

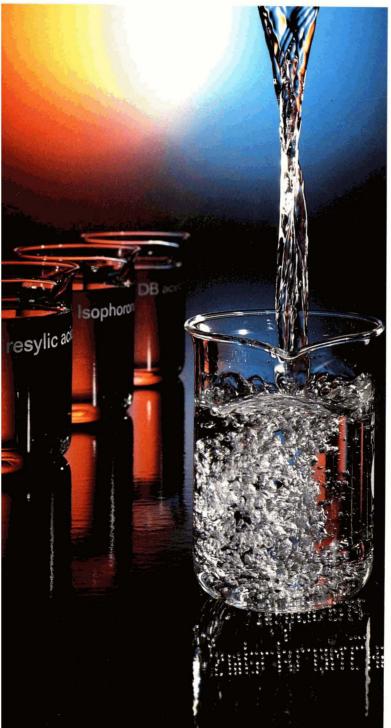
Effect of Molecular Weight On Performance of Cellulosic Thickeners in Latex Paints



JCTAX 55 (701) 1-100 (1983)

JUNE 1983

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JUNE 1983

JOURNAL OF

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NUMBER 701



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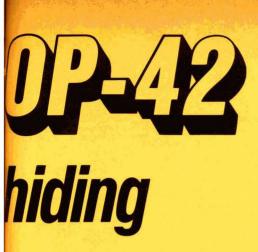
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Comment

Accentuating the PR in PRI

A major effort to upgrade communications is currently being pursued by the Paint Research Institute Board of Trustees, to assure that the coatings industry generally and the Federation membership specifically are kept abreast of PRI activity.

Upcoming shortly in the JCT will be a summary and analysis of the results from the recent industry survey on suggested areas for PRI research, as well as a progress report on work being done by the PRI-sponsored Mildew Consortium.

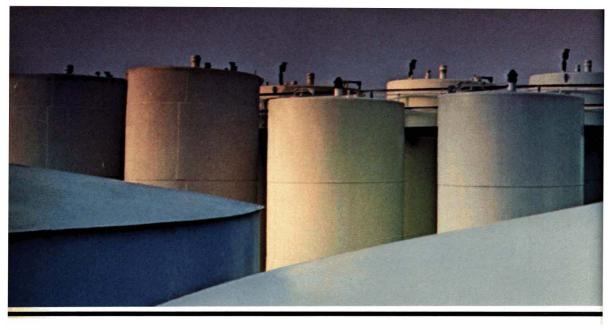
And Technical Committees of a dozen Societies are about ready to begin a cooperative testing program in conjunction with the Mildew Consortium. They will be provided with quantities of a polymer containing an anchored fungicide, to formulate into paint systems for exposure testing. The project will help determine if the anchored-fungicide concept will lead to a paint system that "fights back" against mildew. It also provides an opportunity for Society members to participate in PRI research work.

Meanwhile, liaison with the Constituent Societies is being pursued in other areas. The PRI Trustees have set up a matching funds program to assist Societies wishing to establish joint research projects with their local colleges. In addition, researchers doing work funded by PRI Fellowship grants will report on their activities to Societies in their local area, when and as requested.

PRI's work is important, and so is industry and member awareness of that work. The current emphasis on promoting that awareness should benefit all concerned.

Thomas a. Kois

T. A. Kocis, Contributing Editor



CIBA-GEIGY epoxy resins vs. the toughest applications around

Aggressive chemicals and organic solvents are tough on storage tank linings. Formulating a coating that resists them is not an easy job if you don't have the right components. Now, three new products from CIBA-GEIGY can help you change that.

Coatings based on XU-252.

Our new high performance novolac epoxy resin can take on many aggressive chemicals including chlorinated solvents, ethanol, methanol, aromatic amines, acids, caustic and ammonia—under a variety of service temperatures.



XU-252 based coating

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As you can see in the panels, an XU-252 based coating showed no sign of failure even against a solvent like methylene chloride, while one formulated with a standard bisphenol-A based epoxy resin softened and blistered.





Bisphenol-A based coating.

We also tested XU-252 based coatings for resistance against a range of other chemicals. The chart shows some of the toughest ones. In all cases these coatings exhibited excellent resistance to continuous exposure for a minimum of eight months.

Two new hardeners increase chemical resistance.

HY943 and HY2969 when formulated with XU-252 provide a variety of new properties.

HY943 produces solvent-free and high solid coatings with excellent alcohol and chlorinated solvent resistance.

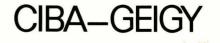
HY2969 produces solvent-free systems resistant to acids and aromatic solvents even as tough as benzene.

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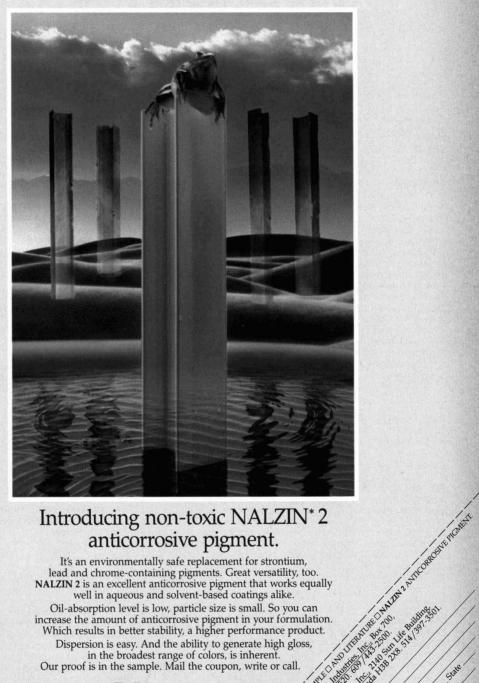
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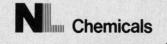


