calculated from the mandrel bend test, of unaged coatings shows that the thermoplastic materials have high elongation while the thermoset resins, with exception of Epoxy A, have relatively low elongation. The effect of thermal aging on the elongation of all these resins is shown in *Table* 4.

Aging at 100°C caused a significant decrease in the elongation of Epoxy C, a general purpose epoxy resin.

Table 4—Effect of Aging on Elongation

		% Elongation[a] After Aging at:								
		100°C			150°C			200°C		
Coating	Unaged	8 wk	12	30	4 wk	12	20	3 wk	10	16
Phenolic	<3		No change			No change		<3	-[b]	
Phenolic-epoxy	<3		No change			No change		<3	-[b]	
Polyester	>21	12	12	21	21	13	8	11	7	<3
Epoxy A	>23		No change		13	10	<3	<3	<3	<3
Epoxy B	4	<3	<3	<3	<3	<3	<3	<3	<3	<3
Epoxy C	>22	12	<3	<3	<3	<3	<3	<3	-[b]	
PPS	>21		No change			No change		No change		<3
E-CTFE	>21		No change			No change		No change		<3
PTFE	>21		No change			No change		No change		
Polyimide	>21		No change		>21	<3	12	5	4	<3

(a) Elongation measured by mandrel bend test (ASTM D-1737).
(b) Not tested further due to deterioration of the coating.

Table 5—Embrittlement by Thermal Aging

Coating	Rating[a] Unaged	Time (wk) to Embrittlement[a] At Temp. of:		
		100°C	150°C	200°C
Phenolic	Brittle	—	—	—
Phenolic-epoxy	Brittle	—	—	—
Polyester	Brittle	—	—	—
Epoxy A	Average	16	4	1
Epoxy B	Brittle	—	—	—
Epoxy C	Average	8	4	1
PPS	Flexible	>30	>20	16
E-CTFE	Flexible	>30	>20	10
PTFE	Flexible	>30	>20	>16
Polyimide	Flexible	>30	>20	6

(a) As rated by reverse impact (ASTM D-2794) as follows:
Brittle 6 in-lb (<0.7 Newton-metre)
Average to 140 in-lb (15.8 Newton-metre)
Flexible 140 in-lb (>15.8 Newton-metre)

Other materials were not affected by this exposure. At 150°C, Epoxy A, Epoxy C and polyester suffered significant loss in elongation. At a higher temperature (200°C) these three materials and polyimide lost elongation in three weeks. After ten weeks, phenolic and phenolic-epoxy coatings which had low elongation initially were badly deteriorated. After 16 weeks PPS and E-CTFE were affected. Coatings of PTFE were not measurably altered by these exposures.

REVERSE IMPACT: Reverse impact measurements on the coatings were used to indicate the brittleness of the coating after thermal aging, as is shown in *Table* 5. Several of the coatings were rated brittle (cracking below 6 in-lb) before aging. Epoxy A became brittle after 16 weeks at 100°C, and Epoxy C was embrittled after 8 weeks at 100°C. They both were embrittled after 4 weeks at 150°C and after only one week at 200°C. The PTFE, PPS, E-CTFE, and polyimide coatings remained flexible after 30 weeks at 100°C, and 20 weeks at 150°C. Only the PTFE remained flexible after 16 weeks at 200°C, the others being embrittled after 16, 10, and 6 weeks at 200°C, respectively.

Chemical Resistance

Table 6 shows the results of tests determining the chemical resistance of the various coatings, judged after 24 hr exposure at 90°C. The indications of chemical resistance from this study should be used only as guidelines in the selection of a plastic coating for a particular application. Polytetrafluoroethylene is well known for its outstanding chemical resistance, and was not attacked by any of the reagents tested. Polyphenylene sulfide also has excellent chemical resistance and showed only slight attack by hypochlorite solution (oxidation). Ethylene chlorotrifluoroethylene also has good chemical resistance, but under these conditions showed susceptibility to aliphatic and aromatic amines. The other coatings, phenolic, phenolic-epoxy, epoxies, polyester, and polyimide were, in general, attacked by most aromatics, amines, hypochlorite solution, and dioxane.

Table 6—Chemical Resistance[a]

Exposure: 24 hr/90°C

Chemical	Phenolic	Phenolic Epoxy	Poly-ester	Epoxy A	Epoxy B	Epoxy C	PPS	E-CTFE	PTFE	Poly-imide
1. Methyl ethyl ketone	X	0	0	0	0*	0	0	0	0	S
2. Ethyl acetate	S	0	0	0	0	0	0	0	0	S
3. Chloroform	0	0	0*	0	0	0	0	0	0	X
4. Carbon tetrachloride	0	0	0	0	0	0	0*	0	0	0
5. Gasoline	0	0	0	0	0	0	0*	0	0	0
6. Toluene	0	0	0	0	0	0	0*	0	0	0
7. Benzaldehyde	X	X	X	X	X	X	0*	0	0	X
8. Phenol	0	X	X	X	X	X	0	0	0	X
9. Aniline	X	X	X	X	X	X	0*	S	0	X
10. Benzonitrile	S	S	0	S	0	0	0*	0	0	X
11. Nitrobenzene	0*	S	0	0	0*	0	0*	0	0	X
12. N-Methyl pyrrolidone	X	X	X	X	X	X	0*	S	0	X
13. n-Butyl amine	X	X	X	X	X	X	0	X	0	X
14. n-Butyl alcohol	0	0	0	0	0	0	0	0	0	0
15. Cyclohexanol	0	S	0	X	X	0	0	0	0	0
16. p-Dioxane	X	X	X	X	X	S	0*	0	0	X
17. Air	0	0	0	0	0	0	0	0	0	0
18. Water	0	0	0	0	0	0	0*	0	0	0
19. 30% Sodium hydroxide	X	X	0*	0	0*	0*	0	0	0	X
20. 5% Sodium hypochlorite	X	X	X	S	X	0*	S	0	0	S
21. 10% Sulfuric acid	0	0	0	0	0	0	0	0	0	X

(a) Ratings are indicated as follows:
X = Severe attack
S = Slight attack
0 = No attack
0* = No attack, but coating dulled.

Table 7—Overall Comparison

Coating Type	Relative Cost[a]	Advantages	Disadvantages
Phenolic	Low	Hard, tough; low cure temp.	Brittle, poor thermal stability, limited chemical resistance
Phenolic-epoxy	Low	Excellent coverage with thin film; good appearance	Brittle, poor thermal stability, limited chemical resistance
Polyester	Low	Good appearance; excellent all-purpose coating	Limited thermal stability and chemical resistance
Epoxies	Low	Many types available with varying properties; good all-purpose coating	Limited thermal stability and thermal resistance
PPS	Med.	Excellent chemical resistance; good thermal stability and flexibility	Dark color; high cure temp.
PTFE	Med.	Very inert coating; unsurpassed chemical resistance and thermal stability	Soft; primer needed; high fusion temp.
E-CTFE	High	Excellent chemical resistance; good thermal stability and flexibility	Soft; primer needed for some metals
Polyimide	High	Good appearance and gloss; hard coating resistance	Solvent based varnish; limited chemical

(a) Cost basis as follows:
Low — <$2/lb
Med. — $2-10/lb
High — >$10/lb.

SUMMARY

Table 7 gives a comparison of the outstanding features as well as the weaknesses for each type of plastic coating in this study. The coatings were not evaluated strictly from a cost standpoint, but only from the generally recognized cost of each type of coating material. The lower-cost powder coatings - phenolic, phenolic-epoxy, polyester, and epoxies - are well suited for many general applications and for many applications with limited exposure to elevated temperatures and with chemical exposure limited to mostly aliphatic organics or aqueous solutions. For a powder coating at modest cost, polyphenylene sulfide offers excellent chemical resistance and thermal stability, but compared to many coating systems has the disadvantage of requiring a high curing temperature. At somewhat higher cost, E-CTFE also offers excellent chemical resistance and thermal stability and requires only sufficient baking to fuse the coating properly. As a coating resin PTFE is unsurpassed in thermal stability and chemical resistance but has a definite cost disadvantage, is relatively soft, often requires a multicoat (primer) process and requires a high temperature bake cycle.

References

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Survey of the Applications, Properties, and Technology Of Crosslinking Emulsions

Part II

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This review summarizes most of the principal concepts which have been employed over the last ten years to develop thermosetting or crosslinking emulsions. The conceptual elements which have formed the foundation for this new generation of coatings systems are discussed in terms of the science, technology, properties, and applications of the emulsion systems which have been reported in the literature. This survey presents the advantages and limitations afforded by each of the functional monomers or polymer classes which have been investigated for the development of crosslinking emulsions. Part I of this review presented a state of the art introduction into crosslinking emulsions and discussed the development of conventional emulsions which produce homogeneously crosslinked coatings through the utilization of N-methylolacrylamide. *(See June 1978 JCT—Ed.)* Part II extends the discussion of conventional emulsions which produce homogeneously crosslinked coatings to include systems containing N-(alkoxymethyl)acrylamide, hydroxymethylated diacetone acrylamide, allyl N-methylolcarbamate, N-formyl-N′-acryloylmethylenediamine, vinyl and acryloxysilanes, and autooxidizable monomers. This six-part review, all of which will be published in the JOURNAL OF COATINGS TECHNOLOGY, serves as an introduction to a series of publications from these laboratories describing the research and development of crosslinking emulsions which are new and novel, or which have not been adequately described in the present literature.

CONVENTIONAL EMULSIONS WHICH PRODUCE HOMOGENEOUSLY CROSSLINKED COATINGS

Homogeneously crosslinked systems require crosslinking sites which are statistically distributed throughout and between the polymer particles; such systems are produced by the conventional emulsion polymerization of monomers which are capable of self-condensation or autooxidization. For example, self-condensable monomers include N-methylol(meth)acrylamide, N-(isobutoxymethyl)acrylamide, hydroxymethylated diacetone acrylamide, acryloxyalkylsilanes, and peroxide compounds with vinyl functionality, while autooxidizable monomers include cyclohexenyl acrylate, allyl acrylate, and vinyl condensation products of drying oils.

N-(alkoxymethyl)acrylamide, $CH_2 = CHCONHCH_2OR$

Because of the many advantages which self-condensable monomers such as N-methylolacrylamide (N-MA) offer in the production, application, and product performance of thermosetting systems, the coatings industry has sought to develop an improved generation of self-crosslinking monomers which retain the desirable qualities of N-MA but which provide improved resistance to premature crosslinking, increased miscibility with organic monomers, diminished toxicity, and reduced sensitivity to water. The functional monomers which have been found to provide the greatest potential as substitutes for N-MA include both N-alkyl and N-alkyl ethers of N-MA. The N-alkyl ether derivatives most commonly reported in the literature include N-(methoxymethyl)acrylamide,[1-3] N-(isopropoxymethyl)acrylamide,[4,5] N-(butoxymethyl)acrylamide,[6,7] N-(isobutoxymethyl)acrylamide, N-(octyloxymethyl) acrylamide, and N-(carboxymethyleneoxymethyl) acrylamide.[9]

Since etherified methylol groups are kinetically less active than unblocked methylol groups, the alkyl ether derivatives of N-MA provide greater resistance to premature crosslinking[10,11] and, therefore, offer greater product stability and allow for more convenient handling procedures than do products based upon N-MA. The crosslinking reaction of these derivatives, which forms methylene-bis-acrylamide structures, is second order; and the activation energy for the process is sufficiently low so that the condensation reaction is rea-

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Table 1—Emulsion Polymerization Procedure For a 47/40/10/3-Butyl Acrylate/Styrene/IBMA/Acrylic Acid Copolymer

Ingredient	Grams Used
Reactor Charge	
Water	753.33
Aerosol A-102	50.81
Aerosol OT-75	8.40
Ammonium persulfate	3.15
Monomer Mix	
Butyl acrylate	296.10
Styrene	252.00
IBMA	66.32
Acrylic acid	18.90
Latex Characteristics	
% Surfactant	3.5%
% Coagulum	≈0.10%
% Solids	44–45%
Particle size	500-1200Å (>90%)
Film properties	Clear, continuous film, only slight grit

Procedure: The contents of the reactor charge were purged with nitrogen for 15 min and heated to 65°C, whereupon 10% of the monomer mix was added to the reactor charge over a 10–15 min period. After initiation (slight exotherm) the monomer flow was reduced so that the balance of the monomers were added in 2–3 hr. After addition of the monomers, the contents of the reactor were maintained for 1 hr at 65°C. The resulting emulsion was cooled and filtered through a 20 mesh screen into a suitable container.

sonably efficient.[12] Thermosetting systems based upon N-alkyl ethers of N-MA normally require curing cycles of 20 to 30 min at 150–170°C to obtain optimal film properties.

Among the common derivatives, the alkyl ether of N-MA whose characteristics are suggested as being the most complementary to those of N-MA is N-(isobutoxymethyl)acrylamide (IBMA). IBMA is available commercially as a clear, colorless liquid (85% pure) which is insoluble in water but soluble in common solvents and monomers. Being insoluble in water, IBMA is dispersed primarily in the organic phase of the emulsion environment and, therefore, produces emulsion systems with a relatively homogenous distribution of crosslinking sites throughout the latex particle. In general, products which employ the alkyl ethers of N-MA display greater uniformity and more homogenous structures than those which are based upon N-MA.[13]

IBMA can be polymerized by any of the conventional emulsion techniques. *Table* 1 illustrates a starting point formulation which employs a monomer-feed technique and which demonstrates the monomer's solubility in the organic phase.[14]

IBMA is compatible with a variety of common monomers, and similar polymerization techniques have been used to prepare emulsion copolymers of IBMA containing acrylonitrile,[15] ethyl acrylate,[16,17] 2-(perfluorooctyl)ethyl acrylate,[18] 2-ethylhexyl acrylate,[19] ethylene and vinyl acetate, vinyl chloride,[20] and vinylidene chloride.[21] Small amounts of hydroxyethyl acrylate and/or acrylic acid[22] are usually included in the monomer charge to enhance the crosslinking efficiency.

Applications

Being a derivative of N-MA, the areas of application for IBMA are similar to those of its parent. For example, some of the suggested areas of application for IBMA-based emulsions include coil coatings,[23] coatings[24] and adhesives for paper,[25] adhesives for plastics[26] and for general use,[27] appliance coatings,[28] textile coatings and finishes, and coatings for electrodeposition applications.[29,30]

In textile applications, IBMA based emulsions are claimed to provide enhanced tensile strength and elasticity, improved resistance to abrasion, creasing, heat, and chemicals.[31] For example, a nonwoven fabric impregnated with a 55/36/9-vinyl acetate/ethylene/IBMA copolymer emulsion and subsequently cured for 10 min at 80°C followed by 5 min at 140°C demonstrated a tensile strength of 152 Kg/cm^2, an elongation of 14%, crease recovery of 90%, a compression modulus of 88%, and a weight retention of 99% after washing. A control nonwoven fabric impregnated with a vinyl acetate-ethylene copolymer emulsion which did not contain IBMA demonstrated a tensile strength of 71 Kg/cm^2, an elongation of 10%, crease recovery of 80%, a compression modulus of 75%, and a weight retention after washing of 0% after an equivalent cure cycle.[32]

Coatings suitable for textile applications requiring improved dry-cleaning fluid resistance were prepared from a 49.4/36/14.6-ethylene/vinyl acetate/IBMA copolymer emulsion which employed sodium lauryl sulfate and a Pluronic-type nonionic as the emulsifier mixture. The self-crosslinking emulsion was applied to glass and cured 5 min at 140°C to yield coatings which were unaffected after 12 hr immersion in Perclene® at 60°C.[33]

Binders for nonwoven fabrics providing improved tensile strength, durability, and hand were prepared from a 66.5/27.0/3.4/3.1-vinyl chloride/ethylene/acrylamide/IBMA copolymer emulsion. The bifunctional emulsion was applied to a 75/25-cellulose/nylon nonwoven fabric and cured at moderate temperatures

Table 2—Effect of Curing Conditions on Film Properties Of an IBMA Based Emulsion

	Cure Temperature for 10 min/C				
Property	**93**	**121**	**149**	**177**	**121**
Catalyst[a]	None	None	None	None	0.5%
Film thickness micrometers	23	23	24	25	16
Hardness, Knoop					
Initial	3.1	6.7	7.0	9.3	14.8
After 10 days	3.2	6.8	7.8	11.5	—
After 50 days	5.0	10.3	10.8	12.1	—
Hardness, pencil					H-2H
Impact resistance Reverse, in/lb	<10	<10	<10	<10	<10
Adhesion, crosshatch	Pass	Pass	Pass	Pass	Pass
MEK double rubs[b]	—	—	5-10	20	200+

(a) Catalyst = p-toluenesulfonic acid.
(b) MEK = methyl ethyl ketone.

Perclene is a registered trademark of E.I du Pont de Nemours & Co., Inc.

Table 3—Parameters Investigated in the Crosslinking of IBMA Based Emulsions

Sample	Polymer Composition[a] Sty/EA/MAA/HEA/IBMA	Monomer Preferentially Located in Particle		Parameter Investigated
		CORE	SHELL	
1	45/45/0/0/10	—	—	Control
2	45/40/0/5/10	—	—	Presence of OH group
3	40/40/5/5/10	—	—	Presence of OH & COOH group
4	40/40/5/5/10	MAA/HEA	IBMA	Version of sample 3; IBMA in shell
5	45/40/5/0/10	—	IBMA	Presence of COOH group; IBMA in shell
6	45/40/5/0/10	—	IBMA	High M.W. version of Sample 5
7	45/40/5/0/10	MAA	IBMA	COOH group in core; IBMA in shell
8	35/35/10/10/10	—	—	High conc. COOH & OH groups; low MW
9	35/35/10/10/10	—	—	High MW version of sample 8
10	40/40/10/0/10	—	—	High conc. of COOH groups; high MW

(a) Sty = Styrene, EA = ethyl acrylate, MAA = methacrylic acid, HEA = 2-hydroxyethyl acrylate.

to yield a fabric with a hand and drape rating of four, a tensile strength of 7.2 lb/in, and a toughness of 3.1 in-lbs.[34]

Binders for nonwoven fabrics with improved chemical resistance and adhesion were derived from a 66.5/27.0/3.4/3.1-vinyl chloride/ethylene/acrylamide/IBMA copolymer which possessed a molecular weight of 64,000 and a glass transition temperature of –7°C. The bifunctional emulsion was applied to a 75/25-cellulose/nylon textile and cured 2 min at 118°C to yield a fabric with a tenacity of 1.29 Kg/cm and 71% insolubility in tetrahydrofuran (THF).[35]

Primer coatings for metal with improved intercoat adhesion to radiation-curable topcoats were formulated from 100 parts of a 68/20/10/2-butyl acrylate/styrene/IBMA/acrylic acid copolymer emulsion (50% solids), 10 parts Epikote® 1001 emulsion (50% solids), 10 parts $SrCrO_4$, and 7 parts TiO_2. The paint was applied to galvanized steel sheets and cured 30 sec at 240°C to give 15 μm coatings with good impact and salt water resistance, and which also demonstrated excellent adhesion to the substrate and topcoat.[36]

Anticorrosive coatings for steel sheet were prepared by blending 80 parts of a 42/41/15/2-ethyl acrylate/styrene/IBMA/methacrylic acid copolymer emulsion with 20 parts of a 40/39/15/6-ethyl acrylate/styrene/IBMA/itaconic acid water-dispersible resin. Phosphated steel sheets were coated with the blend and cured 20 min at 180°C to give crack free, hot water resistant, anticorrosive coatings with a pencil hardness of 2H.[37]

Formulation Variables

Since blocked methylol groups are more resistant to self-condensation than are free methylol groups, the film properties displayed by IBMA based coatings are more greatly dependent upon the curing temperature than are coatings based on N-MA. *Table* 2 indicates the effect that various curing conditions have on the film properties of a 45/25/20/10-methyl methacrylate/butyl acrylate/IBMA/hydroxyethyl acrylate copolymer emulsion.[38]

These results are similar to those displayed by N-MA based coatings in that the greatest degree of crosslinking occurred when the system was both catalyzed and thermally cured.

In addition to the effect that thermal curing has on the film properties of IBMA based coatings, some of the compositional and topographical parameters which influence the physical properties of these systems have also been studied.[39] In this investigation, the degree of crosslinking was followed by thermal evolution analysis (TEA) and thermal gas chromatography (TGC), both of which measure the amount of isobutanol liberated from the system as IBMA self-condenses. The compositional and topographical parameters investigated were the presence of hydroxyl groups, the presence of carboxyl groups, the effect of increased concentrations of both hydroxyl and carboxyl groups, the effect of different molecular weights and, consequently, varying concentrations of chain end-groups, the effect of localizing IBMA in the particle shell or distributing it throughout the latex particle, and the effect of catalysis by means of p-toluenesulfonic acid. The copolymer compositions used to evaluate each of these parameters are presented in *Table* 3.

The emulsions possessing a core and shell with different compositions were prepared by dividing the styrene-ethyl acrylate monomer feed into two portions and adding the functional monomer in either the first or second half of the divided feed.

The methods used to separate and rank the various parameters were the determinations of (1) threshold or onset crosslinking temperature (T_o), (2) reaction rate constant (K, min^{-1}), and (3) reaction activation energy (E_{act}, Kcal $mole^{-1}$). The results from these determinations are presented in *Table* 4 which ranks the systems according to T_o and in *Table* 5 which ranks the systems according to K and E_{act}.

In both tables, the parameters are listed in order of decreasing ability to promote crosslinking. The some-

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Epikote is a registered trademark of Shell Chemical Co.

Table 4—Effect of Formulation Parameters On Crosslinking Temperatures of IBMA Emulsions

Sample[a]	Formulation Parameter	Onset Temp. °C[b]	Reaction Peak Temp. °C[c]
5	Presence of OH, IBMA in shell	108	162
5	PTSA catalyzed version of Sample 5	110	165
7	Presence of COOH, IBMA in shell	117	165
4	Version of sample 3, IBMA in shell	118	167
4	PTSA catalyzed version of Sample 4	120	168
3	Presence of COOH and OH groups	125	174
6	High MW version of Sample 5	125	176
2	Presence of OH groups	120	178
1	Control	120	185

(a) Sample designation and composition as specified in *Table* 3.

(b) Onset temperature was determined by means of thermal evolution analysis whereby the evolution rate of volatile by-products generated during the crosslinking reaction was measured as a function of temperature. From graphic plots of evolution rate as a function of temperature, the onset temperature was obtained by extrapolating a line from the peak through the point of maximum to the base line.

(c) Temperature at which the largest amount of isobutanol was liberated.

Table 5—Effect of Formulation Parameters on Rate Constants And Activation Energies for Crosslinking of IBMA Emulsions

Sample[a]	Formulation Parameter	K, (min^{-1})[b] 143°C	167°C	E_{act}[c] (Kcal/mole)
8	High conc. COOH and OH groups, Low MW	0.043	0.114	14.8
5	PTSA catalyzed version of sample 5	0.036	0.128	19.2
10	High conc. COOH groups, High MW	0.033	0.150	23.0
5	Presence of COOH, IBMA in shell	0.030	0.088	16.3
6	High MW version of Sample 5	0.029	0.102	19.1
9	High MW version of Sample 8	0.029	0.150	24.9
3	Presence of COOH and OH groups	0.025	0.116	23.3
4	PTSA catalyzed version of sample 4	0.024	0.133	26.0
4	Version of Sample 3, IBMA in shell	0.022	0.098	22.6
1	Control	0.018	0.066	19.7
2	Presence of OH groups	0.017	0.099	26.7

(a) Sample designation and composition as specified in *Table* 3.

(b) Reaction rate constants were determined from the thermal gas chromatography measurements of the amount of isobutanol liberated at a given temperature during a given time period. The slope of a semi-log plot of the fraction of isobutanol remaining in the film as a function of time provided the rate constant values according to the equation $f = e^{-kt}$, where f is the fraction of IBMA unreacted, t is the time, and k is the rate constant.

(c) The isobutanol evolution data collected at several temperatures was used to calculate activation energies by applying the expression

$$E_{act} = \left[\frac{RT_1T_2}{(T_2-T_1)} \right] \ln \left(\frac{K_2}{K_1} \right)$$

where K_1 and K_2 are the rate constants at two different temperatures, T_1 and T_2, and R is the gas constant.

what tentative conclusions drawn from the data presented in these tables were:

1. The crosslinking reaction is promoted by the presence of copolymerized acid;
2. Low molecular weight polymer showed preferential crosslinking over high molecular weight polymer;
3. For enhanced crosslinking a localized concentration of IBMA in the shell area is preferred to a uniformly distributed concentration of IBMA;
4. Catalysis by p-toluenesulfonic acid had little effect on the rate of crosslinking; and
5. The presence of hydroxyl groups had little effect on the rate of crosslinking.

In order to correlate the trends established from threshold temperature, reaction rate constant, and activation energy data with practical curing conditions, the 40/40/5/5/10-styrene/ethyl acrylate/methacrylic acid/hydroxyethyl acrylate/IBMA latex (Sample 3 from *Table* 3) was formulated into a thermosetting paint and evaluated under various curing conditions. The results obtained from the evaluation for two modes of catalysis and for four curing profiles are described in *Table* 6.

These data indicate that little crosslinking occurred below 107°C and that 121°C (250°F) was required for the development of more optimal properties. The threshold temperature for Sample 3 was 125°C.

Sample 3 was then formulated with TiO_2 at a pigment to binder ratio of 0.8/1.0 and tested under conditions required by higher performance coatings. The evaluation utilized a 4 hr chemical and stain resistance test and a 4 hr immersion test in 57°C water. The results are presented in *Table* 7.

The results indicate that reasonably good properties can be obtained by curing at 121°C and that 149°C is required for a complete cure. This trend was in agreement with the TEA data which indicated that a maximum evolution of isobutanol occurred somewhat above 149°C.[39] It should also be noted that in spite of the high concentration of IBMA and other hydrophilic monomers, the formulation showed no loss in initial hardness after a 4 hr immersion in water.

IBMA has also been found to provide excellent color stability to over-baking at 149°C as indicated by the data presented in *Table* 8. Latex films containing IBMA were found to yellow less on over-baking than corresponding films that did not contain IBMA.[38]

Some of the performance characteristics, properties, and applications of self-crosslinking emulsions which contain less commonly recognized methylol-functional monomers are discussed in the following sections.

Hydroxymethylated Diacetone Acrylamide, $CH_2 = CHCONHC(CH_3)_2COC(H)_{3-x}(CH_2OH)_x$

When formaldehyde is reacted with diacetone acrylamide, a self-crosslinking, methylol-functional monomer is produced which was marketed under the tradename of hydroxymethylated diacetone acrylamide (HMDAA). Because the alkyl moiety of the substrate monomer possesses reactive hydrogen atoms, the methylol groups become preferentially attached to the carbon atoms alpha to the keto carbonyl

Table 6—Cure Response of an IBMA Based Coating[a]

Catalyst	Cure Cycle Min/Temp °C	Pencil Hardness	Acetone Rubs	Water Immersion, 2 hr[b] Adhesion	Blush	Pencil Hardness
None	Air dry	2B	4	0	6	5B+
	20/71	B	3	0	9	5B+
	20/93	HB	14	0	8	5B+
	20/107	HB	100+	10	10	B
	20/121	HB	100+	10	9	HB
PTSA, 1 phr	20/71	HB	2	0	0	4B
	20/93	F	3	4	4	5B
	20/107	HB	10	6	6	3B
	20/121	F	68	10	9	2B
$MgSiF_6$, 1 phr	20/80	HB	6	0	2	5B+
	20/93	HB	8	0	2	5B+
	20/107	HB	100+	0	8	4B
	20/121	H	100+	10	10	HB

(a) The paints were formulated in the conventional manner using the vehicle of sample 3 and lead chromate-phthalo green at a 0.3/1.0 pigment to binder ratio together with a 50/50 blend of n-butanol and butyl carbitol as a coalescing aid. The paint was spray-applied onto Bonderite 1000 test panels at an approximate dry film thickness of 1 mil. The coatings were aged 3 days at ambient before baking.
(b) Adhesion and blush ratings from 0 (complete failure) to 10 (no change).

rather than at the amide nitrogen. The resulting molecule, which contains one amide hydrogen and one or more free methylol groups, is capable of a high degree of hydrogen bonding, thereby enhancing the properties associated with higher cohesive energy densities and water sensitivity. Since HMDAA is water soluble and possesses unetherified methylol groups, the properties of self-crosslinking emulsions containing HMDAA more closely resemble the physical and topographical characteristics of N-MA based emulsions than those based on IBMA.

Emulsions containing HMDAA can be prepared with most of the common vinyl-type monomers such as styrene, vinyl acetate, ethyl acrylate, butyl acrylate, and acrylonitrile. Small amounts of acrylic or methacrylic acids are usually included in the composition to enhance the crosslinking efficiency.

In accord with the effects displayed by N-MA based emulsions, the free methylol groups of HMDAA based emulsions may also provide an increase in colloidal stability through a partial lowering of the interfacial tension. For example, a 76.5/20.5/3-vinyl acetate/butyl acrylate/HMDAA copolymer emulsion was prepared by using an anionic-nonionic surfactant blend consisting of 1% sodium lauryl sulfate and 1.4% Triton® X-405. The emulsion possessed only 0.4% wet coagulum at 47.8% solids. Also a 67.2/16.2/10/5.5/1.1-ethyl acrylate/butyl acrylate/acrylonitrile/HMDAA/acrylic acid copolymer emulsion was prepared from an anionic-nonionic surfactant blend consisting of 3.5% Triton X-301 and 1.5% Triton X-405. This emulsion contained no coagulum. Even though HMDAA may provide increased colloidal stability, the choice of surfactants used to polymerize these systems is almost completely governed by the HLB requirements of the general polymer composition.

HMDAA based emulsions can be prepared by means of most of the conventional polymerization techniques.

Table 7—Cure Response of an IBMA Latex Formulated as a White Enamel[a]

	Cure Conditions (Minutes/°C) 20/107	20/121	20/149
60/20° Gloss	42/15	55/14	26/7
Pencil hardness	H	H	2H
Dry adhesion	10	10	10
Impact, F/R (in/lbs)	—	50/12	—
Spot tests[b]			
$EtOH/H_2O$	3	9	9
3% NaOH	1	0	8
20% NaOH	0	0	4
1% H_2SO_4	1	1	9
2% HOAc	8	10	10
Black ink	4	4	7
Mercurochrome	3	6	7
Mustard	2	5	7
Immersion test[c]			
Blush resistance	4	8	9
Wet adhesion	0	10	10
Wet hardness	>5B	HB	2H

(a) The enamel was formulated in the conventional manner using the vehicle of sample 3 and TiO_2 at a pigment/binder ratio of 0.8/1.0; the use of coalescing aid was not indicated. The paint was spray-applied onto Bonderite 37 test panels at an approximate dry film thickness of 1 mil. The coatings were aged 3 days at ambient before baking.
(b) 4 hr spot test rated on scale 0-10; 10 = No change and 0 = complete failure.
(c) Rated after 4 hr immersion in 57°C water.

Table 8—Film Yellowness Index Data[a]

Composition[b]	% IBMA	Exposure at 149°C 0 Hr	2 Hr	6 Hr	24 Hr
65/35-EA/MMA	0	–0.009	0.048	0.080	0.113
	10	0.0	0.029	0.029	0.067
84/16-VA/EHA	0	–0.003	0.019	0.025	0.079
	10	+0.011	0.011	0.019	0.040

(a) ASTM D2244-68.
(b) EA = ethyl acrylate, MMA = methyl methacrylate, VA = vinyl acetate, EHA = 2-ethylhexyl acrylate.

Triton is a registered trademark of Rohm and Haas Co.

Table 9—Scrub Resistance of Latex Paints Containing HMDAA[a][44]

Latex Composition[b]	Adhesion %	Scrub Cycles[c]
80/20-VA/BA	0	3600
77/20/3-VA/BA/HMDAA	50	5000
75/20/5-VA/BA/HMDAA	100	5000
Commercial vinyl latex[d]	0	200
grafted with 3% HMDAA	90	5000
grafted with 5% HMDAA	100	5000

(a) Air dried coating applied over gloss alkyd enamel.

(b) VA = vinyl acetate, BA = butyl acrylate.

(c) Tests were run to 5000 cycles using a Gardner Straight Line Washability and Abrasion Machine #702. The test charts were scrubbed under distilled water using a natural bristle brush.

(d) Grafting Procedure: 400g of a commercial vinyl latex, 2.0 ml t-butyl hydroperoxide, and optionally 8.8 g of Triton X-405 were heated to 70°C in a conventional emulsion polymerization apparatus. While maintaining the reaction temperature between 70°C and 75°C, a mixture consisting of 12.0 g HMDAA, 14 g water, and 1.6 g sodium sulfoxylate formaldehyde was added to the latex mixture over a 30 min period. After a conversion period, the latex was cooled to ambient temperature and filtered through cheesecloth.

However, a preferred procedure utilizes an emulsion-feed technique whereby a pre-emulsion containing 87.7% of the monomer feed is added to an initiated pre-emulsion containing the remaining amount of monomers.[44]

Many of the application areas for HMDAA based emulsions are similar to those of emulsions based on N-MA or IBMA. HMDAA based emulsions have been recommended as binders and adhesives for paper,[40] textiles,[41,42] and wood;[43] as vehicles in coil coating; as appliance coatings; and in automotive finish applications.

In architectural coatings applications, HMDAA based emulsions have been found to offer increased wet adhesion to alkyd substrates. *Table* 9 illustrates the advantages provided by emulsions containing 3% and 5% HMDAA in a scrub resistance test.

For industrial applications requiring greater physical integrity and higher performance characteristics, HMDAA based emulsions may be combined with melamine resins such as Cymel® 301 or Uformite® MM-83. However, as the data in *Table* 10 indicate, systems containing in excess of 5% HMDAA may not benefit greatly from the addition of the external curing agent.

Table 10—Effect of External Curing Agents On Insolubility of HMDAA Copolymers

Emulsion Composition[a]	Cymel 301 % by weight	% Acetone Insolubles[b] Cured 90 sec at 107°C	% Acetone Insolubles[b] Cured 90 sec at 163°C
70/15/14/1	0	—	10
EA/BA/AN/AA	3.6	—	8
70/15/12.8/1.2/1	0	57	72
EA/BA/AN/HMDAA/AA	1.0	78	87
	2.3	80	86
70/15/10.75/3.25/1	0	88	90
EA/BA/AN/HMDAA/AA	2.3	90	93
	3.6	90	92
70/15/9/5/1	0	90	90
EA/BA/AN/HMDAA/AA	2.3	92	93
	3.6	90	92
	4.9	90	90

(a) EA = ethyl acrylate, BA = butyl acrylate, AN = acrylonitrile, AA = acrylic acid.

(b) Extraction test consisting of immersing a 2.4 mil free-film (approx. 1g) in 100 mil of acetone for 2 hr at ambient.

For applications requiring intermediate performance characteristics, an emulsion containing 3% HMDAA in combination with 1% to 2.5% melamine resin generally provides adequate crosslink densities and film integrity.

Emulsions not containing an external curing agent are capable of producing highly crosslinked films over a broad range of pH. *Table* 11 presents the effects of varying the pH on the acetone insolubility of a 70/15/10.75/3.25/1-ethyl acrylate/butyl acrylate/acrylonitrile/HMDAA/acrylic acid copolymer emulsion.[44]

The insensitivity of HMDAA based coatings to strong acid catalysis, i.e., p-toluenesulfonic acid, is similar to the response found with coatings containing IBMA. Other uses and advantages provided by HMDAA based emulsions are described in the following examples.

Binders for textile and wood applications were prepared from a 88/10.5/0.8/0.4/0.3-vinyl acetate/ethylene/HMDAA/itaconic acid/diallyl maleate copolymer emulsion which utilized 5.7% Triton X-405 and 1% Pluronic®F-68 as the emulsifier mixture. Films of the emulsion which were cured for 3 min at 133°C were 67.4% insoluble after an 18 hr immersion in 24°C benzene. Besides providing chemical resistance, these products also demonstrated good resiliency and compressibility and possessed excellent clarity and resistance to discoloration.[45]

Oil and water-repellent coatings for fabrics were prepared from a 73/25/2-perfluoroalkyl acrylate/vinyl chloride/HMDAA copolymer emulsion which utilized 6% $C_{17}H_{33}COO(CH_2CH_2O)_2CH_2CH_2OH$ as the polymerization surfactant. The improvements provided by this HMDAA-based emulsion in oil and water repellency, hand, and softness as compared with an equivalent emulsion which did not contain HMDAA are illustrated in *Table* 12.[46]

Exterior can coatings and reflowable automotive coatings were prepared from a 85.5/9.3/3.9/1.3-methyl methacrylate/butyl acrylate/acrylic acid/HMDAA copolymer emulsion which was prepared in the pres-

Table 11—Effect of pH on Crosslinking Ability Of Emulsion Coatings Containing 3.25% HMDAA[a]

Latex pH	Adjusted By	% Acetone Insolubles
3.4	H_2SO_4	88
5.9	—	87
9.2	NH_4OH	87
9.4	NaOH	87

(a) Films dried 3 hr at room temperature and cured 90 sec at 163°C.

Cymel is a registered trademark of American Cyanamid Co.
Uformite is a registered trademark of Rohm and Haas Co.
Pluronic is a registered trademark of Wyandotte Chemicals Corp.

Table 12—Property Improvements Provided by HMDAA Based Emulsions

Latex Composition[a]	Test Fabric[b]	Softness[c]	Hand Feel	Initial Repellency		Repellency after 5 washings		Repellency after 5 dry cleanings	
				oil[d]	water[e]	oil	water	oil	water
73/25/2	wool	0.99	Exc.	130	100	100	80	100	90
FA/VC/HMDAA	35/65-P/C	1.00	Exc.	120	100	100	70	100	80
75/25	wool	0.97	Fair	120	90	60	0	80	70
FA/VC	35/65-P/C	0.99	Fair	110	90	50	0	70	50

(a) FA = perfluoroalkyl acrylate, VC = vinyl chloride.
(b) P = polyester, C = cotton.
(c) Softness = Stiffness of untreated fabric/stiffness of treated fabric.
(d) Measured by falling drop method in n-heptane and Nujol and mixtures thereof. Oil repellency of 150 is associated with a composition of 100% n-heptane and 0% Nujol while oil repellency of 50 is associated with a composition of 0% n-heptane and 100% Nujol.
(e) Water-repellency was measured in accord with ASTM D-583-63 and rated accordingly. 100 = no wetting of surface, 0 = complete wetting of both surfaces.

ence of 26 phr of dibenzyl diethylene glycol diadipate polyester plasticizer. The resulting blend was combined with 0.6%, based on total resin solids, of monoethanol amine and mixed with a white tinter to form a paint with a pigment to binder ratio of 57/100. The paint was sprayed onto zinc phosphated steel and cured 30 min at 149°C. The resulting 40μm coating displayed a 20° gloss of 60% and provided excellent resistance to condensing humidity at 66°C.[47]

Vehicles for architectural applications which provided improved wet adhesion to alkyd surfaces were prepared by polymerizing a monomer mixture consisting of 54.4/29.0/14.7/1.5/0.4-vinyl chloride/2-ethylhexyl acrylate/n-butyl acrylate/HMDAA/acrylic acid in the presence of a poly(vinyl chloride) seed latex. The polymerization utilized 0.8% sodium dodecyl benzene sulfonate and 2.1% octadecyl sulfosuccinamate as the surfactant mixture and 1% of a 34.5% poly(vinyl chloride) seed latex with a particle size of 0.035 microns. The resulting "grafted" latex possessed 52.8% solids, a pH of 8.8, viscosity of less than 75 cps, excellent mechanical stability, and a particle size of 0.35 μm. Thirty-three parts latex was combined with 66.7 parts of a pigment slurry (38.1 wt.% pigment), and the resulting paint was applied over a pre-aged alkyd undercoater. After a 24 hr drying period at ambient conditions, samples of emulsion were immersed in room temperature water for 20 min. After immersion, masking tape was applied over a scored area of the coating. The emulsion was found to pass five pulls of masking tape while two similar emulsions containing zero and 0.75% HMDAA failed after one and two pulls of masking tape, respectively.[48]

High performance emulsion coatings which were useful in various industrial applications were prepared from a 87/11.2/1.5/0.3-ethyl acrylate/acrylonitrile/HMDAA/methacrylic acid copolymer emulsion. The latex was prepared by means of an emulsion-feed technique and utilized 3.5% Igepal® CO-897 and 2.3% Igepal CO-530 as the surfactant mixture. The emulsion was then formulated into white enamels at pigment volume concentrations of 56% and 70% and evaluated against a similar emulsion containing 1.5% N-MA. The results of evaluation are presented in *Table* 13.[49]

HMDAA is no longer available from its initial commercial developer since systems based upon HMDAA were not significantly different from those based upon N-MA.[50]

OTHER FUNCTIONAL MONOMERS CONTAINING METHYLOL GROUPS

The applications, properties, and technology of emulsions containing other methylol-functional monomers are given in the following examples.