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EFFECT OF MOLECULAR WEIGHT ON PERFORMANCE OF CELLULOSIC THICKENERS IN LATEX PAINTS— D.M. Blake

Abstracts of Papers in This Issue

Journal of Coatings Technology, 55, No. 701, 33 (June 1983)

A latex paint was formulated in five versions, each thickened with hydroxypropyl methylcellulose of a different molecular weight. The five were brought to essentially equal viscosity for comparison of thickener efficiencies. To determine and compare other effects of thickener molecular weight variation, the five were evaluated for: rheology at low-shear and high-shear rates; flow and leveling characteristics; spatter resistance; foaming tendency; 'scrub resistance; and color acceptance. Thickener molecular weight was found to affect the paint's high-shear rheology, spatter resistance, and foaming tendency; it has no effect on low-shear rheology, scrub resistance, or color acceptance. The color acceptance testing indicated shortcomings in the commonly used hand rubup test procedure.

RHEOLOGICAL PROPERTIES OF ALKYD SOLUTIONS AND THEIR RELATION WITH SOLUBILITY PARA-METERS OF SOLVENTS—K.M.A. Shareef and M. Yaseen

Journal of Coatings Technology, 55, No. 701, 43 (June 1983)

The rheological properties of alkyd solutions in poorly and moderately hydrogen bonded solvents have been studied by viscometry. The data at six concentrations and at eight shear rates have been analyzed with the help of standard and proposed equations. The concentration dependence and solubility parameter dependence of several rheological parameters were obtained. This information was used for determining the solubility parameter of the alkyd.

SURFACE COLORING OF GLASSY POLYMERS BY SOLVENT DIFFUSION-G. Guerra, C. Paolone, and L. Nicolais

Journal of Coatings Technology, 55, No. 701, 53 (June 1983)

The technique of surface coloring of polymeric glassy materials based on the swelling properties of the organic solvents is suitable for solving particular problems in decoration of plastic objects.

The depth of dye penetration and the concentration in the surface layer, and hence the coloring effect, are functions of diffusion conditions. Therefore, a knowledge of the solvent diffusion process is preliminary to the control of the surface coloring process.

In this paper a comparison between diffusion kinetics of methanol in polymethylmethacrylate samples, obtained by different processing technologies, is reported.

STUDY ON THE WORKABILITY OF PAINTS. FORCES EXERTED DURING BRUSHING AND THE RHEOLOG-ICAL CHARACTERISTICS OF PAINTS—Y. Kuge

Journal of Coatings Technology, 55, No. 701, 59 (June 1983)

The brush drag and the pressure applied on the brush were quantitatively measured under various brushing conditions using an apparatus which has a reciprocator, two-dimensional dynamometer, and brush. The theoretical equations to correlate the drag and pressure with rheological characteristics of paints were derived and the validity was tested with experiment. Equations for the drag, Fh, and pressure, Fv, were found to agree with the experimental results.

FACTORIAL EXPERIMENTAL DESIGN: DETERMINA-TION OF HYDROXYL CONTENT IN POLYMERS—B.O. Demarest and L.E. Harper

Journal of Coatings Technology, 55, No. 701, 65 (June 1983)

A statistical method for experimentation facilitated investigation of the following methods for determining hydroxyl content in polymers: an imidazole catalyzed esterification with pyromellitic dianhydride; two acid catalyzed acetylations utilizing perchloric and p-toluenesulfonic acids as catalysts; and a dibutyltin dilaurate catalyzed urethane formation with phenyl isocyanate. The statistical method involved a factorial experimental design where three independent variables were evaluated at two levels (two level three factorial design). This approach facilitated comparison of the methods for determination of hydroxyl content in polymers by efficiently optimizing reaction parameters.

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Federation News

Exhibitor Interest Insures 'Sellout' Of First Paint Industries' Show in Canada

In its initial appearance in Canada, the 48th Annual Paint Industries' Show will feature the exhibits of over 170 supplier companies who have purchased all available booth space. In addition, with over 40,000 sq. ft. of exhibit space, the 1983 Paint Show is the largest in Federation history.

The Show will be held October 12–14 in conjunction with the 61st Annual Meeting of the Federation at the Place Bonaventure in Montreal, Quebec.

The Paint Show is the only national exhibit of raw materials, and equipment used in the manufacture of paints and related coatings, and participating firms will have their top technical personnel on hand to discuss the latest developments in coatings manufacturing technology. The list of current exhibitors is on page 00.

Exhibit hours will be 11:30 am to 5:30 pm on Wednesday, October 12; 9:30 am to 5:30 pm on Thursday, October 13; and 9:30 am to 3:00 pm on Friday, October 14.

Annual Meeting Program

The 61st Annual Meeting program will open Wednesday, October 12, at the Place Bonaventure, and Program Chairman John Hiscocks, of CIL Paints, Toronto, Ontario, has announced the theme, "Knowledge Applied Profitably." He and his Program Steering Committee are developing a schedule of presentations around this topic.

Headquarters Hotel

The Queen Elizabeth will serve as headquarters hotel. Other hotels with blocks of rooms set aside for the Annual Meeting are the Hotel Bonaventure, L'Centre Sheraton, Regence Hyatt Montreal, Chateau Champlain, Hotel Meridien Montreal, Ramada Inn, Holiday Inn Downtown, and the Mt. Royal.

Room Reservations

All requests for rooms and suites must be sent to the Federation office on the official housing form which has been mailed to all members and is included in this issue (see pages 22-30). Additional housing forms are available from the Federation headquarters office.

Registration Fees

Advance registration fees are available for \$50 for members and \$65 for nonmembers. Fee for spouses activities is \$35 in advance.

Once again there will be a special registration fee of \$25 each for retired members and their spouses. This applies to advance registration only.