Allyl N-Methylolcarbamate, $CH_2 = CHCH_2OCONHCH_2OH$

Allyl N-methylolcarbamate (AMC) is prepared by reacting a solution consisting of 44 g allyl carbamate in

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Igepal is a registered trademark of GAF Corp.

Table 13—Comparison of Latex Paints Formulated From HMDAA and N-MA Based Emulsions

Latex Function Monomer	Paint PVC	Scrub Resistance (cycles)	Enamel Holdout	Burnish Resistance	Stain Removal (Strokes)			Viscosity (KU)	
					China marking	Pencil	Crayon	Initial	After 3 wks. at 120°F
1.5% N-MA	56	2610	70.1%	Excellent	35	15	15	83	82
1.5% HMDAA	56	2600	70.1%	Excellent	35	15	15	83	82
1.5% N-MA	70	130	40.6%	Good	32	8	12	89	69
1.5% HMDAA	70	130	40.6%	Very good	32	8	12	90	69

300 g water with 16.5 g paraformaldehyde and about 3 ml 50% aqueous NaOH. The mixture was heated for 3 hr at 75°C while the pH was maintained at 11 with NaOH. The product contained 16.9% AMC dissolved in water. A 94.5/3.1/2.4-vinyl acetate/acrylamide/AMC copolymer emulsion was prepared using sodium formaldehyde sulfoxylate at 53°C and a pH of 2.5-3.5. The emulsion yielded films which were 89.5% insoluble in $CHCl_3$ after curing 45 min at 110°C. The products were useful as textile or ink binders.[51]

Emulsions which were useful as laminating compounds or as binders for textiles were prepared by polymerizing a pre-emulsion consisting of 93.2/3.4/3.4-vinyl chloride/acrylamide/AMC in the presence of a poly(vinyl chloride) seed emulsion, maleic anhydride, and ethylene. The polymerization employed 2.9% sodium lauryl sulfate and 2.9% dioctyl sodium sulfosuccinate as the surfactant mixture and 1.4% poly(vinyl chloride) seed emulsion. The resulting "grafted" latex displayed a composition of 51.1/45/1.8/1.8/0.3-vinyl chloride/ethylene/acrylamide/AMC/maleic anhydride, a solids content of 37.5%, particle size of 0.225 microns, and a Brookfield viscosity of 1000 cps. The polymer demonstrated an intrinsic viscosity of 1.24 in dimethylformamide (DMF) at 30°C and produced films which were 55.1% and 52.8% insoluble upon adjustment to a pH of 2.7 and 8.0, respectively.[52]

An emulsion demonstrating good crosslinking ability was prepared from a similar monomer composition, but employed a more conventional polymerization procedure. The emulsion also contained 1.8% AMC in the final product.[53]

Emulsion systems based on the ethers of AMC, such as allyl N-ethoxymethylcarbamate, were claimed to produce coatings with greater crosslinking efficiency than emulsions based upon AMC. These products were recommended to be useful as binders for nonwoven fabrics, as impregnants for woven fabrics, as binders in printing pastes, and as laminating adhesives.[54]

N-Formyl-N'-Acryloylmethylenediamine, $CH_2 = CHCONHCH_2NHCOH$

N-formyl-N'-acryloylmethylenediamine (FAMD) is prepared by reacting 450 g formamide with 300 g paraformaldehyde for 1 hr at 110°C. The resulting N-methylolformamide was cooled to 40°C and condensed with 710 g acrylamide in two liters of cyclohexane containing 30 g of the monomethyl ether of hydroquinone and 75 ml concentrated HCl. Water was azeotropically distilled from the reaction mixture, and FAMD was produced in 97% yield. Self-crosslinking coatings were prepared from a 89.4/5.3/5.3-butyl acrylate/acrylonitrile/FAMD copolymer emulsion prepared at 46-50°C. The films displayed good flexibility and chemical resistance after curing at 95°C.[55]

Emulsions which were useful as coatings or as binders for textiles were prepared from a 72/23.3/3.5/1.2-butyl acrylate/acrylonitrile/N-formyl-N'-methacryloylmethylenediamine/N-MA copolymer which utilized 3.7% Mersolat H as the emulsifier. The polymerization was conducted at 63-65°C and yielded an emulsion with 29.8% solids and a pH of 6.7. Films cured at 90°C were tough and insoluble in water and organic solvents.[56]

VINYL AND ACRYLOXYSILANES, $CH_2 = CHSi(OR)_3$ AND $CH_2 = CHCO_2RSi(OR)_3$

Since the late 1960's, the coatings industry has attempted to produce "advanced generation" emulsions by combining silicone chemistry with emulsion technology. These coatings were sought for high-performance applications which demanded the superior coatings properties that silicone-containing systems uniquely provide. Silicone-based crosslinking emulsions would theoretically afford greater water and chemical resistance, superior gloss and gloss retention, excellent adhesion to metal and coated surfaces, enhanced scratch and mar resistance, improved impact resistance and flexibility, and greater heat resistance.

While much of the information on these systems remains proprietary, some of the advantages and limitations of silicone based emulsions are presented in the examples which follow.

In general, silicone-based crosslinking emulsions are most commonly produced from silane monomers such as vinyltrimethoxysilane, vinyltriethoxysilane, vinyltris(2-methoxyethoxy)silane, gamma-methacryloxypropyltrimethoxysilane and vinyltriacetoxysilane.[57] Most of these derivatives are available commercially as adhesion promoters.

Vinyltrimethoxysilane (VTMS) has been emulsion copolymerized with most of the common comonomers such as vinyl acetate, alkyl acrylates, and methacrylates and dialkyl maleates. The polymerizations usually employ various conventional addition techniques and utilize a mixture of nonionic and anionic surfactants. For example, a 94/5/1-vinyl acetate/2-ethylhexyl acrylate/vinyltrimethoxysilane copolymer emulsion was prepared at 55% solids and a viscosity of 300 cps by using a mixed surfactant system of 2% by weight of monomer Tergitol® NP-44 (a 40 mole ethylene oxide condensate of nonylphenol), 2% by weight Tergitol XD (a polyalkylene glycol ether with a surface tension of 38 dynes/cm in 0.1% aqueous solution), and 0.5% sodium lauryl sulfate. This emulsion was found to produce room temperature crosslinked films which were insoluble in water and common solvents. Paints based on this latex demonstrated a very high degree of scrub resistance at high pigment volume concentrations.[58]

VTMS has not been widely employed in free radical polymerizations since the vinyl group is not sufficiently activated by the silicone atom for efficient chain propagation. The unfavorable propagation kinetics of the vinyl group usually limit the amount of VTMS which can be incorporated into the copolymer to approximately 3% of the total copolymer composition. When the amount of VTMS exceeds 3% of the total monomer concentration, the reaction time usually increases while the percent conversion markedly decreases. For

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Tergitol is a registered trademark of Union Carbide Corp.

Table 14—Water Resistance Provided by Silicone-Crosslinked Emulsions

	Wet Tensile Strength (psi)	
Cure Schedule	**3.2% γ-MAPTMS**	**0% γ-MAPTMS**
10 min at 100°C	11	4
5 min at 130°C	15	5
3 min at 163°C	21	7

applications requiring greater crosslink densities or greater performance properties than those provided by systems containing 3% or less VTMS, gamma-methacryloxypropyltrimethoxysilane (γ-MAPTMS) is usually selected as the functional monomer as this derivative contains an activated double bond and is, therefore, both kinetically and thermodynamically more suited to free radical polymerization than is VTMS.

Emulsion copolymers have been prepared containing up to 50% of γ-MAPTMS without significantly deviating from the expected copolymer kinetics while maintaining high percent conversions.[59] Some of the more common comonomers used with γ-MAPTMS in latex compositions include alkyl acrylates and methacrylates, vinyl acetate, vinyl chloride, styrene, N-methylolacrylamide, and acrylonitrile.

Since γ-MAPTMS is hydrophobic in nature, the silicone functionality has no propensity to locate at or near the polymer-water interface. Therefore, seeding techniques or delayed monomer addition techniques are required to produce latex particles with a localized concentration of γ-MAPTMS in the shell area. A 41/38/18/2/0.3-ethylhexyl acrylate/vinyl chloride/vinylidene chloride/trimethylolpropane triacrylate/γ-MAPTMS copolymer was prepared by a monomer-feed technique whereby the addition of γ-MAPTMS was delayed until 80% of the monomers making up the particle core had been polymerized. A control latex of equivalent composition was similarly prepared except that γ-MAPTMS was excluded from the monomer charge. Bleached Kraft paper ($250\mu m$) having minimum fiber-fiber contact was dipped for 5 sec in the control and silicone latexes, respectively, and both samples were drip-dried for 24 hr at room temperature. The tensile strength data from both samples indicated that the emulsion containing γ-MAPTMS in the shell area produced coatings which were self-curing at room temperature.

The ability of γ-MAPTMS to crosslink at room temperature was also demonstrated by a 40.7/37.6/18.5/3.2-ethylhexyl acrylate/vinyl chloride/vinylidene chloride/γ-MAPTMS copolymer latex. The polymerization utilized a nonionic-anionic surfactant mixture of about 2.5% nonylphenoxy poly(ethyleneoxy) ethanol and about 3% alkyl aryl sodium sulfonate and produced a latex free of coagulum and possessing excellent mechanical stability. Films of this latex and a control were dried for 16 hr at room temperature and then immersed in trichloroethylene for 16 hr at 25°C. The films containing γ-MAPTMS were found to swell linearly 53%, while the control films which did not contain γ-MAPTMS dissolved in the solvent.

The tensile strength data from Kraft paper impregnates of the above latex and its control are presented in *Table* 14. These data reflect one of the major advantages offered by thermally crosslinked latexes containing γ-MAPTMS.[60]

Premature Crosslinking Of Siloxane Monomers

While these studies serve to illustrate some of the advantages offered by silicone-crosslinked emulsions, other investigations have demonstrated some of the weaknesses which have prevented the commercialization of alkoxysilane emulsions. For example, several attempts have been made to prepare emulsions of ethyl acrylate and vinyltriethoxysilane by means of the recipes listed in *Table* 15.

Although these recipes are based upon conventional practices and techniques and are similar to the procedures used in the previous examples, analysis of the

Table 15—Batch Polymerization Recipes for EA/VTES[a] Copolymers

Latex Composition	50/50-EA/VTES		67/33-EA/VTES
Quantity Used	**Ingredient**	**Quantity Used**	**Ingredient**
75 ml	Distilled water	75 ml	Distilled water
4.8 g	Tergitol NPX nonionic surfactant (HLB 13.5)	4.8 g	Triton X-200 surfactant (alkyl aryl polyether sulfonate)
0.8 ml	$FeSO_4$ solution (0.15%)	0.9 ml	$FeSO_4$ solution (0.15%)
0.2 g	Ammonium persulfate	0.2 g	Ammonium persulfate
0.28 g	Sodium metabisulfite	0.2 g	Sodium metabisulfite
2 drops	t-Butyl hydroperoxide	12 drops	t-butyl hydroperoxide
43 ml	Ethyl acrylate (0.02% monomethyl ether of hydroquinone)	43 ml	Ethyl acrylate
43 ml	Vinyltriethoxysilane	21.5 ml	Vinyltriethoxysilane

Reaction Temp. = 70°C.
(a) EA = Ethyl Acrylate.

Table 16—Elemental Analysis For Methacryloxyethoxytrimethylsilane

	Actual	Theoretical
% C	53.70	53.47
% H	9.00	8.91
% O	23.59	23.76
% Si	13.71	13.81

Table 17—Emulsion Polymerization Of 100 Percent Methacryloxyethoxytrimethyl Silane

Sample Number	% Surfactant	Latex Coagulation Time After Initiation	Polymer Solubility
1	3% Triton X-200	65 min	Insoluble in DMF
2	3% Tergitol NPX	74 min	Insoluble in DMF
3	2.25% Triton X-200 0.75% Tergitol NPX	38 min	Insoluble in DMF
4	2.25% Tergitol NPX 0.75% Triton X-200	35 min	Insoluble in DMF

resulting products indicated that a side reaction took place in preference to the desired polymerization. The conclusion was drawn that "the foregoing examples illustrate that solid (insoluble) copolymers are produced . . . by employing an emulsion polymerization system. By comparison, attempts to polymerize vinylalkoxysilanes (through the vinyl unsaturation) in emulsion systems have not met with success.

"The presence of water results in a hydrolysis-condensation reaction of the alkoxy groups which compete with any addition polymerization reaction of the vinyl groups. In all cases in which emulsion polymerization was carried out, only gel-like materials were obtained. Infrared analysis showed no diminution of the vinyl band intensity thereby indicating that the addition polymerization is very much slower than the hydrolysis and condensation reaction."[61] These observations are in agreement with other studies concerning the hydrolysis of trialkoxysilanes and the subsequent condensation of the silane triols which formed.[62,63]

In contrast to these results, a recent investigation has demonstrated the possibility of eliminating the hydrolysis-condensation reaction which occurs during emulsion polymerization by the selection of the proper silane monomer. This investigation utilized methacryloxyethoxytrimethylsilane (MAETMS) which is a monofunctional monomer containing a sterically shielded C-O-Si bond:

$$CH_2 = C(CH_3)CO(CH_2)_2OSi(CH_3)_3.$$

MAETMS is prepared by condensing 2-hydroxyethyl methacrylate with a 5% stoichiometric excess of hexamethyldisilazane. The monomer was distilled from the reaction mixture at 44°C and 0.225 mm Hg (reported B.P. = 45°C at 0.7 mm Hg) and yielded a product with a purity of 91% as determined by gas chromatography. The structure was substantiated by IR and NMR spectroscopy, and elemental analysis results on the purified product are given in *Table* 16.

The preliminary studies involved the homopolymerization of MAETMS in order to establish the parameters which influenced the emulsion polymerization of silane monomers, and to generate model silicone emulsions which would be utilized for further characterization and testing. The polymerization utilized a monomer to water ratio of 20/80, 3% surfactant, 0.1% t-dodecyl mercaptan chain-transfer agent, 0.2% ammonium persulfate initiator (percentages were based on weight of monomer), and a monomer addition rate in accord with the reflux method technique. The data from this study are presented in *Table* 17.

While Triton X-200 and Tergitol NPX may be recommended for the emulsion polymerization of VTMS or γ-MAPTMS (see the previous examples), neither surfactant would provide the stability required for the homopolymerization of MAETMS. As the data in *Table* 17 indicate, all attempts resulted in gross latex instability in addition to polymer insolubility. As a result of these impasses, the priority of developing model silicone emulsions was changed to that of generating stable silicone emulsions which were not crosslinked.

The first approach used to achieve this objective involved the copolymerization of MAETMS with n-butyl methacrylate (nBMA). nBMA was chosen because of its minimum film forming temperature (T_g = 19°C) and its similarity in side chain length to MAETMS. The polymerizations utilized the same ingredients and methods which were employed in the preparation of the homopolymer emulsions, except that the chain transfer agent was increased to 0.5% in order to promote solubility. The data for the copolymerizations are presented in *Table* 18.

These data indicate that copolymerization of MAETMS with nBMA was useful for increasing the latex stability but that copolymerization at the levels chosen had little effect on polymer solubility. However, when the 50/50-nBMA/MAETMS composition was

Table 18—Emulsion Polymerization of n-Butyl Methacrylate With MAETMS at 20% Solids

Monomer(s)	% Surfactant	% Coagulum	% Conversion	Polymer Solubility
100% nBMA	2.25% Triton X-200 0.75% Tergitol NPX	<0.2%	99.5%	Soluble in DMF
75/25-nBMA/MAETMS	2.25% Triton X-200 0.75% Tergitol NPX	1.2%	92.2%	Insoluble in DMF
50/50-nBMA/MAETMS	2.25% Triton X-200 0.75% Tergitol NPX	<1 %	—	Insoluble in DMF

Table 19—Physical Characteristics for nBMA/MAETMS Emulsions

Latex Composition[a]	Surfactants %	Coagulum %	Conversion %	Solids %	Acetone Insolubles %	Silicone Content	
						Theoretical %	Actual %
100% nBMA	2% Triton QS-44	17.5	99+	40.7	0	0	0
87.5/12.5 nBMA/MAETMS	2.25% Triton X-200 0.75% Surfactant L-77	19.7	99+	47.5	0	1.73	1.90
75/25 nBMA/MAETMS	2.25% Triton X-200 0.75% Surfactant L-77	< 1.0	91.2	46.5	0	3.46	3.34
62.5/37.5 nBMA/MAETMS	2.25% Triton X-200 0.75% Surfactant L-77	< 0.5	89.2	46.1	0	5.09	3.93
50/50 nBMA/MAETMS	2.25% Triton X-200 0.75% Surfactant L-77	< 0.5	94.9	50.9	69.4	6.90	4.12

(a) nBMA = n-butyl methacrylate.

polymerized at 50% solids instead of 20% solids, the latex no longer retained its colloidal stability and coagulated after 81 min of reaction time.

Since the emulsifier system used in these polymerizations was found to stabilize particles containing 6.9% silicone (50/50-nBMA/MAETMS) but not those containing 13.84% silicone (100% MAETMS), the colloidal instability of these emulsions was assumed to arise from an unfavorable interaction between the surfactant molecule and the particle surface. For example, if the interaction potential between the particle surface and the surfactant was less than the interaction potential between the surfactant and water molecules, the particle to particle repulsive forces associated with the Zeta layer or an interfacial film layer would be insufficient to prevent particle agglomeration. In accord with this assumption, a surfactant possessing a high affinity for a silicone surface would afford greater colloidal stability than a surfactant which was partitioned between the particle surface and the aqueous phase. Based upon these premises, a survey was conducted of the commercially available silicone surfactants; and of the possible choices available, Union Carbide's surfactant L-77 was chosen as the most suitable candidate for preliminary studies.

Surfactant L-77 is a nonionic surfactant based on an organosilicone block copolymer. It is soluble in water up to 0.1% by weight, and is very efficient at lowering the surface tension of aqueous systems. A 0.01% solution of surfactant L-77 displays a surface tension of 21.3 dynes/cm.

Surfactant L-77 was evaluated in the polymerization of a 50/50-nBMA/MAETMS copolymer at 50% solids. In accordance with the previous polymerizations, the surfactant mixture consisted of 2.25% Triton X-200 and 0.75% surfactant L-77. The polymerization was quite successful and produced a latex displaying no coagulum, 94.9% conversion, and a solids content of 48.3%. The enhanced stability provided by surfactant L-77 was reflected by the emulsion's ability to pass one freeze-thaw cycle.

Based upon the success provided by surfactant L-77, several attempts were made to produce homopolymer emulsions of MAETMS at 50% solids. The polymerizations used 0.75/2.25 and 1.5/1.5 weight ratio percentages of surfactant L-77 to Triton X-200. Since all these polymerization attempts resulted in complete coagulation of the latex, further efforts to produce homopolymer emulsions of MAETMS were not pursued.

Several other studies were conducted which utilized other comonomers such as ethyl acrylate and n-butyl acrylate at 50% comonomer levels, and other surfactants such as Duponol WAQE and Triton QS-44. Unfortunately, all these attempts also resulted in total coagulation of the latex.

Obviously, the parameters associated with the colloidal stability of silicone latex particles were difficult to isolate, and a considerable effort would be required to establish the relationship between the compositional nature of the latex surface and the compositional nature of the surfactant. While such an investigation was surely warranted, the need to investigate the nature of the crosslinking reaction and the need to substantiate the stability of the Si-O-C bond during emulsion polymerization were given greater priority at this stage of the investigation.

The stability of the Si-O-C bond during emulsion polymerization was investigated by preparing a compositional ladder of nBMA/MAETMS emulsions. The characteristics of these emulsions are presented in *Table* 19.

The silicone analysis data indicate that emulsions containing in excess of 25% MAETMS experienced a considerable loss in initial silicone content. The formulations employing monomer feeds of 37.5% and 50% MAETMS generated copolymers containing only 28.4% and 29.7% MAETMS, respectively. The 50/50-nBMA/MAETMS copolymer was found to be crosslinked, containing 69.4% acetone-insoluble polymer. These data suggest that a specific concentration of comonomer (nBMA) is required in order to prevent loss of MAETMS. This concentration may reflect the amount of hydrophobic monomer required to establish a hydrolysis barrier layer between MAETMS and the aqueous phase.

The crosslinking reaction which occurred during the polymerization of the 50/50-nBMA/MAETMS copolymer was not in agreement with theoretical considerations involving hydrolysis of the Si-O-C bond. Since

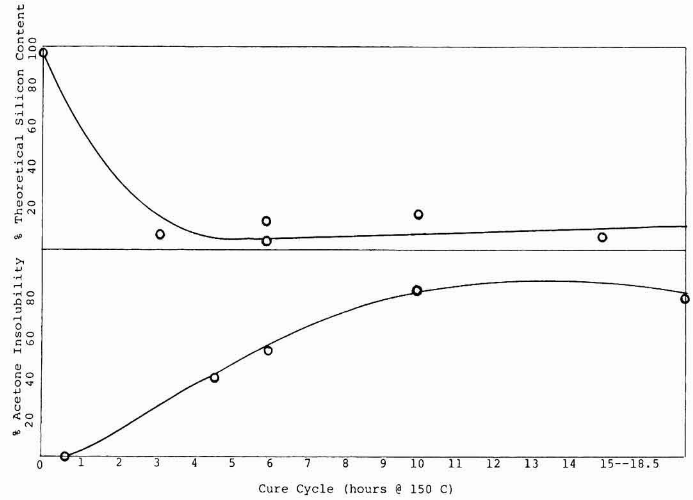

Figure 1—Effect of thermal curing on the silicone content of a 75/25-nBMA/MAETMS copolymer

MAETMS is a monofunctional silyl ether, hydrolysis of the Si-O-C bond should not result in the development of a chemical crosslink (see monomer structure).

The crosslinking ability of MAETMS was investigated by subjecting films of uncrosslinked polymer to normal curing conditions. Films of the 75/25-nBMA/MAETMS latex were cast and dried at ambient conditions and at 150°C for 3 hr. The ambient cured films were found to be soluble in acetone and contained 96.5% of the theoretical amount of silicone, while the sample which was cured at 150°C for 3 hr was insoluble in acetone and DMF and contained only 7.2% of the theoretical amount of silicone. These data were also supplemented with soxhlet extraction determinations in order to establish a clearer relationship between residual silicone content and percent insoluble polymer as a function of the curing conditions. The complete data are presented in *Figure* 1.

While systems based on MAETMS were not expected to produce crosslinked polymer upon cleavage of the Si-O-C bond, the data presented in *Figure* 1 imply that a direct relationship exists between siloxane cleavage and crosslink density. The remaining MAETMS based emulsions produced similar results upon curing at 150°C as illustrated by the partial data presented in *Table* 20.

Table 20—Effect of Thermal Curing On Solubility and Silicone Content

Composition MAETMS/nBMA	% Insoluble Polymer before Curing[a]	% of Theoretical Si before Curing	% Insoluble Polymer after Curing[a]	% of Theoretical Si after Curing
12.5/87.5 ...	0	100	6.8	6.9
25/75	0	96.5	55.8	11.6
37.5/62.5 ...	0	75.8	73.2	15.1
50/50	69.4	60.3	>88.2	7.0

(a) The curing conditions were 6 hr at 150°C.

While it is possible that the silicone losses which occurred upon thermal curing and polymerization may be mechanistically unrelated, the most probable crosslinking mechanism for this system would involve the hydrolysis of polymethacryloxyethoxytrimethylsilane to poly (2-hydroxyethyl methacrylate) and hydroxytrimethylsilane, followed by the self-condensation of each of these products.

If this mechanism applied, ethylene glycol and hexamethyldisiloxane (B.P. 98.5°C) would be the expected by-products of the condensation step. However, thermal evolution-collection studies did not detect the presence of either product.

The hydrolysis-condensation mechanism is predicated upon the transesterification of two 2-hydroxyethyl methacrylate groups in order to effect the crosslinking step. However, since the crosslinking reaction was found to take place as low as 65°C, it is unlikely that such mild conditions would be activating enough for transesterification to occur.

Also, emulsions containing MAETMS showed unexpected resistance to hydrolysis. Latexes stored at 60°C for three months showed no change in solubility or percent silicone content, and the emulsions retained their initial freeze-thaw and mechanical stability.[64]

It is obvious that these results are not in agreement with the hydrolysis-condensation polymerization proposed for vinyltrimethoxysilane[61] since hydrolytically stable polymer of methacryloxyethoxytrimethylsilane was prepared by means of emulsion polymerization. However, the mechanism for the thermal crosslinking of MAETMS will remain speculative until more conclusive data can be generated.

Autooxidizable-Vinyl Emulsions

Vinyl-type systems prepared with autooxidizable oils are considered to be the first developments of a new

Table 21—Air-Drying Ability of Uncatalyzed and Catalyzed Films Containing Various Autooxidizable Monomers

Autooxidizable Monomer	Curing Agent Wt. Percent[a]	Sward Hardness (24 hr)	Sward Hardness (2 weeks)	Percent Benzene Insolubles[b]
10% 2-Ethylhexyl methacrylate (control)	0.05 Co,1.0 MEKP	6	14	0
10% Tetrahydrofurfuryl methacrylate	0.05 Co,1.0 MEKP	6	24	0
10% Allyloxyethyl methacrylate	(Resin gelled during synthesis)			
10% Methylcyclohexenylmethyl methacrylate	None	10	22	0
10% Methylcyclohexenylmethyl methacrylate	0.05 Co	12	32	48
10% Methylcyclohexenylmethyl methacrylate	0.05 Co,1.0 MEKP	12	30	60
10% Cyclohexenylmethyl methacrylate	0.05 Co,1.0 MEKP	8	24	67
10% Cyclohexyl methacrylate (control)	0.05 Co,1.0 MEKP	8	22	0

(a) Co = cobalt naphthenate, MEKP = methyl ethyl ketone peroxide.
(b) 6 hr of soxhlet extraction after 28 days of ambient aging.

generation of emulsion systems designed to meet general industrial requirements. Since these emulsions are produced under conditions which closely control particle size while optimizing polymer molecular weight, the resulting products display excellent exterior weatherability, very good flexibility, and improved solvent and mar resistance.[65] In comparison with systems based on other functional monomers, vinyl-type emulsions containing autooxidizable oils offer such advantages as:

(1) a raw-material basis which is replenishable and quasi-independent of petroleum price structure and supplies;

(2) reduced product-development expenditures resulting from the extensive resources available on the science and technology of autooxidizable systems;

(3) pre-established consumer awareness and acceptance resulting from other oil-based products;

(4) greater marketing appeal in applications such as on-site construction where oven curing is impractical;

(5) greater product uniformity resulting from the nature of the crosslinking groups and their distribution in the microstructure;

(6) improved product integrity resulting from enhanced particle to particle coalescence;

(7) improved adhesion in direct-to-metal applications as well as over primed surfaces;

(8) greater water and chemical resistance resulting from the relatively nonpolar nature and bond stability of carbon to carbon and carbon to oxygen crosslinks;

(9) greater product flexibility and impact resistance as a result of the rheological characteristics of oil systems;

(10) improved pigment acceptance and wettability of the product; and

(11) improved corrosion resistance in primer and enamel applications.[66]

The first emulsion of this type to be offered commercially was Arolon® 110-W-45. This product is based upon an alkyd-vinyl-acrylic copolymer which is produced by means of emulsion polymerization and possesses a particle size in the range of 0.03 to 0.1 microns. The alkyd portion of the copolymer is based upon soya and linseed oils, and films of this system develop good adhesion and excellent water resistance. Arolon 110 displays good air-drying qualities but is not recommended in applications requiring conventional thermal curing.[67]

Several polymerization techniques have been used to develop various vinyl-type emulsions containing autooxidizable oils. For example, an air-drying vinyl emulsion was prepared by copolymerizing vinyl acetate in the presence of an alkyd resin containing maleic acid unsaturation. The technique consisted of adding a pre-emulsion containing 76.2% alkyd resin, prepared from safflower oil, ethylene glycol and maleic acid, and 23.8% vinyl acetate to a reaction mixture consisting of vinyl acetate monomer, surfactant, and initiator dispersed in water. The resulting emulsion contained 55% vinyl acetate and 45% safflower oil alkyd and demonstrated excellent exterior weatherability and molecular weights over 50,000.

Air-drying acrylic emulsions have been prepared by copolymerizing the unsaturation of a linseed-tung oil alkyd resin with methyl methacrylate. The procedure consisted of polymerizing a linseed-tung oil-pentaerythritol-isophthalic acid alkyd dissolved in xylene with methyl methacrylate using benzoyl peroxide as the initiator. The mixture was polymerized until 50% of the methyl methacrylate had reacted. A solution of water, nonionic surfactant, and lithium hydroxide was then added to the reaction mixture to form a water-in-oil emulsion. After 55% of the methyl methacrylate had reacted, the reaction temperature was slightly lowered whereupon the water-in-oil emulsion inverted into an oil-in-water emulsion. The polymerization was continued until 80% of the monomer was consumed. The resulting product contained 61% alkyd resin and 39% methyl methacrylate. The above examples are discussed more completely in the section on post-emulsified alkyd resins.

Other examples which illustrate the applications, properties, technology, and diverse compositional nature of these systems are given as follows:

Air drying emulsions useful as paints or adhesives were prepared by copolymerizing linseed or isomerized linseed oil with vinyl acetate. Isomerized linseed oil

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Arolon is a registered trademark of Ashland Chemical Co.

was found to inhibit the free radical copolymerization of vinyl acetate and yielded emulsions with up to 80% conversion of vinyl acetate after 8 to 10 hr of reaction time and with initiator concentrations greater than 2.5%. These products displayed a marked improvement in water resistance as a result of the unassociated linseed oil which remained free from the vinyl acetate-isomerized linseed oil copolymer.

Bodied linseed oil was found to chain-transfer less than unmodified linseed oil in its copolymerization with vinyl acetate. The resulting emulsions contained less unassociated linseed oil and attained compositions with higher percentages of vinyl acetate than emulsions prepared with unmodified linseed oil. The bodied linseed oil-vinyl acetate copolymer emulsions showed little improvement in water resistance as compared to unmodified poly(vinyl acetate) emulsions.

The copolymerization of unmodified linseed oil with vinyl acetate yielded products which were also a mixture of copolymer and homopolymer. The copolymer was soluble in ether and benzene and contained 5% to 15% vinyl acetate. The polymerizations employed a surfactant mixture consisting of poly(vinyl alcohol) with a Hoeppler viscosity of 50 cps and 0.5% sodium alkylbenzene sulfonate and achieved conversions of 100% vinyl acetate at 1% to 2% initiator. The proportion of linseed oil to vinyl acetate was varied from 0% to 44.5%, and the copolymer containing the optimal content of 30% linseed oil was found to provide films with 20 times the water resistance of films containing no linseed oil.[68]

Other Methods of Preparation

Since the free radical polymerization of vinyl monomers with drying oils is kinetically unfavorable and yields products displaying moderate to marked incompatibility, various procedural and chemical modifications have been used to alter the reaction kinetics, thereby producing copolymers with greater compositional uniformity and less incompatibility. In general, procedural modifications such as delayed addition of vinyl monomer, bulk or solvent copolymerization, or chain-transfer promoted reactions have not given the compositional uniformity of systems which utilize drying oils modified with vinyl groups capable of chain propagation. For example, nonconjugated drying or semi-drying oils can be condensed with alpha-beta ethylenic dicarboxylic acids to form products which yield homogenous, compatible copolymers when polymerized with such vinyl monomers as styrene. The vinyl-modified drying oil is prepared by condensing such compounds as maleic anhydride with a partial fatty acid ester of a polyhydroxyl alcohol. The hydroxyl-functional partial ester is prepared by the alcoholysis of a triglyceride with a polyol such as pentaerythritol, sorbitol, or glycerol. The triglyceride is any nonconjugated semi-drying or drying oil such as linseed oil, soybean oil, perilla oil, cottonseed oil, or safflower oil. The alcoholysis is preferably carried out at 193°C and is catalyzed by the addition of from 0.03 to 0.3% zinc, cadmium, or lead oxides, hydroxides, or acetates. The esterification of maleic anhydride with the hydroxyl-functional, partial fatty acid ester is conducted below 182°C in order to avoid such side reactions as addition of allyl positions on the fatty acid to the maleic unsaturation. The maleic-modified drying oil may then be free-radically polymerized with common vinyl monomers. Usually the vinyl monomer constitutes between 30% to 60% of the total weight of the final product. The modified-oil copolymers are compositionally homogenous and form clear films which display fast, hard drying properties, good water and alkali resistance, and excellent color and gloss retention. The coatings may be used in a wide variety of industrial applications such as in machinery enamels or as finishes for hardware.[69]

Maleic-modified drying oils have also been polymerized with vinyl monomers to produce emulsions which were useful as interior flat finishes, primer sealer coatings, semi-gloss and gloss enamel finishes, and industrial coatings. The emulsions were prepared as follows: a conventional polymerization apparatus was charged with 31.37 parts tap water and a surfactant mixture consisting of 0.40 parts sodium tetradecyl sulfonate, 1.48 parts of a Pluronic-type nonionic surfactant and 19.0 parts of a water-soluble, oil-based oxidizing emulsifier. The oil-based emulsifier consisted of a 83/17-linseed oil/cyclopentadiene copolymer which was reacted with 10% maleic anhydride by means of a Diels-Alder condensation. The copolymer was water solubilized by neutralizing with ammonia. The pH of the water surfactant mixture was adjusted to 8.1 to 8.4 with ammonium hydroxide. Twenty-three parts of a maleic-modified, drying-oil monomer prepared from soybean oil alcoholyzed with pentaerythritol and esterified with 5% maleic anhydride were blended with 3.03 parts vinyl toluene, and the mixture was added to the water-surfactant solution over a 30 min period at a temperature of 57-63°C. After the addition, the reaction temperature was raised to 66-72°C, and the reaction mixture was initiated with 0.37 parts benzoyl peroxide and 0.25 parts potassium persulfate. The reaction temperature was then raised to 86-88°C, and 9.1 parts of vinyl toluene were added over a 2 hr period. A post-initiation mixture consisting of 0.73 parts potassium persulfate and 0.44 parts sodium hydroxide dissolved in 10.46 parts water were added simultaneously with the addition of vinyl toluene. After all the ingredients had been added, the reaction temperature was raised to 96°C and held for 1.5 hr to ensure complete conversion of the monomers. The resulting emulsion displayed excellent stability and produced films possessing excellent gloss, clarity, and resistance to water. In general, these emulsions yielded films which were fast drying at room temperature and provided improved hardness, adhesion, flexibility, toughness, and chemical resistance. Because the oxidizing oil-based emulsifier, which constitutes a major portion of the surfactant mixture, loses its water solubilizing characteristics as it becomes an integral part of the film during the drying process, these products were found to display superior water resistance.[70]

Table 22—Effectiveness of Allyl Methacrylate Based Coatings in Primer Applications

		Allyl Methacrylate Primer		Epoxy Ester Primer	
Exposure Site	**Environmental Resistance**	**Alkyd Topcoat**	**Acrylic Topcoat**	**Alkyd Topcoat**	**Acrylic Topcoat**
Florida	Concave dents				
	Initial No. of Cracks[a]	2	4	1	3
	No. Cracks after Exposure[b]	2	4	1	4
	No. Cracks with Rusting[c]	2	4	0	0
	Convex dents				
	Initial No. of Cracks	7	6	6	6
	No. Cracks after Exposure	7	6	6	6
	No. Cracks with Rusting	7	6	6	3
	Creep under score (in.)	1/32	0	1/32	0
Delaware	Concave dents				
	Initial no. of cracks	1	4	0	4
	No. cracks after exposure	1	4	0	4
	No. cracks with rusting	1	0	0	0
	Convex dents				
	Initial no. of cracks	5	7	4	7
	No. cracks after exposure	5	7	5	8
	No. cracks with rusting	5	1	3	0
	Creep under score (in.)	1/16	1/16	1/8	1/8

(a) Number of cracks resulting from denting.
(b) Number of cracks which developed upon exposure.
(c) Number of cracks developing rust upon exposure.

Air-drying emulsions of similar composition have also been prepared using nonionic surfactants as the sole emulsifiers. For example, a vinyl-modified, drying-oil monomer was prepared by condensing the alcoholysis product of safflower oil and ethylene glycol with maleic anhydride. The maleic-modified drying oil was then polymerized with vinyl acetate using Igepal CO-990 as the surfactant. The resulting emulsion yielded tough, air-drying films whose properties resembled those of a vinyl plastisol.[71]

In addition to maleic-modified drying oils, other monomers capable of room temperature, oxidative coupling have also been used to develop air-drying emulsions. For example, emulsion coatings which cured at room temperature were prepared by blending a vinyl-type emulsion which employed a maleinated polybutadiene resin as a dispersing agent with a water-dispersible copolymer consisting of an autooxidizable monomer, methacrylic acid, and butyl methacrylate. The autooxidizable monomer was prepared by condensing 236 parts safflower oil fatty acid with 119 parts glycidyl methacrylate (GMA). The copolymer contained 44% butyl methacrylate, 40% of the safflower oil fatty acid-GMA condensate, and 16% methacrylic acid. Twenty-five parts of the copolymer containing the autooxidizable monomer were blended with 430 parts of a 40% solids butyl methacrylate emulsion. Films of the blend were cured five days at 20°C and 75% humidity to yield a product which was 78% insoluble after a 24 hr immersion in THF. Similar films which omitted the emulsion were only 4% insoluble after the same treatment.[72] Obviously, films with greater insolubility could be achieved by incorporating the safflower oil fatty acid-GMA adduct into the acrylic emulsion.

Oil-Free, Autooxidizable Emulsions

Air-drying coatings which displayed improved hardness and solvent resistance were prepared from emulsion copolymers containing various derivatives of cyclohexenylalkyl methacrylate. The monomer was prepared from an isomeric mixture of alcohols consisting of 3-methyl-4-hydroxymethyl cyclohexene and 3-methyl-5-hydroxymethyl cyclohexene which were obtained from a Diels-Alder condensation of piperylene and allyl alcohol at 220°C. Forty-three g of the isomeric alcohol mixture was refluxed with 100 g methyl methacrylate for 6 hr in the presence of 3.5 g of a 5% solution of sodium in methanol and 2.5 g p-methoxyphenol. Methanol was removed by azeotropic distillation during the reflux period. After distillation the mixed isomers of cyclohexenylmethyl methacrylate were obtained in 94.5% yield. A 60/30/10-ethyl acrylate/methyl methacrylate/methylcyclohexenylmethyl methacrylate copolymer emulsion (mole percent) was prepared in the conventional manner using 2.9% Triton X-200 as the polymerization surfactant. The resulting emulsion possessed a solids content of 36% and produced clear films at room temperature.