On-site registration fees will be \$60 full time and \$40 one day for members. Nonmember fees will be \$75 full time and \$50 one day. Spouses registration will be \$45 on-site.

Cash payment of on-site registration fees will be accepted in Canadian currency only at the then current rate of exchange. A currency exchange booth will be available in the registration area. Checks in both U.S. and Canadian funds will be accepted.

Registration forms were mailed to all members in April and are included in this issue (see pages 22-30).

Spouses Activities

Included in the spouses registration fee will be a get acquainted wine and cheese social on Wednesday afternoon; continental breakfast on Thursday and Friday at the Queen Elizabeth Hotel; and a tour of Montreal with lunch on Thursday.

Federation Luncheon

The Annual Federation Luncheon will be held on Friday at the Queen Elizabeth Hotel.

Presentations will be made to the recipients of the George Baugh Heckel Award (outstanding individual who has contributed to the advancement of the Federation) and the Flynn Awards (firms judged to have the best exhibit booths in the 1983 Paint Industries' Show).

The featured speaker will be David Broadfoot, Canadian humorist and political satirist.

Program Committee

Assisting Chairman Hiscocks on the Program Steering Committee are: Darlene Brezinski (Vice-Chairman), of De-Soto, Inc., Des Plaines, IL; N. Bradford Brakke, Lilly Industrial Coatings, Inc., Templetom, MA; Glenn Cunningham, PPG Industries, Inc., Allison Park, PA; John Flack, International Paints Ltd., Outremont, Quebec, Canada; and F. Louis Floyd, Glidden Coatings & Resins Div. of SCM Corp., Strongsville, OH.

Host Committee

Members of the Montreal and Toronto Societies are serving on the Host Committee under the General Chairmanship of Horace S. Phillip, Sherwin-Williams Co. of Canada, Montreal. Assisting him are: (Program Operations) Arthur Hagopian, CIL Paints, Inc.; (Information Services) Dennis H. Yokota, Consultant; (FSCT Booth) Andrew J. Jones, Degussa Canada Ltd.; (Registration) Jean Brunet, Van Waters & Rogers Ltd.; (Language) Andre Lamarre, Reichhold Ltd.; and (Publicity and Spouses' Program) Robert O. Payette, L.V. Lomas Chemical Co.

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1983 Paint Industries' Show

Current List of Exhibitors Engelhard Corp., Minerals & Chem.

Aceto Chemical Co., Inc. Air Products & Chemicals, Inc. Alcan Ingot and Powders Aluminum Co. of America C. M. Ambrose Co. Angus Co. Applied Color Systems, Inc. ARCO Chemical Co. Armstrong Containers, Inc. Ashland Chemical Co./Ashland Chem. Canada Atlas Electric Devices Co. B.A.G. Corp. BASF Wyandotte Corp. Beltron Corp. Berol Chemicals, Inc. Blackmer Pump Div., Dover Corp. Bowers Process Equipment, Inc. Brookfield Engineering Labs., Inc. Buckman Laboratories, Inc. Burgess Pigment Co. Byk-Mallinckrodt USA, Inc. Cabot Corp. Canada Colors & Chemicals Ltd. Canada Talc Industries Ltd. Canadian Paint & Ctgs. Association Cargill, Inc. CdF Chimie North America, Inc. **CDI** Dispersions Celanese Chemical Co., Inc. Celanese Specialty Resins CEM Corp. Certified Equipment Mfg. Co. Chemical & Engineering News (ACS) Chemische Werke Huels AG Chicago Boiler Co. Chromalloy, Ind. Minerals Div. Clawson Tank Co. Color Corp. of America Columbian Chemicals Co. Commercial Filters Div., Kennecott Corn Continental Fibre Drum Co. Cordova Chemical Co. of Michigan Cosan Chemical Corp.

Daniel Products Co. Degussa Corp. Frank E. Dempsey & Sons Ltd. Diamond Shamrock Corp., Process Chem. Div. D/L Laboratories Dominion Colour Co. Ltd. Dow Chemical USA Draiswerke, Inc. Drew Chemical Corp. DSET Laboratories, Inc.

Eastman Chemical Products, Inc. Ebonex Corp. E.C.C. America Eiger Machinery, Inc. Elektro-Physik, Inc. EM Chemicals Div. Erwin, Schafer & Associates, Inc. Fawcett Co., Inc. Fed. Socs. Coatings Technology Filter Specialists, Inc. Freeport Kaolin Co. Fricke Enterprises GAF Corp. Georgia Kaolin Co. W.R. Grace & Co., Davison Chem. Div.

Graco, Inc.

Halox Pigments, Div. Hammond Lead Prods. Harshaw Chemical Co., Surface Tech. Harshaw Chemical Co., Color Henkel Corp., Chemical Specialities Henkel Corp. Hercules Incorporated Dr. Hans Heubach Gmbh & Co. KG Hilton-Davis Chemical Group Hockmeyer Equipment Corp. Hooker Industrial & Specialty Chemicals Hoover Universal, Inc. J. M. Huber Corp. Hunter Associates Lab., Inc.

ICI Americas, Inc. Ideal Manufacturing & Sales Corp. Indusmin Ltd. Interstab Chemicals, Inc. ISC Alloys Ltd.

S.C. Johnson & Sons, Inc.

Kay-Fries, Inc. Kay Publishing Co. Ltd. Kenrich Petrochemicals, Inc.

Laporte (United States) Inc. Leneta Co. Liquid Controls Corp. Lorama Chemicals, Inc.