Coatings containing 10 mole-percent methylcyclohexenylmethyl methacrylate were found to air-dry slowly at ambient conditions. The cure time could be shortened by thermal curing or by the addition of a metal drier. *Table* 21 illustrates the effects of catalysis on the air-drying properties of coatings containing methylcyclohexenylmethyl methacrylate in comparison with coatings containing other autooxidizable monomers. Each system was comprised of 45% methyl

While the procedures used to polymerize methylcyclohexenylmethyl methacrylate were found to be unsuitable for the polymerization of allyloxyethyl methacrylate, emulsion polymerization techniques have been used to produce air-drying products containing allyl methacrylate or crotyl methacrylate. For example, a 60/30/10-ethyl acrylate/methyl methacrylate/allyl methacrylate copolymer emulsion was prepared using 4.5% polyoxyethylene lauryl ether as the surfactant. The emulsion was prepared at 5°C using a redox initiator combination consisting of 0.04% p-menthane hydroperoxide, 0.02% ferrous sulfate, and 0.036% sodium formaldehyde sulfoxylate. The resulting latex possessed a solids content of 46.5%, 96% conversion, a pH of 3, and a particle size of 0.09 microns. Films of the latex which were air-dried at room temperature for 1-2 hr displayed a Sward hardness of 12 and were soluble in acetone. After the films were cured 30 min at 150°C, the Sward hardness increased to 20, and the coating became insoluble in acetone.

The latex was formulated into a paint having a pigment volume concentration of 20%, a pigment-vehicle ratio of 50/50 by weight, a pH of 8.5, and a solids content of 55%. The paint was evaluated in comparison with a commercial epoxy-ester formulation for metal primer applications. The allyl methacrylate based primer was cured one hr at 150°C while the epoxy-ester primer was cured 30 min at 163°C. Both primers were then topcoated with an acrylic lacquer or an aminoplast modified alkyd resin, and the coatings were cured 1 hr at 107°C and 30 min at 121°C, respectively. The coatings were then subjected to five months of exterior exposure in Florida and Delaware and evaluated for adhesion, flexibility, and corrosive creep. The results of this evaluation are presented in *Table* 22.

While these results show some variations in the resistance properties provided by the two systems, the emulsion containing allyl methacrylate was felt to provide properties which were competitive with those afforded by conventional epoxy-ester coatings.[74]

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(56) Ribka, J., et al., German Patent 2,251,922; *Chem. Abstr.* 81:92226b.
(57) Plesich, M.F. and Zavaglia, E.A., U.S. Patent 3,729,438; *Chem. Abstr.* 79:6911a.
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Society Meetings

Los Angeles June 14

A moment of silence was observed in memory of Walter G. Ballinger, President of the Golden Gate Society for Coatings Technology, who died recently.

Walter Hunt, of Guardsman Chemicals, and Tom Rumfola, of TCR Industries, were presented with 25-year pins.

Robert Lipscomb, of Engard Coatings Corp., spoke on "THE ALASKA PIPELINE."

Mr. Lipscomb, who previously served as a field engineer on the Alaska pipeline project for Bechtel Co., discussed some of the problems encountered in that work, including temperature extremes of 100° to −80°F. He mentioned that above ground pipes and support structures were initially coated with ethyl silicate-based inorganic zinc, but some had to be removed because of inadequate infrared absorbance and reflectance. Below ground pipe was originally finished with powder coating, which was not completely satisfactory. Of sub-surface pipe, 30% is now coated with inorganic zinc. The pipeline has a 30-year life expectancy.

The thickness of steel used in the pipeline varies from 0.45 to 0.52 in., he said. The pipeline floats on Teflon and in case of an earthquake, it is designed to move laterally for 1½ miles in any direction, or up to 180 feet vertically, Mr. Lipscomb concluded.

ALBERT SENEKER, *Secretary*

Officers of the St. Louis Society for Coatings Technology for the year 1977-78. Left to right: Vice-President—John W. Folkerts; President—Morris Gilliam; Treasurer—Floyd Thomas; Council Representative—Howard Jerome, and Secretary—Herbert Rosenblatt

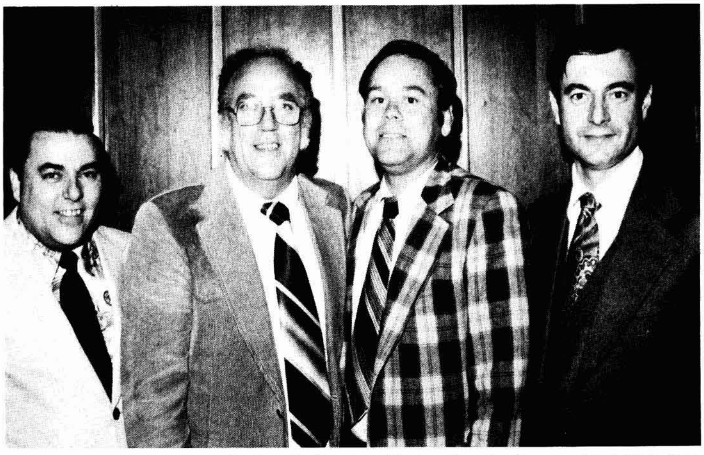
Piedmont June 21

The following slate of officers was elected for the year 1978-79: President — Charles Wilson, of Reliance Universal, Inc.; Vice-President — John Hajnos, of Ashland Chemical Co.; Secretary — William Cunane, of Sherwin-Williams Co.; and Treasurer — James Albright, of the Lilly Co.

Milton E. Woods, of ICM Corp., spoke on "USE OF AMINO ALCOHOL AS A RESIN SOLUBILIZER."

Mr. Woods said that water solubilization is obtained by reacting an amine with an acid salt. A big difference in the polymer occurs when you change the acid, i.e., an oil free alkyd, or an acrylic. A strong or weak amine also makes a big difference in the polymer. Base strength, power as a solubilizer is an important consideration. AMP is an amine alcohol and is a strong base. Ammonia is not a strong base. Another important consideration is amine efficiency. Some buffer to a high pH, some to a low pH. The amine efficiency factor determines whether to replace one amine with another. Because different amines behave in different ways, blends are often used. AMP presents a problem in air dry since it does not evaporate quickly, and is usually blended with ammonia. Various factors such as water resistance, hardness, dry time and dip tank stability must be considered.

JOHN V. HAJNOS, *Secretary*



Officers of the New England Society for Coatings Technology for the year 1977-78. Left to right: Vice-President—George Mulvey; Treasurer—John Guiffrida; President—Paul Mueller; and Secretary—Martin Davis

Pittsburgh June 5

Bonnie Bender, of PPG Industries, Inc., in a two-part presentation, spoke on "COLOR POWER: HOW TO MAKE IT WORK FOR YOU" and "COLOR TRENDS MIRROR CHANGING LIFESTYLES."

Mrs. Bender said that human consciousness is a mixture of memory, reason, attention, and conditioning — an intermingling of the senses — that influence our reaction to the light stimulus which we call color. She said that the power of color can be harnessed for our emotional well-being and general welfare. A telephone company that painted phone booth interiors yellow, so that people would happily shorten their phone calls, and workmen who had more trouble lifting black boxes which were lighter in weight than similar white boxes were two examples of the psychology of color cited by Mrs. Bender.

An illustrated segment on color trends related to current and changing lifestyles in the 20th century concluded the presentation.

ROBERT T. MARCUS, *Secretary*

Elections

BALTIMORE

Active

Mitnick, Howard M. Jr. — Duron Paint Co., Beltsville, Md.
Dowd, Theodore — M.S. Coast Guard., Washington, D.C.
Grumbine, Theodore C. — Jotun-Baltimore Copper Paint Co., Baltimore, Md.

Associate

Joyner, James M. — Harry T. Campbell Sons Co., Towson, Md.
Webster, Jeff — IBM Corp., Baltimore, Md.
Breen, Dan L. — Engelhard Minerals., Arnold, Md.
Carroll, David A.C. — General Mills Chemical's, Newark, Del.

Retired

Gemmill, Lewis T. — Carroll Valley, Fairfield, Pa.

BIRMINGHAM

Active

Stephen, Evans — Arthur Holden and Sons Ltd., Birmingham, Eng.
Harris, Richard R. — Batchelor Robinson Coatings Ltd., Aldridge, Staffordshire.
Griffiths, Malcolm H. — Batchelor Robinson Coatings Ltd., Aldridge, West Midlands
Sadler, Edward J. — Vinyl Products Ltd., Carshalton, Surrey.

GOLDEN GATE

Active

Li, Antony T. — Technical Coatings Co., Santa Clara, Calif.

Associate

Bibo, Ronald I. — Hill Brothers Chemical Co., San Jose, Calif.

MEXICO

Active

Bruschweiler, Peter — CIBA-GEIGY Mexicana, S.A., deC.V., Mexico, 21, D.F.

NEW ENGLAND

Active

Arsenault, Warren J. — Cabot Corp., Billerica, Mass.
Hampl, Eva M. — Cabot Corp., Billerica.
Tilley, Melvin G. — Cabot Corp., Billerica.

Associate

Bowers, Charles R. — R.B. Huber Assoc., Watertown, Mass.
Hoar, Charles J. — Union Chemicals Div., Dedham, Mass.
Townsend, Jerd A. — Herbert B. Lewis Co., Brookline, Mass.

NORTHWESTERN

Active

Peters, Jeffrey R. — Cargill, Inc., Minneapolis, Minn.
Prenosil, Michael B. — Champion International, St. Paul, Minn.
Richards, Thomas M. — IBM Corp., Rochester, Minn.

Associate

Kenefick, Emmett — Thompson Hayward Chemical Co., Minneapolis, Minn.
Swanson, Willard N., Jr. — ChemQuip Corp., Minneapolis.

Technical Articles in Other Publications

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

Double Liaison—Chimie des Peintures

(in French)

Published by Les Presses Continentales, Rue de Cherch-Midi, F-75006, Paris, France

Vol. 25 No. 272 April 1978

Rique, J.P.—"Filiform Corrosion in Airplane Paints;" 23-27.
Van Tongeren, H.P.—"Acrylic Polyols for Two-Component Isocyanate Systems;" 28-34.
McKay, R.B.—"Rheological Characteristics and Colloidal Chemical Nature of Organic Pigment Dispersions;" 35-44.

Vol. 25 No. 273 May 1978

Bonnet, E., Gangneux, Ph., and Marechal, E.—"Synthesis and Study of Photocurable Coloured (Materials) Structures from Oligomers;" 34-38.
Caze, C. and Loucheux, C.—"Study of Alternated Copolymerization of the Monomers Maleic Anhydride/Vinyl Acetate;" 40-47.
Clouet, G. and Brossas, J.—"Macromolecular Fire Resisting Additives;" 48-54.
Boutevin, B., Pietrasanta, Y., and Sideris, A.—"Chemical Modification of Novolac Resins and Polyvinyl Alcohol from Fluorinated Compound Addition;" 56-65.
Anthoine, J.C. and Vernet, J.L.—"Synthesis of Butadiene Oligomers from Telomerisation;" 66-69.
Tshizubu, P.M., Busnel, J.P., and Bruneau, C.M.—"Synthesis and Characterization of Polyol Poly-Caprolactones;" 70-77.
Revillon, A. and Anton, A.—"Phthalic Anhydride/Glycerol Polycondensation. Kinetic Study and Characterization;" 78-87.
Jullien, H., Berteaud, A.J., Henry, F., and Petit, J.—"Macromolecular Film Hydration Studies by Microwave Dielectric Absorption;" 88-93.
Anon.—"French Paint and Varnish Industry and the Recession;" 105-110.

Farbe und Lack

(in German)

Published by Curt R. Vincentz Verlag, 3 Hannover, Postfach 6247, Schiffgraben 43, Germany

Vol. 84 No. 6 June 1978

Bachmann, H.G.—"Recent Developments in Environmental Legislation and Their Effects on the German Paint Industry;" 385-386.
Paul, D.M., Venselaar, H.C.J., and Sketchley, J.M.—"Apparatus for Determining the Sterilization Stability of Can Lacquers;" 387-388.
Funke, W.—"Assessment of the Corrosion Protection Characteristics of Paint Films and other Organic Coatings. (1) Corrosion Test Methods and the Salt Spray Test;" 389-391.
Gross, H.—"Salt Deposits on Corrosion Protection Coatings;" 391-394.
Sütö, J., Zador, M., Winkler, G., and Szebenyi, I.—"Steam Permeability of Exterior Wall Paints;" 394-398.
Snuparek, J., Jr.—"Water Absorption in Films of Acrylic Resin Latices;" 399-403.
Bagda, E.—"Adhesion Between Polymers;" 403-404.
Brushwell, W.—"Development Tendencies in Electrocoating" (Literature review); 405-407.
German Standard Draft DIN 55 977: Testing of Soluble Coloring Matters; Determination of Marginally Insoluble Components; 412-414.
Jostes, K.-H.—"Employment of Safety Engineers and Other Specialists for Works Safety;" 429-431.

I & E C Product Research and Development

Published by American Chemical Society, 1155—16th Street N.W., Washington, D.C. 20036

Vol. 17 No. 2 June 1978

Symposium on Interfacial Phenomena in Corrosion Protection

Muzyczko, T.M.—"Review of Electrochemical Corrosion Fundamentals;" 169-176.
Ashworth, V., Grant, W.A., and Procter, R.P.M.—"Novel Corrosion Resistant Alloys by Ion Implantation;" 176-182.
van Eijnsbergen, J.F.H.—"The Interface: Zinc—Coated Steel—Paint;" 183-184

Journal of the Oil and Colour Chemists' Association

Published by Oil and Colour Chemists' Association, Priory House, 967 Harrow Rd., Wembley, Middlesex, HA0 2SF England

Vol. 61 No. 6 June 1978

Rybny, C.B. and Vona, J.A.—"New Developments in Ultraviolet Curable Coatings Technology;" 179-188.
Holman, R. and Rubin, H.—"Cure Behaviour of Photopolymer Coatings;" 189-194.
De Poortere, M., Ducarme, A., Dufour, R., and Merck, Y.—"Photoinitiator Problems in Clear Coatings;" 195-203.

Paint Manufacture

Published by Wheatland Journals Ltd., 157 Hagden Lane, Watford WD 1, 8 LW, England

Vol. 48 No. 4 May 1978

Walton, A.J.—"Interactions in Non-Aqueous Paint Systems;" 13-14.

Plaste und Kautschuk mit Fachteil Anstrichstoffe

(in German)

Published by VEB Deutscher Verlag für Grundstoffindustrie, 27 Karl-Heine Strasse, 7031 Leipzig, E. Germany DDR

Vol. 25 No. 5 May 1978

Bashirov, A.B., Selenev, Yu. V., and Molotkov, A.P.—"Theoretical Basis for Predicting Polymer Properties;" 257-59.
Noack, R. and Schwetlick, K.—"Light Stability and Stabilization of Polyurethanes;" 259-263.
Toepke, P. and Kühn, S.—"Treatment of Sewage from Dispersal Colour Manufacturing;" 305-306.

Skandinavisk Tidsskrift för Färg och Lack

Published by Dansk Bladforlag K/S, Holbersgade 20, 1057 Copenhagen, Denmark

Vol. 24 No. 4 April 1978

Eiders, E. - "Paint and Cathodic Protection;" 97-105 (in Danish).

Vol. 24 No. 5 May 1978

Ivermark, R.—"Waterborne Paints—a Sound Thought;" 129-146 (11 pages) (in Swedish).

Meetings / Education

Academic and Industry Scientists Discuss Research Needs at Workshop on Science of Organic Coatings

A "workshop" focusing on the science of organic coatings, held June 20-22 at Kent State University, Kent, Ohio, was attended by approximately 100 of the leading coatings-oriented scientists from both academe and industry. The program was jointly sponsored by the National Science Foundation, the Federation's Paint Research Institute, National Paint and Coatings Association, and the Organic Coatings and Plastics Chemistry Division of the American Chemical Society.

The invited attendees included more than 30 university professors, some 55 representatives of coatings manufacturing and supplier firms, several independent consultants, as well as representatives of various industry associations, governmental organizations, and the technical press.

Heading the steering committee for the organization and planning of the event was Dr. John L. Gardon, R & D Director of M & T Chemicals and a Trustee of the Paint Research Institute. Dr. Raymond R. Myers, University Professor, Kent State University, and Research Director of the Paint Research Institute, served as Principal Scientist at the conference.

Stated objectives of the workshop were:

(1) Define the non-proprietary basic research needs of the coatings industry, particularly as related to the fast-changing technologies satisfying ecological, energy-saving and safety requirements.

(2) Establish an intimate contact between leading academic and industrial scientists for bringing to the attention of the academic community research opportunities related to coatings technologies and to acquaint industrial scientists with the thinking of academicians.

(3) Provide a blueprint (through the Proceedings of the Workshop) for basic research which should influence various governmental and industrial funding agencies supporting academic research and guide research policies of universities and industrial organizations.

Anticipated benefits of the workshop include:

(1) Increase in volume of basic academic research relevant to the new coatings technologies.

(2) More mission-oriented funding of such research.

(3) Improved understanding between the academic and industrial scientific communities.

(4) Definition of gaps in our scientific understanding of modern coating technologies.

Each day began with a series of coatings-related plenary lectures by recognized authorities in the field. While most of these dealt with advances in the science and technology of coatings, some were concerned with the more general aspects of the industry's needs and methods of bringing academe and industry into a closer partnership. Concern for the impact of tightening government regulations underlay many of the presentations. Following the lectures, the attendees were divided into four sections for participation in the actual workshops. Each section had a leader and a recorder. Assigned topics were discussed with the aim of committing to paper a comprehensive tabulation of the problem areas associated with the particular field under discussion. The results of each workshop session were later reviewed for the entire group. At the end of the three-day meeting Dr. Frederick R. Eirich, Distinguished Professor of Polymer Chemistry, Polytechnic Institute of New York, presented an overall summary of the entire program.

Impact of Government Regulations

It was evident throughout the conference that stiffening government regulations regarding air and water pollution, the use of toxic substances, and energy conservation were uppermost in the minds of the conferees. Royal A.

(Continued on page 92)

Rolla Announces Paint Short Course Schedule; Coatings Workshop to be Held in St. Louis

The Fall series of Paint Short Courses sponsored by the University of Missouri — Rolla has been scheduled from September 11 to October 20.

John A. Gordon, Jr., Adjunct Professor of Chemistry at Rolla, will serve as Director, while Lewis P. Larson, Guest Lecturer in Chemistry at Rolla, will act as Co-Director for the series.

(Sept. 11-15) 31st Advanced Chemical Coatings Workshop — Departing from previous years, the workshop will be held at the Executive International Inn, St. Louis, Mo. rather than on the Rolla campus. The workshop will focus on solutions to the problems encountered with water-borne trade sales and industrial coatings with respect to recent solvent restrictive regulations. Discussion sessions with a panel of industry experts will examine topics, such as white pigments, oils and resins, dispersion, color pigments, solvents and thinners, and additives. Cost of the workshop is $200.

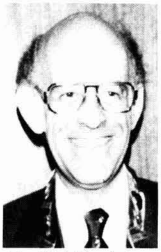
(Sept. 18-22) 37th Introductory Short Course on Composition of Paint — The course will offer basic information on the ingredients used in paint, the machinery and procedures used in the formulation of paint, and the various quality control tests currently in use. Cost of the course is $200.

(Sept. 25-29) 8th Quality Control and Paint Inspector's Short Course — Special emphasis will be placed on product liability and the demands raised by restrictive regulations. Also to be examined are the procedures and equipment used in conducting paint inspections and tests both in-plant and in the field. Cost of the course is $200.

(Oct. 16-20) 28th Short Course for Painting Contractors and Maintenance Engineers — Guest lecturers will discuss paint compostion, cost estimation, surface preparation, application methods, and fire retardant coatings. Cost of the course is $200.