3M Co., Commercial Chemicals Div. Macbeth Div., Kollmorgen Corp.
Manville Products Corp., Filt. & Min. McCloskey Varnish Co.
Meadowbrook Corp.
Mearl Corp.
Merck & Co., Inc./Calgon Corp.
Miller Paint Equipment, Inc.
Mineral Pigments Corp.
Mini F1BERS, Inc.
Minolta Corp.
Mobay Chemical Co.
Modern Paint and Coatings
Morehouse Industries, Inc.
Myers Engineering

Nalco Chemical Co. National Assn. of Chemical Distributors National Assn. of Corrosion Engineers Netzsch Incorporated Neville Chemical Co. NL Chemicals/NL Industries, Inc. Northern Pigment, Div. CCMC Ltd. Nuodex, Inc. NYCO, Div. of Processed Minerals, Inc.

Ore & Chemical Corp. Ottawa Silica Co.

Pacific Scientific Co., Gardner/Neotec Paint Research Institute Penn Color, Inc. Pennsylvania Glass Sand Corp. Pfizer, Inc., MPM Div. Plastican, Inc. Polyvinyl Chemical Industries, Inc. PPG Industries, Inc. Premier Mill Corp. Purity Zinc Metals Co. Ltd.

Q-Panel Co.

Reichard-Coulston, Inc. Reichhold Chemicals, Inc. Reichhold Ltd. Reliance Products Ltd. Rohm and Haas Co. Russell Finex, Inc.

Sandoz Colors & Chemicals Semi-Bulk Systems, Inc. Shamrock Chemicals Corp. Sherwin-Williams Chemicals Sherwin-Williams Container Div. Sherwin-Williams Graphic Arts Div. Silberline Manufacturing Co., Inc. South Florida Test Service, Inc. Spencer Kellogg, Div. of Textron, Inc. Standard Container Co. Sun Chemical Corp., Pigments Div. Synray Corp.

Technology Marketing Corp. Thibaut & Walker Co., Inc. Thiele Engineering Co. Tioxide Canada, Inc. Troy Chemical Corp.

Union Camp Corp. Union Carbide Corp., Spec. Pol. & Comp. Div. Union Chemicals Div., Union Oil Co. United Catalysts, Inc. Universal Color Dispersions University of Detroit University of Missouri-Rolla

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

Wacker Chemical Co. Weathering Research Service Co. Wellco Products & Itasco Ind. Wilden Pump & Engineering Co. Witco Chemical Corp.

Zeelan Industries, Inc. Carl Zeiss Canada Ltd.

BETTER PAINT. IT'S IN THE BAG. Celite: the consistent, high quality filler... in a wide variety of grades.

Manville Celite is mined from separate quarries at the largest diatomite producing location in the free world. Each quarry produces a distinct, but consistent composition. Adjacent to the quarries is the world's largest diatomite milling plant, capable

of processing large quantities in a wide variety of grades.

The very size of the operation enables long production runs and thus, less changeovers. The result, a highly consistent product. Twenty-four-hour-a-day quality control sampling procedures also assure grade consistency.

Because the Manville mine operation has five separate calcination systems, more Celite grade varieties are possible . . . each controlled to the closest possible tolerances.

The operation provides standard grades as well as grades for special applications. For example, extensive marketing and technical experience has created

special grades for the paint industry. Each offers its own unique, consistent characteristics providing you maximum flexibility and control of your paint product.

So whether your market is industrial or consumer, if you need consistent quality and uniform gloss/sheen control, it's in the bag... the Manville Celite bag.

For additional information on suggested grades of Celite filler and usage ratios, or our complete line of filter cartridges, call your Manville sales representative or write: Manville, Filtration and Minerals, P.O. Box 5108, Denver, CO 80217. (303) 978-2656.

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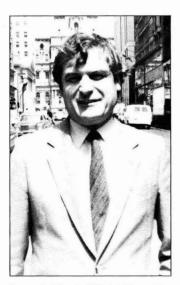
Federation News (continued)

Polymer and Coatings Students Aided by FSCT Scholarship Funding

The Federation, through its Educational Committee (James A. Hoeck, Chairman), currently offers scholarship monies to six universities which have a curricula in polymers and coatings technologies. These schools are: Eastern Michigan University, Ypsilanti, MI; Kent State University, Kent, OH; North Dakota State University, Fargo, ND; University of Detroit, Detroit, MI; University of Missouri-Rolla, Rolla, MO; and University of Southern Mississippi, Hattiesburg, MS.

Although eligible children of Federation members receive preference in scholarship selection, the main objective is to provide a continuing supply of welleducated, technical personnel for the coatings industry.

The following is a list of Federation scholarship recipients and their present status:



A recent visitor to Philadelphia and the Federation office was John Gilmour, Secretary of the Oil and Colour Chemists' Association-Australia. Mr. Gilmour, who is Registrator of the National Association of Testing Authorities, Chatswood, Australia, was in Philadelphia for meetings at ASTM

Eastern Michigan Univ.

CURRENT RECIPIENTS: John Crimmins Thaddeus Lepkowski

Kent State Univ.

1982 GRADUATES: Adarsh Krishen, Northeast Ohio College of Medicine, Rootstown, OH Daniel Sayre, Research Organics, Inc., Cleveland, OH 1983 GRADUATE: Cynthia Colby, Uncommitted CURRENT RECIPIENTS: Janet Domer, North Canton, OH Alita Durney, Warren, OH Ferdinand Fojas, Steubenville, OH Tricia Richardson, Hudson, OH Glenn Rothhaas, Tallmadge, OH

North Dakota State Univ.

CURRENT RECIPIENTS:

Gary Asmus, Lakeville, MN Steve Conyne, Bismark, ND Bruce Harkness, Moorhead, MN Gregory Kaster, Erskine, MN Thong Bao Ky, Fargo, ND Rick Lau, Fargo Micheline Liddle, Fargo Mark Murphy, Kansas City, MO Alan D. Rott, Bismark

Univ. of Detroit

CURRENT RECIPIENTS: Tony Busuttil, Windsor, Ont., Canada Charles W. Kirby, Farmington Hills, MI Jagdish P. Tarpara

Univ. of Missouri-Rolla

1982 GRADUATES. Laura Plyler, Uncommitted Steve Roth, Carboline Co., St. Louis, MO Garth Studebaker, Pursuing Master's Degree CURRENT RECIPIENTS: Mike Beckman Mark Monique **David Price** Marlene Sermeno

Univ. of Southern Mississippi

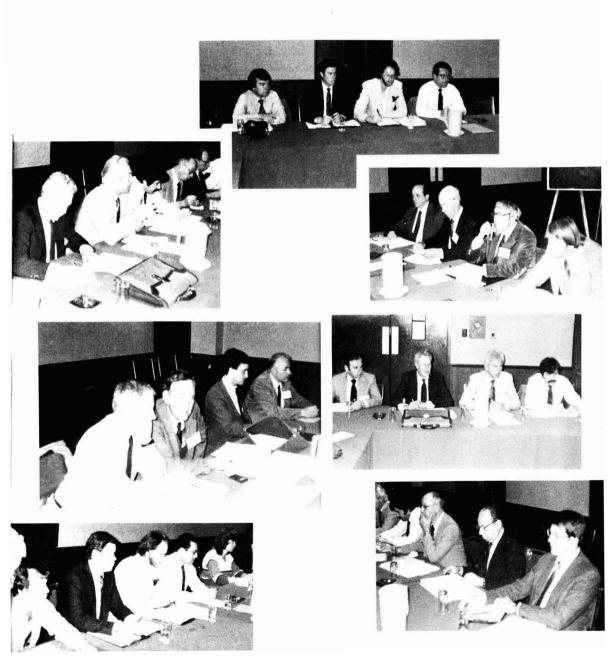
1982 GRADUATES: Ralph M. Gonzalez, Martin-Marietta Aerospace Corp., New Orleans, LA Patricia M. Lewis, Sylacauga Calcium Products, Sylacauga, AL Alan K. Randall, PPG Industries, Inc., Pittsburgh, PA Gregory L. Wascomb, Bywater Coatings, Belle Chasse, LA CURRENT RECIPIENTS: Terry Asher Brian D. Blackledge Robert H. Bloodworth Manuel L. Bosarge Daniel L. Butcher Jon C. Carr Glen S. Cook Kenneth W. Cooley Carla T. Cotruvo K.Y. Craft Dianne L. Dearman Sonja A. Draughn Robert J. Halley Brent G. Howell Thomas M. Keel Patrick J. Kergosien Brian J. Kinkopf Teresa A. Kloske Susan D. Leonardt James C. Lockard Angela L. Marks Walter D. Muse Tammy L. Nattrass Wayne K. Ng Terry B. Parsons Leah A. Peavy Michael E. Price Rhonda K. Purer *Franklin D. Rector John G. Santobianco Gordon A. Smith Charles A. Smith William M. Sullivan Rebeca L. Sullivan Phillip H. Tally Randall L. Thompson Joseph G. Tomei Robert T. Watts David B. Wiggins

*son of Federation member

For information concerning scholarship availability and application, please contact the Federation headquarters office at 1315 Walnut St., Philadelphia, PA 19107.

Society Technical Committee Chairmen Meet with FSCT Advisory Committee

For the eighth year, chairmen of the Technical Committees of Constituent Societies attended a joint meeting with the Federation Technical Advisory Committee (TAC) to discuss their respective programs. Society members in attendance April 28–29, in Kansas City, were: David Heath, of Birmingham Club; Gerry K. Noren, of Chicago; Mike McGroarty, of C-D-I-C; Carl J. Knauss, of Cleveland; Richard G. Gohman, of Dallas; Tim Specht, of Golden Gate; Elder C. Larson, of Houston; Richard McFadden, of Kansas City; V.C. "Bud" Jenkins, of Los Angeles; Don Collier, of Louisville; Luigi Cutrone, of Montreal; Maureen M. Lein, of New England; Dan DeChaine, of Northwestern; Lothar Sander, of Philadelphia; John Hajnos, of Piedmont; William Meadows, of Rocky Mountain; and Rob DeRuiter, of Toronto. TAC members included: Chairman William Holmes, of Dallas; Jan Grodzinski, of Toronto; Colin Penny, of Baltimore; and Saul Spindel, of New York. Also attending were: Royal A. Brown, FSCT Technical Advisor; Chuck Reitter, Editor of *American Paint & Coatings Journal;* Thomas A. Kocis, FSCT Director of Field Services; and Robert Ziegler, Editor of JCT.



Consensus Rule on PCBs Submitted to EPA

The Chemical Manufacturers Association and two public-interest organizations have submitted to the Environmental Protection Agency a proposed "consensus final rule" covering the "inadvertent generation" of polychlorinated biphenyls (PCBs).

The proposed censensus final rule, which was presented to EPA officials at a meeting held at the agency's Washington, DC headquarters, was developed by CMA, the Natural Resources Defense Council, and the Environmental Defense Fund. It is the result of several months of discussions between the three organizations aimed at reaching an agreement on how PCBs that are inadvertently generated as an unwanted byproduct of various manufacturing processes are to be regulated.

CMA President Robert A. Roland called the proposed consensus rule significant for two reasons. First, it establishes the framework for effective and reasonable controls over the inadvertent generation of PCBs. Second, and equally important, it represents what the industry hopes will be the beginning of a period of greater cooperation between industry and organizations like NRDC and EDF.

CMÅ, EDF, and NRDC have demonstrated that negotiation and agreement are not only possible between the organizations, but are more beneficial and productive alternatives to litigation.