For additional information, or to register, contact Norma Fleming, Education Coordinator, Extension Div., University of Missouri — Rolla, Rolla, Mo. 65401.

Dr. M.F. Kooistra, of The Netherlands, Elected President of FATIPEC, 1978-80



Dr. M.F. Kooistra

Dr. M.F. Kooistra, of AKZO Coatings BV, The Netherlands, was elected 1978-1980 President of the Federation of Associations of Technicians in the Paint, Varnish, Lacquer, and Printing Ink Industries of Continental Europe (FATIPEC) at the XIVth Congress held during the week of June 5, 1978, in Budapest, Hungary.

Dr. Kooistra, immediate Past President of The Netherlands Association (NVVT), succeeds Dr. Lajos Kovacs, of the Hungarian Chemical Society, and will serve through to the XVth FATIPEC Congress to be held during June 1980, in Amsterdam.

Congress

The Congress is held biennially within the country of the organization whose turn is next in the rotation process. The host group prepares and arranges the entire schedule of technical and social activities.

The Congress runs a full five days, beginning with a reception on Sunday evening. On Monday, Tuesday, Wednesday, and Friday, the technical program consists of plenary (unopposed) papers in the morning; concurrent sessions are held in the afternoon. Social activities are planned each evening and a sightseeing trip is usually arranged for Thursday.

Simultaneous translation (into five or six languages) is provided at all paper sessions.

The Congress also includes exhibits, however, they are usually small in number.

The registration fee includes the "FATIPEC Congress Book," a monumental volume of all the papers presented. Publication of each paper is in the language of the author. The rear section of the "Book" includes the administrative and membership directory of FATIPEC.

Congress in Budapest

About 1,000 people (700 men, 300 women) from 23 countries attended the XIVth Congress in Budapest. (Papers presented are given at the conclusion of this article.)

Dr. L. Kovacs

The exhibition, which ran concurrently with the technical program, was sponsored by twenty companies. The list, as published in the program, included: Erichsen, Opton, Tioxide, Eastman Chemical Products, Polyvinyl Chemie, Robbe-Hyfran, Silberline Mfg. Co. Ltd., Synres Chemical, Anic, Cirp, Montedison, Byk Mallinckrodt, Emser Werke, Pevdi, Zeofinn, English China Clays, Chem. Werke Huls, Rhone-Poulenc, Schwegmann, and Veba Chemie.

U.S. Attendance

The Federation of Societies for Coatings Technology (FSCT) was represented by President John J. Oates and Executive Vice-President Frank J. Borrelle.

Other attendees from the U.S. were: Fred Ball, of Eastman Chemical Products, Inc.; W.B. Bartelt, of Rust-Oleum Corp.; Drs. Newell P. Beckwith and B. Burachinsky, of Inmont Corp.; Drs. J.H. Braun, G.I. Mulholland, and D.T. Wu, of the du Pont Co.; Drs. R. Christenson and Percy E. Pierce, of PPG Industries, Inc.; Fred K. Daniel, of Daniel Products Co.; William Georgov, of J.M. Huber Corp.; Dr. Jacob Hortensius, of Synres Chemical Co.; G. Hudson and Dick White, of Mobay Chemical Co.; Lewis P. Larson, of Painteco, Inc.; Drs. Howard L. Gerhart and D.C. Prieve, of Carnegie-Mellon University; Dr. Zeno Wicks, of North Dakota State University; and John Rooney, Consultant. Drs. Wu, Pierce, Hortensius, Gerhart, and Wicks presented papers.

OCCA and SLF

Representing the Oil and Colour Chemists' Association was Angus McLean, President. The Scandinavian Federation of Paint and Varnish Technologists (Skandinaviska Lackteknikers Forbund) was represented by its President, Borje Andersson.

FATIPEC Organization

FATIPEC, founded in 1950, is the European federation of paint/ink industry technical organizations in seven countries: Belgium, France, German Federal Republic, Hungary, Italy, The Netherlands, and Switzerland. Total membership is about 2,500. Each has its own officers who serve two-year terms.

The affairs of FATIPEC are managed by a permanent Secretary-General, Christian Bourgery, whose office is located at 28 Rue St. Dominique, Paris 7, France. The Presidency rotates every two years among the member countries.

A meeting of personnel from four paint industry organizations was held during the Congress. Standing (left to right): F.J. Borrelle (FSCT); Dr. H.L. Gerhart (FSCT); C. Bourgery (FATIPEC): H. Rechmann (FATIPEC); M.F. Kooistra (FATIPEC); D. Pagani (FATIPEC); and J. Roire (FATIPEC). Seated: J. Oates (FSCT); L. Kovacs (FATIPEC); Mrs. Bourgery; B. Andersson (SLF); and A. McLean (OCCA). Not shown: F. Daniel (FSCT), who took picture

Papers Presented at the XIVth FATIPEC Congress

"Electrochemical Properties of Organic Coatings in the Protection of Steel Against Corrosion" — Henri Jullien, Andrew-Jean Berteaud, Francois Henry, and Jean Petit (France).

"Preparation of Stable Absorption Layers and Their Importance for Pigment Dispersion" — Karl Hamann and Richard Laible (West Germany).

"Value of the Electrochemistry in the Study of the Corrosion of Painted Metals" — P. Janssen Bennynck (Belgium).

"Water-Borne Vehicles for Industrial Coatings" — Jacob Hortensius, (U.S.A.).

"Electric and Electrochemical Investigations of Paint Coatings" — Jozsef Devay, Lajos Meszaros, and Ferenc Janaszik (Hungary).

"Air Dry Maintenance Coating Emulsions and New Developments in Air Drying Gloss Paint Emulsions" — Amleto Poluzzi (Italy).

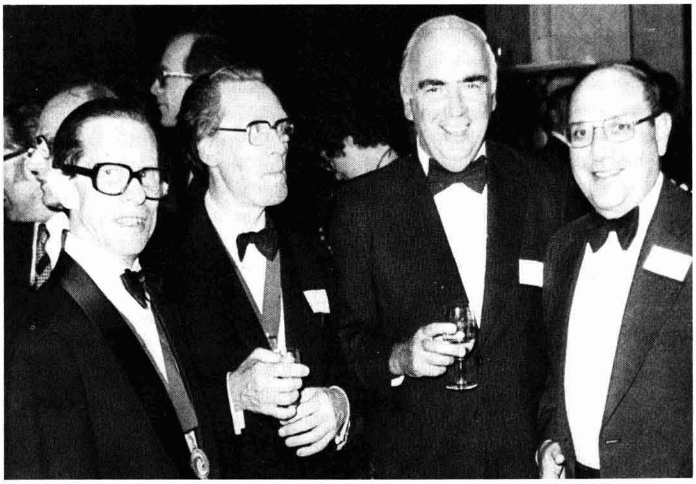
"Weathering Resistance of Coating Films on Metals - Faith and Reality" — Von Erich V. Schmid (Switzerland).

"Novel Thermosetting Epoxide Resin Systems for the Formulation of High Solids Coatings" — Tadeusz Audykowski and Rolf Schmid (Switzerland).

"Possibility of the Prediction of the Viscosity of Polymer Solutions" — Engin Bagda (West Germany).

"Cationically-Non-Ionically Stabilized Emulsions of Polyester Resins Carrying Oxazoline Groups" — Klaus Behmel and Norbert Wamser (Austria).

"Some Practical Problems of Wetting Characteristics in Water-Soluble Paint Systems" — Gyozo Benyi, Laszio Nemet, Lajos Szabo, and Tibor Varadi (Hungary).



Left to right: Borje Andersson, President of the Scandinavian Association (SLF); Angus McLean, President of OCCA; John Oates, President of FSCT; and Frank Borrelle, Executive Vice-President of FSCT

"High Solids Coatings Based on New Epoxy Resin Systems" — Dieter Berger (West Germany).

"Pigmentation of High Solid Paints" — Christian Bertrand and Eliane Gosselin (France).

"Relation Between the Gloss of Paint Films and the Pigmentation in Alkyd Enamels" — Francesco Biglieri and Vincenzo di Paolo (Italy).

"Electrochemical Processes of the Electrodeposition of Resins on Metals" — P.L. Bonora, R. Calvillo, C. Trombetti, and G. Bianchini (Italy).

"Chromatographic Study of Autoxy-Polymerization Process of Oils" — R. Clement (France).

"Theoretical Basis of the Continuous Dispersing Procedure" — Janos Czbik (Hungary).

"Modified Epoxy-Acrylic Systems Curable by UV Radiation" — S. Vargiu, B. Passalenti, S. Bollani, and E. Dall'Orto (Italy).

"Air Drying, Self-Crosslinking Polyacrylates" — Rolf Dhein, Lothar Fleiter and Rolf Kuchenmeister (West Germany).

"Extruder Processed Epoxy Powder Coating Systems: a Method for Determining the Optimum Dispersion of the Powder Components" — Von B. Dreher (Switzerland).

"Colour Match Prediction Regarding the Hiding Power" — Dieter Eitle and Hartmut Pauli (Switzerland).

"Elucidation of Analytical and Structural Problems Related to Coatings and Anticorrosion by the Electron

Exhibition was held during Congress. Shown are Eastman exhibit (above) and Silberline exhibit (right)

Microprobe X-Ray Analyzer Scanning Microscope" — Ortwin Enge (East Germany).

"Methods of Assessing Dispersion Behaviour of Pigments: Experience with DIN 53 238" — Ludwig Gall and Jurgen Spille (West Germany).

"Pigments Orientated to Optic Behavior" — Friedrich Glaser (West Germany).

"Comparative Investigations of Corrosion Resistance of Paint Systems for Vehicles by Different Methods of Accelerated Weathering Including Electrochemical Methods" — Wolfgang Goring, Emma Kosters and Helmut Noack (West Germany).

"Ultra-Violet Curing of Pigmented Finishes" — Peter Hauser, Rolf Osterloh and Manfred Jacobi (West Germany).

"The Characteristic of Parameters of Polycromatic Finish Influenced by the Particle Size Distribution of the Tinting Paste" — Miklos Hauska (Hungary).

"Preparation, Investigation and Application of Modified Silicone Resin Based Varnish Paints" — Jozsef Nagy, Pal Hencsei, Laszlo Ambrus, Laszlo Bihatsi, and Erzsebet Fabulya-Palfi (Hungary).

"Solubility Parameter in Multivariate Analysis" — F. Henry (France).

"Influence of the Paper Surface on the Light Fastness of Prints" — Willy Herbst and Kurt Merkle (West Germany).

"New Electro-Physical Measuring Principles Applied in Development and Use of Synthetic Resins for Electrodeposition" — Helmut Honig (Austria).

"Dispersion of Organic Pigments in Solvent-Free Acrylic Systems" — Volker Kaden and Peter Eckert (Switzerland).

"New Possibilities for the Quantitative Analysis of Photochemical Degradative Reactions on Pigments Paint Films and Plastics" — Gunther Kampf, Hans G. Volz and Aloys Klaeren (West Germany).

"Cathodic Electrocoating and Corrosion Protection" — Fritz Erdmann Kempter, Erich Gulbins and Friedrich Caesar (West Germany).

"On the Determination of Particle Size Distribution of Inorganic Color Pigments with a Spectrophotometric Method" — Fredrich Kindervater (West Germany).

"Pigment-Covering, Pigment-Wetting, Degree of Milling and Paint-Quality" — K.M. Oesterle, W. Wiedemann, N.J. Koll, and K. Klotzsch (Switzerland).

"Study into the Effectivity of Active Pigments in Modern Binders on the Corrosion Protecting Properties" — A. Koopmans (The Netherlands).

"Rheological Methods in the Characterization of Coatings in Relation to Application and Film Formation" — Lars Ole Kornum (Denmark).

"Adhesion of Gloss Emulsion Paints on Coatings Drying by Oxydative Means" — Heribert Kossmann and Gerhard Florus (West Germany).

"The O_2 Permeation Through Paint Films and its Importance for Anti-Corrosive Action" — Peter Kresse (West Germany).

"Polyurethane Coatings on Concrete, Taking Into Account the Physical Properties Relevant to the Building Sector" — Werner Kubitza (West Germany).

"Microgel Separation by GPC of Alkyd Resins: its Formation and Properties in Paints" — H. Hata, J. Kumanotani, Y. Nishizawa, and H. Tomita (Japan).

"Epoxy Coatings and Floorings in Nuclear Power Plants" — Ferenc Laczo and Gustav Klein (Switzerland).

"New Instrumentation for Accelerated Corrosion Tests in a Damp SO_2-Containing Atmosphere" — Marcel Leclercq (Belgium).

Bjarne Martensson, of Zeofinn Oy, Finland (left) and William Georgov, of J.M. Huber Corp.

"Coatings Exposed to Radiation in Nuclear Power Units" — Klaus-Dietrich Ledwoch (West Germany).

"Properties and Performance of Vinyl Acetate/Ethylene/Vinylchloride Terpolymer Emulsion" — Heinz Lehmann and Werner Stelzel (West Germany).

"Investigation into the Effects of Particle Size on the Optical Properties of Paint Films Containing Flash-Calcined Clay" — David L. Light and David G. Jeffs (England).

"Surface Characterization of Pigment Powders" — Robert Rhodes Mather (Scotland).

"Some Factors Affecting the Formulation and Application of Radiation Curable Coatings" — K. O'Hara (United Kingdom).

"Influence of Certain Latex Paint Formulation Variables on Exterior Durability" — Leslie A. Simpson (United Kingdom).

"Application of Mathematical Modeling and Computer Simulation Techniques in the Development of Coatings Formulation" — Tao-Tsing Wu (U.S.A.).

"Advances in the Technology of Solvents in Coatings" — Charles M. Hansen (Denmark).

"Pigment Dispersion — In Principle and Practice" — Geoffrey D. Parfitt (United Kingdom).

"Kinetics and Mechanism of Film Growth During the Electrodeposition Process" — Percy Pierce, Zlata Kovac, and Clark Higginbotham (U.S.A.).

"Some Experiences in Curing Epoxide Resins with Derivatives of Dicyandiamide" — S. Lunak, J. Kalban, J. Vladyka, and J. Smreka (Czech.).

"Investigations About the Stability of Aqueous Alkyd-Melamine Systems" — Jurgen Luthardt (Netherlands).

"Effect of the Reactivity of Thermosetting Systems Based on Alkyd-Melamine Resins to Their Storage Sta-

Mr. and Mrs. Lewis P. Larson (left) and Mr. and Mrs. John Rooney

bilities and the Crosslinking of Their Films" — H. Magdanz, K. Daehre, E. Huttmann, I. Peter, and H. Riede (East Germany).

"Investigations into the Crosslinking of Thermosetting Acrylic Resins in Aqueous Emulsions with Amino Resins" — M. Mateescu, N. Moga, and R. Serban (Romania).

"Determination and Improvement of the Surface Gloss of Pigmented Paint Films" — Bernhard Medinger (Switzerland).

"Comparative Study of Results Obtained with Different Types of Hardness Pendulums" — Radu Metz and Mihaela Vieru (Rumania).

"Process of Film Formation of Aqueous Polyvinyl Ester Dispersions" — Hubert Wiest (West Germany).

"Silica, Constituent of Titanium Dioxide Pigments" — Helmut Weber (West Germany).

"Rheological Investigations of Pigment Dispersions at Low Shear Stresses" — Albrecht Zosel (West Germany).

"Flow Properties of Families of Paints: A Generalized Representation Through a Master-Curve Procedure" — R. Lapasin, A. Papo, and G. Torriano (Italy).

"Electrodeposition Process of Varnishing Products with Particular Regard to the Conduction of an Industrial Plant" — D. Pagani, P. Ruspino, A. Bertoldi, and A. Marzorati, (Italy).

"Variation of the Electrodeposition Parameters in Comparison with the Composition of Certain Acrylic Resins" — Constantin Robu (Romania).

"Chemiphoresis — A Method for Deposition of Polymer Coatings Without Applied Electric Current" — D. Prieve, H. Gerhart, and R. Smith (U.S.A.).

"Some Observations About Precipitation of Chrome Yellow Pigments" — Nicolae Orban and Iuliu Galfi (Romania).

"Influence of Pour Volume and Particle Size of Micronized Silica Gel on the Matting Performance in Clear Coatings" — S. Algieri, W.E. Clark, and A.R. Wackernagel (West Germany).

Secretary-General of FATIPEC, Christian Bourgery and wife

Dr. Percy Pierce, of PPG Industries, Inc. (left) and Fred Ball, of Eastman Chemical Products, Inc.

"Effect of Different Factors on the Pot-Life of Two Component Systems Consisting of Hydroxy Acrylic Resins and Polyisocyanates" — N. Moga and M.-I. Mateescu (Romania).

"Electropolymerized Coatings on Mild Steel" — J.D. Scantlebury, V. Ashworth, and T. Underwood (United Kingdom).

"Accelerated Weathering Tests to Determine the Outdoor Performance of Paints on Galvanized Steel" — Gerhard Neubert (West Germany).

"New Method to Characterize the Durability of Titanium Dioxide Pigmented Coatings" — Rolf Schwindt (West Germany).

"Evaluation of Weather Resistance of Coil Coatings" — D. Schinstad (Norway).

"Cracks Propogation in the Paint Film, Its Measuring and Importance for the Study of Paint Film Structure" — Karel Rehacek (Czech.).

"Epoxy-Acrylic Esters as Film-Formers for UV-Curing" — J. Mleziva, J. Jarusek, V. Cermak, and M. Vecera (Czech.).

"Relationship Between Technical Performance, Crosslinking Mechanism and Surface Finish of Powder Coatings" — Felix Schulde (West Germany).

"Special Problems Occurring in Color Matching for Letterpress and Offset Printing Inks" — Horst Schmelzer (West Germany).

"Realization of the Continuous Production of Synthetic Resins in the Tisza Chemical Works" — S. Palfi and G. Bekefi (Hungary).

"Modern Application Units for Two-Pack PUR Coatings" — Walter Uerdingen and Georg Wawra (West Germany).

"Quantitative Polymer Solubility Maps" — John Ramsbotham (Netherlands),

"Measurement of Progression and End of the Grinding Process in Paint Manufacturing, Dependant of the Kind of Grinding Machine" — K.M. Oesterle (Switzerland).

"Role of the Diffusion in Chromate Pigments Action" — C. Defosse and M. Piens (Belgium).

"Surface Analysis of Mineral Pigments by E.S.C.A." — A. Toussaint (Belgium).

"On the Automation of Pigment Testing" — Friedrich Vial (West Germany).

"Experimental Determination of Coloristic Data of Organic Pigments with the Aid of Simple Colorimetric Apparatus (3-Filters)" — L. Wihan and H. Ehrsam (Switzerland).

"Pigment-Vehicle Interaction in Water-Dispersible Epoxy Formulations" — S. Wilska, P. Talvitie, and P.O. Makinen (Finland).

"Epoxy Resin Characterization by G.L.C." — P. Alessi, I. Kikic, A. Papo, and G. Torriano (Italy).

"Air Drying Epoxy-Phenolic Systems" — S. Vargiu, B. Passalenti, G. Giuliani, and F. Scalisi (Italy).

"Influence on Polyurethane Coatings on the Mechanical Properties of Plastics" — Manfred Schonfelder and Gerhard Mennicken (West Germany).

"Malonic Ester Blocked Isocyanates" — Zeno Wicks and Barry Kostyk (U.S.A.).

"Increasing the Resistance to Changes of Temperature of Coatings of Unsaturated Polyester Varnishes on Wood" — B. Svoboda and M. Mach (Czech.).

"Hydroxylated Vinyl Resins in Wood Coatings Based on Curable Binders" — J.M. Pouchol (France).

"Tendencies of Development of Electroinsulating Lacquers and the Related Economic and Environmental Problems" — Mihai Motoiu (Romania).

"Study of Sound and Thermal Insulation Features of High Thickness Wall Facings" — Maria Pintus and Franco Lucherini (Italy).