PCBs are no longer commercially manufactured in this country but remain in equipment such as electrical transformers installed before the government banned PCBs in 1979. PCBs are also unintentionally generated in many manufacturing processes.

PCBs are organic chemicals which contain various configurations of chlorine, carbon, and hydrogen. Some PCBs are persistent and resist breaking down in the environment into more simple compounds. Since the ban four years ago, the amount of PCBs in the environment has steadily declined because of curtailed use, proper disposal, and environmental degradation.

The proposed consensus rule is a result of negotiations between the three organizations that followed a 1980 federal appeals court decision that struck down parts of the agency's original regulations implementing the Toxic Substances Control Act. If adopted by EPA, the consensus rule would be the latest in a series of regulations covering PCBs following the court's decision. The proposed consensus rule would specify how inadvertently generated PCBs are to be measured and establish concentration limits on release of PCBs to products, air, water—25 ppm average per year and 50 ppm at any given time in products; 10 ppm at any given time for releases to air; and 0.1 ppm in water discharged from manufacturing sites. Also, it would maintain process waste disposal requirements and create reporting mechanisms by which EPA will be informed of any significant quantities of PCBs released into the environment or in products.

Battelle to Study Markets For Application of Plasma Technology

A study to forecast uses and markets for applications of plasma technology to polymer coatings and surface treatments is under way at Battelle's Columbus Laboratories, Columbus, OH.

The study also will review the current state of the art of plasma polymerization in the deposition of polymeric materials. It is being sponsored by a number of companies, and will help participants determine whether plasma polymerization represents an opportunity or a threat to their operations.

According to Dr. Edmund J. Drauglis, who heads Battelle's study team, economical and environmental considerations recently have prompted increased interest in plasma technology and its potential applications.

The technology can be used to apply thin, pinhole-free, conformal, adherent polymeric coatings to a variety of substrates, such as glass, metals, wood, paper, plastics, and elastomers.

Such coatings are particularly useful for resistance to corrosion, abrasion, and moisture; lubrication; biocompatibility; and gas-barrier selectivity.

They also can be used to modify surfaces of polymeric materials. In this way, they can be used for cleaning surfaces, improving adhesion or printability, reducing tack, or for activating surfaces for subsequent grafting reactions.

In addition, because plasma polymerization does not use solvents, as do conventional coating methods, it is pollution-free. In the face of environmental restrictions, this characteristic of plasma polymerization is making the process attractive to many companies.

To carry out their study, Battelle researchers are analyzing available data and gathering field information about various subject areas, including materials and equipment, processing, coatings, and surface treatments, properties of coatings and treated surfaces, process economics, applications, markets, patents, and new concepts.

Researchers then will access the information before analyzing and forecasting market size through 1992.

Participation in the one-year study is still available to interested companies for \$8,500. More information may be obtained from Dr. Edmund J. Drauglis, Battelle's Columbus Laboratories, 505 King Ave., Columbus, OH 43201.

Hazardous Waste Program Proposed by Battelle

The challenges involved in managing and safely disposing of hazardous wastes will be addressed in a new group program being proposed by Battelle-Geneva.

The program will help companies identify and develop containment matrix systems and materials for the terminal storage and disposal of wastes.

During the study, researchers will:

- Review the cost-effectiveness of containment matrix systems based on cements, silicates, inorganic and polymeric materials, and composites suitable for encapsulating hazardous wastes.
- Assess the long-term durability of waste/matrix composites under a variety of environmental conditions.
- Analyze potential applications of encapsulation-isolation technologies and final storage/disposal.
- Evaluate future trends in waste treatment, containment matrix systems, and final disposal management.

For information on the program, please contact Battelle Memorial Labs., 505 King Ave., Columbus, OH 43201.— Battelle Today

Journal of Coatings Technology

GLOSS TO MEASURE

Powder coating hardeners for high-gloss to dead-mat coatings – without additives

Fashion trends and many application conditions call for different degrees of gloss – of powder coatings, too. By selecting one or a combination of our special hardeners B 31, B 55, B 68 or B 73, coatings, from highgloss to mat, can be achieved without additives for epoxy powders or blends (hybrids). And this with good resin and pigment compatibility.

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- resistant to chemicals and detergents

• very good corrosion protection Post the adjacent coupon for more details. And we shall be pleased to give you technical advice.

Our product range equally includes:

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Federation of Societies for Coatings Technology

61st ANNUAL MEETING 48th PAINT INDUSTRIES' SHOW



1983

HONTRÉAL Place Bonaventure October 12, 13, 14



1983 Annual Meeting Paint Industries' Show October 12, 13, 14 Place Bonaventure Montreal, Quebec, Canada

TO OUR MEMBERS AND FRIENDS:

As the fifth Canadian President of the Federation, it is a distinct pleasure and privilege for me to invite you to the first Annual Meeting and Paint Industries' Show to be held in my native country.

The two host Societies—Montreal and Toronto—are eagerly awaiting this event and have been very busy in making arrangements so that you will have an enjoyable and memorable visit to the beautiful city of Montreal.

The Paint Show, which we expect will be the biggest ever, will be held in one of Canada's top showplaces, Place Bonaventure. This magnificent building, truly a city within a city, sits on top of another city within a city—Place Ville Marie.

Program sessions will be geared to the theme of the meeting, "Knowledge Applied Profitably." The selection of papers, workshops, and seminars being arranged by Peter Hiscocks and his Program Committee will send you home with a pocketful of ideas to put to profitable use in your companies.

On behalf of the 800 Canadian members associated with the Montreal, Toronto, Detroit, Northwestern, and Pacific Northwest Societies . . . Bienvenue a Canada.