(Continued on next page)

Dr. D.T. Wu, of the du Pont Co. (left) and Dr. Zeno Wicks, of North Dakota State University

Organic Coatings Workshop Deals with Industry/Government Topics

(Continued from page 87)

Brown, Vice-President - Technical, National Paint and Coatings Association, summarized the dilemma facing the coatings industry in part as follows: "In recent years the United States Congress has passed many laws designed to clean up the air in industrialized areas and purify our rivers, lakes and water supply. Other laws have been enacted which ban or greatly limit our exposure to substances which are potentially harmful to our health and safety. Still other laws are being considered which will limit the use of certain sources of energy and will force us to make use of different forms of energy."

These regulatory actions by government, Mr. Brown said, present a strong challenge to the industry and he questioned whether it will be possible to carry out all the "defensive" research necessary to stay in business and still do the basic research required to keep the United States in the forefront of world industry.

"My contention," he said, "is that basic research done in industry will suffer. Help is needed from other sources and the academic world is the logical group to whom we must turn for help."

Keynoting the initial session, Dr. George Bugliarello, President of the Polytechnic Institute of New York, decried the fact that government is doing more and more basic research and industry less and less. The coatings industry, he pointed out, is one of the most underrated of all industries, when one considers that 1-3 mils of coating protects millions of dollars worth of equipment and machinery on which this country depends.

Academe/Industry Interplay

Dr. John Gardon stated that if we are to solve the monumental problems facing the paint industry, we must understand the basics of what we are trying to do. The empirical approach will no longer suffice. The workshop principle, involving as it does the cross-fertilization of ideas from both academe and industry, is a step toward the development of the necessary understanding.

Dr. Albert C. Zettlemoyer, Vice-President and Provost, Lehigh University, emphasized that there has not been enough interplay between industry and academe. An adhesive interface must be developed, he said, and recommended the sponsorship of academic fellows by industry as a road to the establishment of such an interface. He pointed out that high technology industries have always been more successful than low technology ones.

Another of the keynote speakers, Neil Estrada, Vice-President of Reichhold Chemicals, Inc. and FSCT Past-President, reinforced the above views and offered some additional implementation steps. We need more institutions, he said, that educate along the lines of industry. We also need more chemistry students in the colleges, and he proposed programs in the high schools that would stimulate students to pursue scientific careers. More funds are needed from industry to support graduate students in coatings-oriented research, he said; but unfortunately our business has a low profile and doesn't provide sufficient promise of challenging jobs to attract a large segment of the best students. He mentioned the need of a good correspondence course in coatings and the current lack of a good textbook.

One of the highlights of the conference was an address by Dr. Richard Heckert, Senior Vice-President of the E. I. duPont de Nemours & Co., Inc., on the subject of industry-government interaction. Government restrictions, Dr. Heckert said, while necessary to a reasonable degree in many instances, have now become so stifling as to constitute a serious threat to our economy unless they can be softened to provide a more sensible balance. Unfortunately, the American people distrust bigness, whether it be in industry, trade unions or the federal government. The one body in which they have a considerable degree of confidence is the scientific community. The universities, therefore, can do the country a great service by helping to convince the people and the government agencies which represent them that industry is not being purely self-serving in its objections to some of the more crippling restrictions. He felt that society would regard scientists as the honest referees needed to clarify the facts and criticize excesses from all sides.

In his overall summary of the workshop program, Dr. Eirich had some additional proposals for encouraging the establishment of a more fruitful interaction between academic and industrial scientists. He suggested that a number of bright young college professors be paid to serve three months each of three years in industrial laboratories and, conversely, some of the best young people in industry serve a like amount of time in universities. This cross-pollination, he noted, was bound to be helpful in increasing the efficiency of coatings-oriented research.

An indication that the workshop program is already bearing fruit became evident even before the conference closed. Five of the university professors in attendance came forward with a proposal that a meeting be arranged for them with a group of scientists from industry to discuss the implementation and funding of an interdisciplinary research program to solve selected urgent problems facing the industry. The Paint Research Institute Board of Trustees was quick to seize this opportunity for a cooperative basic research program, and it is anticipated that details of the proposed conference will be crystallized within the next few months.

XIVth FATIPEC Presentations

(Continued from page 91)

"Investigations on the Temporal Characteristics Concerning Temperature of Painted Bodies, Made from Metal or Wood, in Dependance of Their Color and Reflectance of Heat" — W. Goring, B. Hantschke, P. Lessmeister, and K. Zwernemann (West Germany).

"Experiences with a New Procedure of the Determination of the Performance of Anti-Corrosive Coatings" — H.G. Peters and H. Schutte (East Germany).

"Effect of Surface Preparation of Hot-Dipped Zinc Coated Steel on the Properties of Paint Coating" — S. Szaniewski, H. Rycerska-Lazecka, and A. Potapowicz-Lewandowska (Poland).

"Studies of Polyurethane-Based Shop Primers" — M. Sonntag, B. Riberi, and W. Slawyk (West Germany).

"Organic Coating and Cathodic Protection" — J.H. de Vlieger (Holland).

"Painting on Zinc Metallized Surfaces" — J. Vandermaele (France).

"Talks Available" Booklet Expands to 39 Presentations

The 1978-79 listing of "Talks Available for Constituent Societies" contains a total of 39 presentations — 18 new titles — which are available for the upcoming meeting season.

The list has been compiled and distributed to the Societies by Joseph T. Sullivan, of Rohm and Haas Co., Chairman of the Federation's Public Relations Committee. The booklet includes for each presentation: (1) Title; (2) Name of speaker; (3) Company affiliation, address, and telephone; (4) Geographical areas where talk is available; (5) Equipment needed; (6) Abstract; (7) Biographical sketch of speaker; and (8) if applicable, where and when talk has been previously presented.

The following presentations are listed:

Additives

"Use of Organo Titanates in Coatings" — Kenrich Petrochemicals, Inc.

"Foam in Coatings — Its Prevention and Destruction" — Nalco Chemical Co.

"Screening Procedures of Antifoams, and the Dilemma of Predicting Success in Use" — Nalco Chemical Co.

"Dispersants and Dispersing Resins" — Rohm and Haas Co. (Research Laboratories)

"Selecting an Effective Non-Mercurial Mildewcide for Trade Sales Paints" — Rohm and Haas Co.

"Heavy Metal Additives for Coatings — A Prospectus" — Tenneco Chemicals, Inc.

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

Colors and Pigments

"Polymeric Organic Dispersants for Pigments — Principles and Practices" — Carnegie-Mellon Institute of Research.

"Concepts in Trade Sales Color Systems" — Chemetron Corp., Pigments Div.

"Volumetric Computer Color Matching" — Chemetron Corp., Pigments Div.

"Efficient Formulating Techniques with Clays" — Burgess Pigment Co.

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

"Pipe Your Pigment and Pump Up Your Profits" — Engelhard Minerals & Chemicals Corp.

"Particle Size and Shape of Extender Pigments and Effect on Water-Based Coatings" — Georgia Kaolin Co.

"Fifty Years of Optical Properties in the Paint and Coatings Industry" — Hunter Associates Laboratory, Inc.

"The Microprocessor Has Revolutionized the Art of Color Measurement" — Hunter Associates Laboratory, Inc.

"Toxicity" — H. Kohnstamm & Company, Inc.

"Assessment of Pigment Dispersion by a Colorimetric Technique" — Pfizer, Inc., MPM Div.

"Color Measurement in the Iron Oxide Pigment Industry" — Pfizer, Inc., MPM Div.

The Use and Misuse of Aluminum Pigments in Surface Coatings" — Silberline Manufacturing Co., Inc.

"Environmental Update" — Sun Chemical Corp.

Production

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

"UV Photochemistry in Industry: New Opportunities" — Fusion Systems Corp.

"Manufacture and Application of Spheres" — Ultraspherics, Inc.

Resins

"Conservation Oriented Coatings Systems" — Ashland Chemical Co.

"Formulation Aspects of Industrial Water Reducible Coatings" — Cargill, Inc.

"Why High Solids?" — Cargill, Inc.

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

"Water-Borne Coating for Furniture" — Polyvinyl Chemical Industries.

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

"Aqueous Industrial Coatings" — Rohm and Haas Co.

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

Solvents

"Economic Recovery of Paint Wash (Solvent)" — DCI Corp.

"Viscosity Variation and Solvent Balance in Water Soluble Coatings" — North Dakota State University.

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

"Evaporation of Solvents: Comparison of Evaporation Rates from Filter Paper and from a Smooth Surface" — Shell Development Co.

Testing

"Accelerated Weathering and Fading *or* Natural Outdoor Testing" — Atlas Electric Devices Co.

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

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

PRI Research Efforts Aided By First-Half Contributions

Contributions for the 1978 research projects of the Paint Research Institute were received at Federation headquarters during the first six months of this year. These supplement funds committed by the Federation to PRI.

Contributions received to June 30, 1978 include:

Societies

Baltimore; C-D-I-C; Golden Gate; Montreal; New York; Pacific Northwest; Rocky Mountain; Southern; and Toronto.

Corporations

Bate Chemical Co. Ltd.; Body Bros., Inc.; The M.F. Cachet Co.; Canadian Industries Ltd.; Dar-Tech, Inc.; DeSoto, Inc.; Donald McKay Smith, Inc.; Dow Chemical USA; Dumar Paints & Chemicals Ltd.; Gulf and Western Chemicals Division — Titanium; Inmont Canada Ltd.; Jamestown Paint & Varnish Co.; L.V. Lomas Chemical Co.; Mobil Chemical Canada Ltd.; Benjamin Moore & Co. Ltd.; Nacan Products Ltd.; Reichhold Chemicals Ltd.; Shell Chemical Co.; and Tioxide of Canada Ltd.

Associations

Baltimore Coatings Association; and Canadian Paint & Finish Magazine.

Individuals

Mr. and Mrs. James W. Broggini; Ross Galati; and Mr. and Mrs. Flemming Kress.

Coatings Lab Dedicated At University of Detroit

The University of Detroit, Polymer Institute, recently dedicated its newly constructed coatings laboratory, marking the culmination of a three-year effort which involved the Detroit Society for Coatings Technology, Detroit Paint and Coatings Association, and 22 Detroit-area firms.

Solicitation of funds to finance the project was initiated in 1974 and resulted in donations totaling $40,000 to construct and equip the laboratory.

A coatings education program was initiated at the university in 1969 by the Detroit Society's Education Committee, with the assistance of Dr. Kurt Frisch, Chairman of the Polymer Institute. Subsequently, Dr. Taki J. Anagnostou was recruited as an instructor. Dr. Anagnostou, of Mortell Co., received the first Ph. D. granted by North Dakota State University under the late Dr. A. E. Rheineck, and was appointed Adjunct Professor. He continues to teach in the coatings program.

Classes are held week nights and Saturdays, and are designed to enable persons employed full-time and graduate students to obtain advanced degrees. There are currently 23 coatings chemists pursuing masters' degrees.

Representatives from the donating companies were present at the dedication ceremony and were given a brief history of the coatings program.

Dr. Harry Szmant (left), Associate Dean of Science at the University of Detroit, accepts check from Thomas White, President of the Detroit Society as part of the Society's ongoing donation to help with expenses for new coatings laboratory at University of Detroit's Polymer Institute.

NACE Schedules Education Courses For Dallas and Houston

Two continuing corrosion education programs have been planned for presentation during September 1978 by the National Association of Corrosion Engineers (NACE). The education programs will be held at the Dallas Hilton Hotel, Dallas, Tex., September 10-15, and at the Astrovillage Motel, Houston, Tex., September 24-28.

Each session will feature the three NACE Continuing Corrosion Education courses: the NACE Basic Corrosion Course, Corrosion Prevention by Cathodic Protection, and Corrosion Prevention by Coatings.

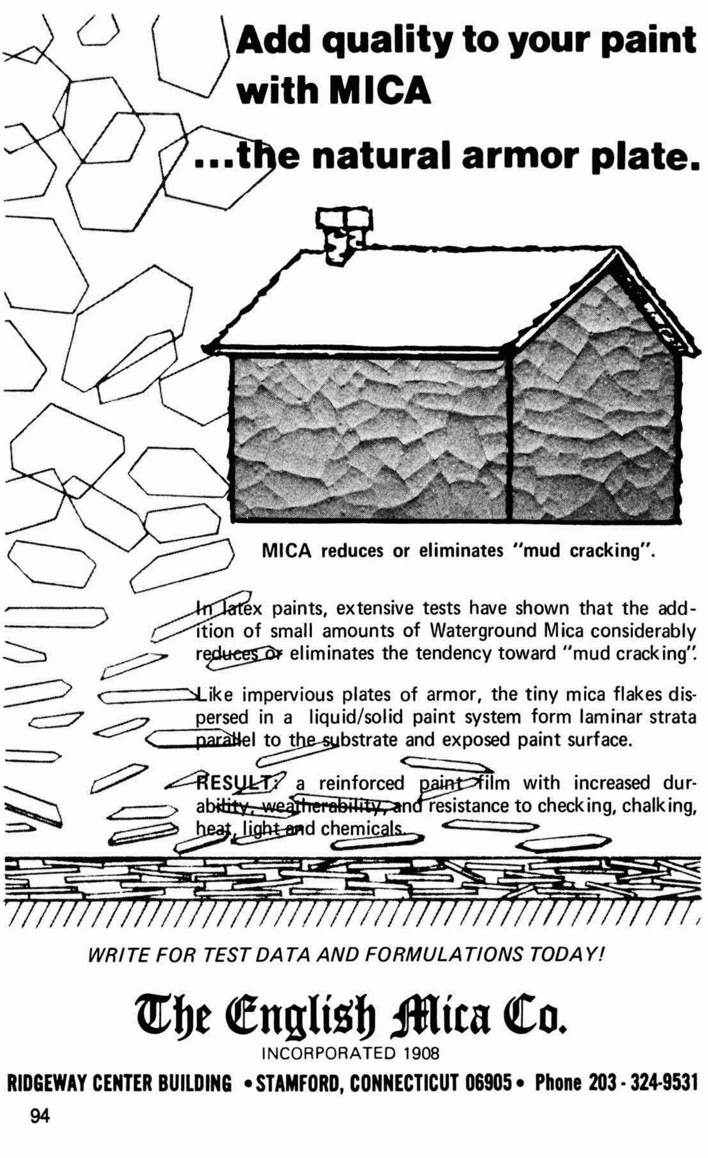
The NACE Basic Corrosion course, attended by more than 3,000 students, is written for technicians and engineers who have little previous training in corrosion and for those persons desiring to broaden their general knowledge.

The Corrosion Prevention by Cathodic Protection course was developed toward educating engineers and technicians in the principles of cathodic protection.

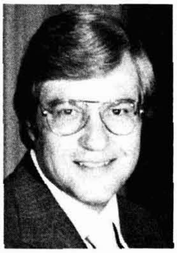
The Corrosion Prevention by Coatings course provides instruction and training for engineers who require practical knowledge of the use of protective coatings in corrosive environments.

Each of the three NACE courses is presented in a 34-hour (4½ days) seminar with a final examination at the conclusion. The course registration fee of $350 ($300 to NACE members) includes text books and materials, lunch, and refreshment breaks.

For further information write National Association of Corrosion Engineers, Education Dept., P.O. Box 986, Kay, Tex. 77450.



People



Robert A. Roland, who was associated with the National Paint and Coatings Association for 20 years, joined the Manufacturing Chemists Association, Washington, D.C., on August 1 as President. He succeeded **William J. Driver** who retired from MCA after serving as President since 1969.

As President of MCA, Mr. Roland will become the chief spokesman for the 202 U.S. and Canadian member companies of MCA. These firms represent more than 90% of the production capacity of basic industrial chemicals within these countries.

Mr. Roland joined NPCA in 1958 as Director of Chemical Coatings. He then served two years as Assistant to the President and 14 years as Executive Vice-President. In 1975, his title was changed to President.

A native of Philadelphia, Mr. Roland is a Board member of the Chamber of Commerce of the United States and Chairman of the Chamber's Association Committee. He also is a Director of Citizens Choice, Vice-President and a Director of the American Society of Association Executives (ASAE), Secretary and a Director of the National Association Executives Club, Chairman of the Industry Sector Advisory Committee #7 serving the U.S. Department of Commerce, and an Advisory Director of Union First National Bank.

A 1952 graduate of Villanova University with a Bachelor's Degree in Economics, he earned his Law Degree from the University of Pennsylvania Law School in 1955. Mr. Roland joined NPCA in 1958, after a period of active duty with the U.S. Navy. He is a member of the Pennsylvania and U.S. Supreme Court bars.

James A. Leider, Jr. has retired as Chief Chemist for the Preservative Paint Co., Seattle, Wash., following 38 years service in many different positions. Mr. Leider, a charter member of the Pacific Northwest Society for Coatings Technology, served as its President (1963), and was the recipient of the Society's Outstanding Service Award in 1976. For over 24 years he has served as the Society's Administrative Secretary.

S.B. Levinson

W.A. Wentworth

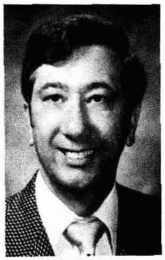
Sidney B. Levinson, President of D/L Laboratories, New York, has been elected Chairman of Committee D-1 of the American Society for Testing and Materials. He has been active in ASTM for over 30 years and has been Chairman of D-1's Subcommittee on Architectural Finishes for 10 years. Mr. Levinson is a Past-President of the New York Society for Coatings Technology and received the Society's PaVaC Award in 1960 as well as presenting the PaVaC lecture in 1977.

ASTM national officers were elected during the group's 81st annual meeting in Boston. Named were: President—**Joseph G. O'Grady,** of Public Service Electric and Gas Co., Maplewood, N.J.; Vice-President—**Sydney D. Andrews,** of Florida Dept. of Standards and Consumer Services, Tallahassee, Fla.; remaining as a Vice-President is **Wayne P. Ellis,** of H.B. Fuller Co., Philadelphia, Pa.

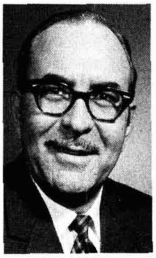
Dr. William A. Wentworth has joined the Jones-Blair Co., Dallas, Tex., as Chief Chemist of the resin laboratory. He will be responsible for resin R&D. Dr. Wentworth was previously associated with Napko Corp., Houston, and was the Secretary of the Houston Society for Coatings Technology.

John J. Galligan, Jr. has been named Sales Representative in the Cleveland area for the RBH dispersions lines of Inmont Corp.'s Color Systems Group. He will assume the accounts previously managed by **Robert J. Ebert,** who is retiring after 39 years with the company.

Cook Paint and Varnish Co., Kansas City, Mo., has appointed **Dean Kincheloe** Director of the Purchasing Div. He succeeds **Henry Jurvis,** who will retire from the company following 46 years of service.



C.C. Colelli



F.J. Hahn

Dover Chemical Corp., Dover, Ohio, has named **Camillo C. Colelli** Vice-President of Research and Development. He was previously Manager of Technical Service.

Frank J. Hahn has retired following a 40-year career in the coatings industry, 32 of which were spent with Monsanto at Springfield, Mass. where he was a Science Fellow in research. A member of the New England Society, he has also served as a member of the PRI Grants Committee. Dr. Hahn will continue to serve the industry as a Consultant. He will reside in Wilbraham, Mass.

The Sherwin-Williams Co. has merged its Specialty Products and Chemicals Groups to form the General Products Group. **Geoffrey L. Tickner** has been elected to head the new group. He was formerly Vice-President of the Specialty Products Group.

Hercules Incorporated, in a series of managerial appointments in its Advertising and Public Relations Dept., has named **Charles Trayford III** Advertising Div. Manager; **Anthony J. Kobus** was appointed Product Promotion Div. Manager; and **Robert W. Goodman** was named Control Manager.

George D. Kennedy was elected President of IMC Corp., Libertyville, Ill., by the company's board of directors. **R.A. Lenon,** formerly President of the company, will continue as Chairman and Chief Executive Officer.

The Trade Sales Div. of The O'Brien Corp. has named **Lew E. Davis** Hawaii Area Sales Manager. **Ferrall R. Johnson** has been appointed to succeed Mr. Davis as Intermountain Area Sales Manager and will be head-quartered in Salt Lake City.