A. Clarke Boyce President Federation of Societies for Coatings Technology

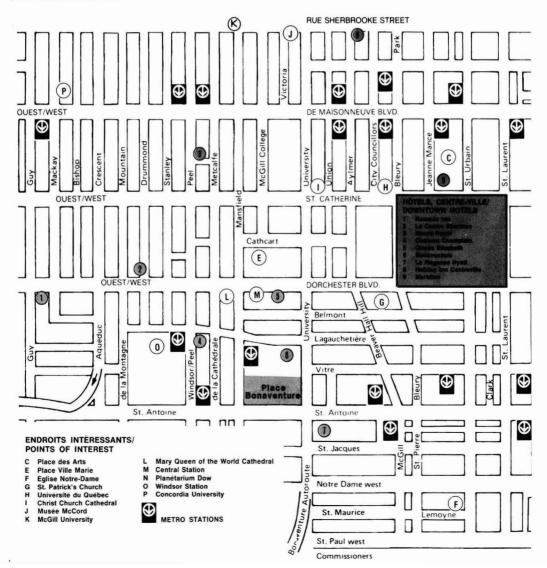


HOTEL INFORMATION AND RATES

All Rates are in Canadian Funds

Singles	Doubles Twins	Parlor & 1 Bedroom	Parlor & 2 Bedrooms	Parlor & 3 Bedrooms
		\$208/220/246	\$376/382/305	
\$79	\$89	271/280	420	\$544/635
\$95	\$105	\$250/400	\$475	\$525
\$83	\$96	\$200/426	\$430	
		\$164/220/330	\$184/220/330	
\$83	\$95	480/600	480/600	
\$84	\$96	\$210/275	\$375	
\$78	\$88			
\$59	\$69			
\$68	\$74	\$105		
\$53	\$63			
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*Requests for accommodations at either the Queen Elizabeth or the Bonaventure will be limited to five rooms per company. A parlor counts as one room.



FSCT 1983 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW PLACE BONAVENTURE, MONTREAL, QUEBEC, CANADA OCTOBER 12, 13, 14 (Wednesday, Thursday, Friday)

APPLICATION FOR HOTEL ACCOMMODATIONS

MAIL	Fed. Socs. Coatings Tech.
TO:	1315 Walnut St.—Dept. H
	Philadelphia, PA 19107

All reservations will be processed by the Montreal Convention and Visitors Bureau.

Please indicate below the type of accommodations desired and choice of hotels. Assignments will be made in accordance with prevailing availability. Three to four weeks after mailing this application you will receive a confirmation from the hotel to which you have been assigned.

To make any inquiries regarding hotel reservations, please observe the following procedure:

Prior to Receiving a Confirmation: Phone the Housing Supervisor of the Montreal Convention and Visitors Bureau. (514-871-1129).

After Receiving a Confirmation: Phone the Reservations Dept. of the hotel. (Phone numbers are given in this brochure).

All reservations will be held until 6:00 p.m. and none can be guaranteed after September 12.

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

CHOICE OF HOTELS:
1st
2nd
3rd
4th

NAMES AND ADDRESSES OF ROOM OCCUPANTS AND DATES OF ARRIVAL/DEPARTURE

Type of			Dates	
Type of Room	Name	Address	Arrive	Depart
	N/- TL			
		6-6 - 10 W		

Please Type Additional Reservations on a Separate Sheet and Attach to This Form

SEND CONFIRMATION FOR ALL RESERVATIONS TO:

Name	
Company	
Address	
City	State or Province
Country	Mailing Code
Note: Requests for accommodations a	t either the Queen Elizabeth or the Bonaventure will be limited to five rooms per

Note: Requests for accommodations at either the Queen Elizabeth or the Bonaventure will be limited to five rooms per company. A parlor counts as one room.

HORTRÉAL

THE CITY

There's no place like Montreal. Here you'll discover a blend of the best of French and English, of European and North American ways. Montreal is the second largest French-speaking city in the world, and wholly cosmopolitan.

The old and new co-exists comfortably in Montreal, and for all its respect for tradition, this is one of the world's best planned cities. Montreal is a major international business center, a major university center, and the largest inland port in the world. And, it is also a city of arts, home to La Place des Arts, one of the world's best-known entertainment centers.

The "City Below" is Montreal's answer to downtown congestion. Instead of building skyscrapers in the air and chaos on the ground, Montreal put into action an idea first suggested more than 500 years ago by Leonardo da Vinci—a multilevel city which separates people from traffic so that both can move freely: cars and trains through separate tunnels and free-ways, pedestrians through their own enclosed, air-conditioned streets.

The City Below stretches eight miles beneath the avenues of Montreal. Brief, silent (rubber-tires) Metro rides lead to boutiques, department stores, hotels, restaurants, theatres, art galleries and more.

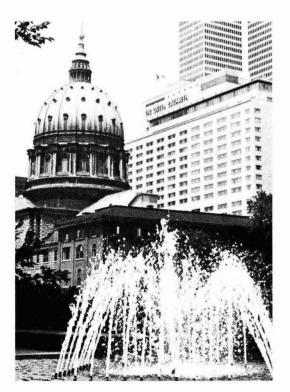
OLD MONTREAL

People frequently portray Montreal as "a little bit of Europe in North America." This description is particularly applicable to that part of the city known as "Old Montreal," the site of the early colonization of the region. The first permanent settlement of Montreal was established by the French explorer, De Maisonneuve in 1642. The early history of French Canada is preserved through the existence of many of the original structures, as well as statues and plaques commemorating the exploits of early explorers, settlers, and military figures.