People (Continued)

Kerr-McGee Corp. has named **William J.F. Francis** Executive Vice-President for Marketing, a new position. He is presently Executive Vice-President of Chemical and Refining Operations. Mr. Francis will participate with susidiary and division heads in planning marketing strategy for all areas of the company's worldwide marketing efforts.

Union Carbide Corp. has promoted **Jerry J. Hufnagle** to Area Sales Manager in the Cellular and Elastomers Dept. He will be located in Hackensack, N.J.

L.J. Coughlin has been named Sales Manager for Freeport Kaolin Co. He will be responsible for all domestic sales activities.

Dr. Stanley Rudoff has been named General Manager of Crompton & Knowles Orlex Chemicals Corp., Fair Lawn, N.J.

Buckman Laboratories International, Inc., Memphis, Tenn., has appointed **William R. Dalrymple** International Marketing Coordinator. He joins Buckman after 20 years experience in marketing with Hercules Incorporated.

Jack Meyerhardt has been promoted to Vice-President and General Manager of the Foam Div. of Tenneco Chemicals, Inc.

Air Products & Chemicals, Inc., Allentown, Pa., has named **Donald J. Orr** Director of Materials Management and **Harry A. Quigley** Director of Development Appraisal in the Chemicals, Plastics, and Polymers Group. Mr. Orr will coordinate the Group's distribution, purchasing, and customer service activities, while Mr. Quigley will monitor the Group's commercial development activities and coordinate market research planning, economic evaluation, and operations analysis.

Meanwhile, the company's Chemical Additives and Acetylenics Divisions have merged to form the Performance Chemicals Div. **Robert Blumquist** is General Manager and **Hugh Gallagher** is Assistant General Manager and Director of Marketing.

John Lynch as been named Operations Manager for the Sun Chemical Corp., Pigments Div. manufacturing facility in Muskegon, Mich. The operation, now under construction, is expected to be on stream by early 1979.

The Union Chemicals Div., of Union Oil Co. of Calif., has promoted **William F. Murphy** to Manager, Planning and Sales Service for its Petrochemical Group. He will be located at the division's headquarters in Schaumburg, Ill.

William M. Kraus has been named Director, Sales and Marketing for the Coatings and Inks Div. of M&T Chemicals, Inc. He was formerly Business Manager in the company's Chemical Div.

Napko Corp. has appointed **Philip J. Barden** Central District Trade Sales Manager. He will be responsible for the marketing programs in northern and central California and Nevada.

Piedmont Paint Mfg. Co., Greenville, S.C., has announced the retirement of **Parker W. Layman,** Executive Vice-President, after 30 years of service with the firm. The retirement is effective August 1, 1978.

Mr. Layman, a veteran of 44 years in the coatings industry, was with Southern Varnish Corp. (now Reliance Universal) and Dixie Paint & Varnish Co. (now Dixie-O'Brien) prior to joining Piedmont in 1948.

He has been active in both the Southern Society and the Federation for more than 30 years. He was President of the Southern Society in 1956 and Council Representative from 1963-65. He served on the Federation Board of Directors in 1970.

Mr. Layman and his wife, Audrey, will move to St. Simons Island, Ga.

Troy Chemical Corp., Newark, N.J., has appointed **Ted Jefferys,** of Cascade Chemical Commodities Ltd., as their sales agent for the western provinces of Canada.

Carl Fuller has been named Technical Service Manager of Reichard-Coulston, Inc., He replaces **Morton Treade,** who recently retired. Mr. Fuller is the 1978-79 President of the Philadelphia Society for Coatings Technology, and is Chairman of the ASTM D-1 Subcommittee on Pigments.

Daniel Products Co., Jersey City, N.J., has been acquired by Synres Chemical Corp. U.S.A. Daniel Products will operate as an autonomous affiliate of Synres with **Fred Daniel** continuing as President and Director of the company.

Obituary

Walter G. Ballinger, of Glidden Coatings & Resins Div. and President of the Golden Gate Society for Coatings Technology, died June 9. He was 49 years old. Born in Scotland, Mr. Ballinger became involved in the coatings industry in London during 1950 and moved to South Africa in 1954. Five years later he came to the United States where he joined Specialty Resins, in Lynwood, Calif. A member of the Golden Gate Society since 1969, he was a Senior Chemist at Glidden.

Committee Activities

ENVIRONMENTAL CONTROL

NPCA Air Quality Task Force Calls Attention to Proposed State Regs

Because the Federal EPA has declared most State Implementation Plans (SIP's) to be inadequate, many states are now in the process of rewriting their air pollution control regulations. To aid the states in this matter, the EPA published a Conceptual Regulation in March 1977, but many states either do not know about it, do not understand it, or think that they know better. Since NPCA believes the Conceptual Regulation to be a reasonable one, representatives of the Association have been visiting various states to explain the Conceptual Regulation to them. Conferences have been held with state and regional administrative personnel in major industrial areas, where the regulations in question affect users of industrial coatings. EPA Regions II, V, and VI and the states of Texas, Louisiana, Illinois, New York, New Jersey, and Massachusetts are of particular concern at this time. Manufacturers of industrial coatings in those areas should be alert to impending changes in the applicable SIP's which could affect them.

NPCA representatives also met recently with California Air Resources Board (CARB) administrative personnel, regarding regulations being considered for can/coil coatings, large appliances, metal furniture, and marine coatings. Public hearings regarding metal furniture and marine coatings are being scheduled, and coatings suppliers to those industries in California should be alerted in order to respond to proposed regulations that may adversely impact on their products.

GABRIEL MALKIN
Delegate to NPCA
and Governmental Agencies
(Environmental Control)

METRIC SYSTEMS

Conversion Beginning to Make Steady Progress in U.S.

Partial returns are in on a survey of several Constituent Societies currently being taken on the state of metric awareness and preparedness. When completed, results will be published in JCT. However, for now it is worth noting that many of those who have responded are very unaware of metric conversion activities outside the U.S.

In the last 10 years there has been enormous progress in the conversion to metrics throughout the world. Great Britain, South Africa, and nearly all the member nations of the British Commonwealth—notably Canada, India, Australia, and New Zealand have either completed their conversion or have progressed well along the road to become predominantly or completely metric. In most cases, conversion has been smooth and with fewer difficulties than expected. The U.S. is currently the only industrial nation of consequence which has not yet officially begun converting to metric.

The American public, unfortunately, has been poorly informed on this important subject—and sometimes downright misinformed! But this is changing. President Carter's appointees to the U.S. Metric Board (USMB) were confirmed by the Senate in March. The Board, which held its first meeting early in April, has stated that it will rely heavily on the work done by the American National Metric Council. ANMC was formed in 1973 and since then has done a great deal of studying, preparing and drawing up conversion activities and schedules for the various industrial sectors. This is much like the work of the Canadian Metric Commission's Sector.

It should be recalled that ANMC's committees receive input from industry, and thus represent industry's viewpoint. Because of this early spade work, progress can be expected to be rapid.

It must be emphasized that metric conversion is not enforcement of a blind decree. On the contrary, it is being brought about by the full and voluntary cooperation of industry and other important segments of the community with government for the common good in a changing and expanding world.

E.L. HUMBURGER, *Chairman*

Literature

Dispersing Resin

An aqueous dispersing resin designed for use in water-borne coatings is discussed in literature now available. Characteristics such as low foaming, gloss and viscosity stability, and hardness are covered. For more information about Acrysol I-98, write Marketing Services, Rohm and Haas Co., Independence Mall West, Phila., Pa. 19105.

Painting Systems Catalog

The 1978 edition of "Painting Systems for Specifiers and Applicators," a 60-page catalog, is now available. The literature offers selection charts enabling quick determination of proper coatings products for all types of interior and exterior surfaces under normal exposures; a selection guide for special purpose, heavy duty coatings; and directions for writing coatings specifications. Product descriptions include recommended uses, performance and application information, drying times, coating analysis, film characteristics, surface preparation and priming instruction, and surface burning characteristic data on flame spread, fuel contributed, and smoke developed, as determined by standard testing methods. Painting contractors and architectural and industrial specifiers can obtain the catalog by writing to General Manager, Professional Coatings Div., The Sherwin-Williams Co., 101 Prospect Ave., N.W., Cleveland, Ohio 44115.

ASTM Standards

The "Annual ASTM Standards 1978," Part 27 — Paint, Tests for Formulated Products and Applied Coatings contains 208 standards of which 10% are new, revised, or changed in status since the 1977 edition. Some standards covered in Part 27 are paint, varnish, and laquer as liquids; dry film properties; and schedules of examination for testing paint products and general test methods. The 1012-page book, Publication Code No. 01-027078-14, is listed at $30.

Part 28 — Paint, Pigments, Resins, and Polymers contains 138 standards including pigment specification; pigment test methods — chemical, general, physical, color, and opacity tests; resins and polymers. The 574-page book, Publication Code No. 01-028078-14, is listed at $19.

To obtain copies of Parts 27 and 28, write American Society for Testing and Materials, Attn: Sales Service Dept., 1916 Race St., Phila., Pa. 19103.

Flame Retardant

Three bulletins now available describe a crystalline powder with high bromine content and minimum additive level. The bulletins detail the thermal and toxicological properties of Firemaster 680, as well as its specific properties when used in ABS resins and in high impact polystyrene. For copies of the bulletins, write Chemicals and Resins Business Group, Velsicol Chemical Corp., 341 E. Ohio St., Chicago, Ill. 60611.

Spectrophotometer Bulletin

The first section of the 37-page booklet, "Maintaining Optimum Spectrophotometer Performance," is a digest of parameters and their significance, including definitions of terminology employed in spectroscopy.

The second section includes procedures for monitoring performance, and concludes by identifying sources of instrument performance degradation and a list of recommended practices for preventive maintenance. Bulletin No. ADS-109 can be obtained from the Perkin-Elmer Corp., Instrument Div., Main Ave., Mail Station 12, Norwalk, Conn. 06856.

Book Review

EPOXY RESINS AND PRODUCTS
Recent Advances

M. William Ranney
Noyes Data Corp.
Park Ridge, N.J.
1977
$39.00

Reviewed by
Charles M. Vetters
Dow Chemical U.S.A.
Freeport, Texas

The introduction of this text provides a very succinct historical review and market description of epoxy resins in the United States. From this brief introduction, the book then proceeds to describe the most currently issued patents in various market areas for epoxy resins. The text contains a very well organized technological review of these various market areas for epoxy resins. The review is both complete and easy to read through the use of descriptive chemistry, formulations, and specific end use examples. A very thorough job has been done in reducing the reviewed patents to a concise, readable form.

In addition, the index contains an excellent patent technology reference. It is organized by company, inventor, and patent number for convenient cross-reference.

This is an excellent reference book for any R&D laboratory interested in the current state of the art in epoxy resins. It should serve as a valuable starting point for any new investigations.

Cleveland Society Invites Papers For 22nd Symposium, March 20-21, 1979

"Advances in Coatings Technology" will be the theme of the 22nd Symposium of the Cleveland Society for Coatings Technology to be held at Baldwin-Wallace College, Berea, Ohio, March 20-21, 1979.

Under the sponsorship of the Society's Educational Committee, the Symposium will deal with the broad interdisciplinary character of coatings technology. The Symposium Committee welcomes papers on any aspect of coatings science and engineering.

Preliminary abstracts should be submitted by December 15, 1978 to George R. Pilcher, The Sherwin-Williams Co., Cleveland Technical Center, P.O. Box 6027, Cleveland, Ohio 44101.

Coming Events

FEDERATION MEETINGS

(Nov. 1-3)—56th Annual Meeting and 43rd Paint Industries' Show. Conrad Hilton Hotel, Chicago, Ill. (FSCT, Suite 830, 1315 Walnut St., Philadelphia, Pa. 19107).

SPECIAL SOCIETY MEETINGS

(Sept. 20-21)—Montreal and Toronto Societies Symposium on "Coatings Potpourri." In Montreal on the 20th at Bill Wong's Restaurant. In Toronto on the 21st at Seaway Beverly Hills Motor Hotel. (R. Rauch, Tioxide of Canada Ltd., P.O. Box 580, Sorel, Quebec, Canada J3P 5P8).

1979

(Feb. 12-14)—Sixth Annual Water-Borne and Higher-Solids Coatings Symposium. Hyatt Regency Hotel, New Orleans, La. Sponsored by Southern Society and University of Southern Mississippi. (Dr. B. George Bufkin, Dept. of Polymer Science, University of Southern Mississippi, Southern Station, Box 276, Hattiesburg, Miss. 39401).

(Feb. 28-Mar. 2)—Western Coatings Societies' Symposium and Show. Fairmont Hotel, San Francisco, Calif. (Ed Kevin, The O'Brien Corp., 450 E. Grand Ave., South San Francisco, Calif. 94080).

(Mar. 20-21)—22nd Symposium of the Cleveland Society, "Advances in Coatings Technology." Baldwin-Wallace College, Berea, Ohio. (Sid Lauren, Coatings Research Group, Inc. 2340 Hamilton Ave., Cleveland, Ohio 44114).

OTHER ORGANIZATIONS

(Sept. 10-15)—176th American Chemical Society National Meeting. Miami Beach, Fla. (A.T. Winstead, ACS, 1155 16th St., N.W., Washington, D.C. 20036).

(Sept. 11-Oct. 20) — Paint Short Courses at University of Missouri — Rolla. Advanced Chemical Coatings Workshop — Sept. 11-15, Executive International Inn, St. Louis, Mo.; Composition of Paints and Coatings — Sept. 18-22; Quality Control and Paint Inspectors — Sept. 25-27; and Painting Contractors and Maintenance Engineers — Oct. 16-20. (Norma Fleming, Extension Div., University of Missouri — Rolla, Rolla, Mo. 65401).

(Sept. 12-14) — Liquid Coatings Conference of Association for Finishing Processes of SME. Neil House Hotel, Columbus, Ohio. (J. E. Cherryholmes, AFP/SME, One SME Dr., P.O. Box 930, Dearborn, Mich. 48128).

(Sept. 12-15)—Scientific Colloquium on Corrosion in Water. Pecs, Hungary. (Scientific Society of Mechanical Engineers, 1372 Budapest, P.O. Box 451, Hungary).

(Sept. 14)—NPCA Regional Seminar on "Fire Protection." New York, N.Y. (Georgene Savickas, Director, Meetings and Conventions, National Paint and Coatings Association, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(Sept. 19)—NPCA Regional Seminar on "Energy Analysis and Conservation." Chicago, Ill. (Georgene Savickas, Director, Meetings and Conventions, National Paint and Coatings Association, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(Sept. 26-28) — Fourth International Conference on Radiation Curing, sponsored by Association for Finishing Processes of SME. Holiday Inn O'Hare/Kennedy, Chicago, Ill. (J.E. Cherryholmes, AFP/SME, One SME Dr., P.O. Box 930, Dearborn, Mich. 48128).

(Sept. 27-29)—National Coil Coaters Association Fall Technical Meeting. Hyatt Regency Hotel, Washington, D.C. (Don White, NCCA, 1900 Arch St., Phila., Pa. 19103).

Coming Events (Continued)

(Oct. 3-4) — "Update in Pretreatments for Improved Finishing Systems Quality," sponsored by Association for Finishing Processes of SME. Holiday Inn O'Hare/Kennedy, Chicago, Ill. (J.E. Cherryholmes, AFP/SME, One SME Dr., P.O. Box 930, Dearborn, Mich. 48128).

(Oct. 4-5)—Society of Plastics Engineers RETEC, "Coloring of Plastics XII—Automotive Color." Saw Mill Creek Inn, Huron, Ohio. (Louis J. McDonald, W.R. Grace & Co., 880 Deepwoods Dr., Medina, Ohio 44256).

(Oct. 10-11)—National Association of Corrosion Engineers North Central Meeting on "Coatings and the Law." Marriott Motor Inn, Cleveland, Ohio. (NACE, P.O. Box 986, Katy, Tex. 77450).

(Oct. 10-12)—Fifth Annual UMR-DNR Conference and Exposition on Energy. Conducted by the University of Missouri - Rolla and the Missouri Dept. of Natural Resources. (Dr. J. Derald Morgan, Conf. Dir., 108 Electrical Engineering Dept., University of Missouri - Rolla, Rolla, Missouri 65401).

(Oct. 15-19)—Architectural Aluminum Manufacturers Association 42nd Annual Convention. The Contemporary, Walt Disney World, Fla. (Barbara Lewke, Meetings Coordinator, AAMA, 35 E. Wacker Dr., Chicago, Ill. 60601).

(Oct. 28-Nov. 3)—Fifth Annual Meeting of Federation of Analytical Chemistry and Spectroscopy Societies. Hynes Memorial Auditorium, Boston, Mass. (P. Lublin, GTE Labs, 40 Sylvan Rd., Waltham, Mass. 02154).

(Oct. 30-Nov. 1)—National Paint and Coatings Association Annual Meeting. Palmer House, Chicago, Ill. (Georgene Savickas, NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(Nov. 8-9)—Powder Coating Conference. Convention Center, Cincinnati, Ohio. (Association for Finishing Processes of SME, 20501 Ford Rd., Dearborn, Mich. 48128).

(Nov. 13-15)—Society of Plastics Engineers National Technical Conference, "Plastics in Packaging." Hyatt Regency O'Hare, Chicago. (Eugene E. Wilson, SPE, 656 W. Putnam Ave., Greenwich, Conn. 06830).

(Nov. 17-19)—Annual National Decorating Products Association Convention and Show. Alfonso J. Cervantes Convention and Exhibition Center, St. Louis, Mo. (National Decorating Products Association, 9334 Dielman Industrial Drive, St. Louis, Mo. 63132).

1979

(Feb. 11-14)—"Judd Memorial Conference on Color Metrics," sponsored by Inter-Society Color Council. Williamsburg Lodge, Williamsburg, Va. (S. Leonard Davidson, c/o N L Industries, P.O. Box 700, Hightstown, N.J. 08520).

(Mar. 5-9)—30th Pittsburgh Conference on "Analytical Chemistry and Applied Spectroscopy." (Pittsburgh Section, Analytical Group, et al., Convention Center, Cleveland, Ohio).

(Mar. 12-16)—National Association of Corrosion Engineers Annual Conference and Materials Performance and Corrosion Show, CORROSION/79. Atlanta Hilton, Atlanta, Ga. (NACE, P.O. Box 986, Katy, Tex. 77450).

(Mar. 22-23)—Coatings - 79: International Symposium on Coatings. Carillon Hotel, Miami Beach, Fla. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont., K6H 5V7, Canada).

(Mar. 29-30)—International Conference on Spectroscopy. Konover Hotel, Miami Beach, Fla. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont. K6H 5V7, Canada).

(Apr. 1-6)—Pacific Chemical Conference: 1979. Honolulu, Hawaii. (A.T. Winstead, ACS, 1155 - 16th St., N.W., Washington, D.C. 20036).

(Apr. 3-6)—OCCA-31. Oil and Colour Chemists' Association 31st Annual Technical Exhibition. Alexandra Palace, London, England. (The Director & Secretary, Oil and Colour Chemists' Association, Priory House, 967 Harrow Rd., Wembley, Middlesex, HAO 2SF, England).

(Apr. 19-20)—Second Canadian Chromatography Conference. Hampton Court Hotel, Toronto, Canada. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont. K6H 5V7, Canada).

(Apr. 25-May 3)—70th Annual Meeting of the American Oil Chemists' Society. Fairmont Hotel, San Francisco, Calif.

(May 18-19)—International Symposium on "Flammability and Fire Retardants." Maria Isabel Sheraton Hotel, Mexico City, Mexico. (V.M. Bhatnagar, Alena Enterprises of Canada, P.O. Box 1779, Cornwall, Ont. K6H 5V7, Canada).

(June 20-23)—Oil and Colour Chemists' Association Conference, "The Challenge to Coatings in a Changing World." Stratford Hilton Hotel, Stratford-on-Avon, England. (The Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HAO 2SF, England).

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