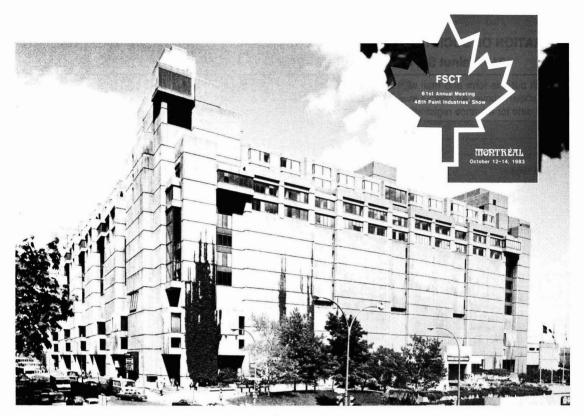
A stroll through Old Montreal is a treat for even the most casual observer. Some of the oldest and most beautiful architecture in North America is to be found in this quarter.

WEATHER

During the month of October, Montreal's climate ranges from a low of 46° to a high of 57°. Medium to heavy-weight apparel will keep you comfortable. Autumn draws out the best in Mother Nature. Bring your camera to capture the "gold rush" in thick autumn foliage.







Place Bonaventure

AIR TRANSPORTATION

Two major airports serve the city: Dorval, located 16 km (10 miles) from downtown, which handles flights to and from all points in Canada and the U.S.; Mirabel, some 55 km (35 miles) from downtown, for international passenger traffic.

THINGS TO DO

Montreal's 5,000 restaurants, of which more than 100 are internationally rated, serve dishes and specialties from all corners of the earth, making Montreal a gourmet's paradise.

Exquisite French cuisine, wholesome French Canadian dishes and quick snacks are readily available throughout the city, providing quality, variety and convenience.

Vegetarians and health food enthusiasts will find that Montreal leads the way in the preparation and service of specialty foods. Kosher and seafoods also number among Montreal's specialties. The city's culinary shops and delicatessens offer meats, cheeses and other specialties from all over the world.

Montreal has justly earned its reputation as a "city of the world," where life is "a la Quebecoise," at an American pace, with a touch of European charm. It is stimulating, exciting and truly cosmopolitan.

GETTING THERE

If you are a citizen or permanent resident of the U.S., you can cross the Canadian border and return without any difficulty or delay. Passports or visas are not required. Native-born U.S. citizens should carry identification papers such as a birth, baptismal or voter's certificate, and proof of residence. Naturalized U.S. citizens should carry a naturalization certificate or other evidence of citizenship.

After 48 hours in Canada, U.S. residents may take home, duty free, \$300 worth of goods for personal and household use. These must accompany the traveler. Family members traveling together may combine their personal exemptions.

PLACE BONAVENTURE

Place Bonaventure is a city within a city. It's a trade city: marketplace of the world's buyers and sellers, showplace for the merchandise of many nations and a rich source of information on trade and tourism.

It covers a six-acre multilevel site, containing not only the Exhibition Hall, where Paint Show exhibits will be on display, but a busy, bustling, boutiquefilled Shopping Concourse, complete with stores, bars, restaurants, movies, entertainment, activities and services. And, atop the Place Bonaventure complex, is Hotel Bonaventure with its rooftop gardens.

1983 ADVANCE REGISTRATION

FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY

1315 Walnut St., Philadelphia, PA 19107

Please fill out this form and mail with a check in the correct amount (made payable to the FSCT) to the Federation address shown above. All checks must be payable in U.S. Funds, and in U.S. Banks. Any that are not will be returned. Deadline date for advance registration is September 9. None will be accepted after that date.

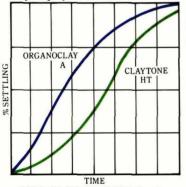
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Society		<u> </u>											
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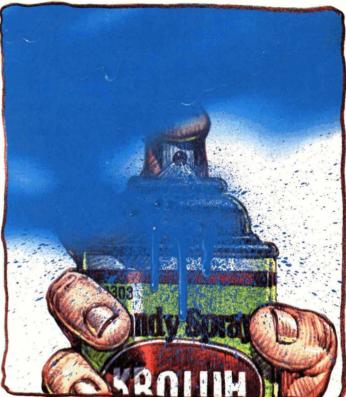
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OCTOBER 15-17, 1983

Saturday, October 15

9:00 am -- Depart downtown Montreal hotel by charter bus for Quebec City, taking the scenic "Chemin du Roi" highway.

1:00 pm -- Approximate arrival at Quebec City -- Chateau Frontenac Hotel. No planned activities.

Sunday, October 16

2:00 pm -- Sightseeing tour of Quebec City and then to Ste. Anne de Beaupre Shrine.

Monday, October 17

10:00 am -- "Au Revoir" Quebec. Board bus for return to Montreal Airport (Dorval). Arrive approximately 12:30 pm (express route). Transportation to Mirabel Airport available from Dorval Airport.

> PRICE: \$127.50 U.S. per person - twin occupancy \$152.50 U.S. per person - single occupancy

Included: - Round trip transportation by chartered bus.

- Two nights hotel accommodation, based on twin or single occupancy.

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<u>Deposit Required</u>: \$50 U.S. per person with Registration Form - plus optional Cancellation Insurance (\$8.50) in case of illness.

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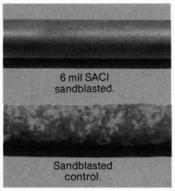
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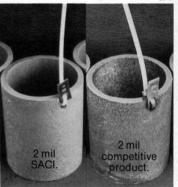
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Effect of Molecular Weight On Performance of Cellulosic Thickeners In Latex Paints

D. M. Blake The Dow Chemical Company*

A latex paint was formulated in five versions, each thickened with hydroxypropyl methylcellulose of a different molecular weight. The five were brought to essentially equal viscosity for comparison of thickener efficiencies. To determine and compare other effects of thickener molecular weight variation, the five were evaluated for: rheology at lowshear and high-shear rates; flow and leveling characteristics; spatter resistance; foaming tendency; scrub resistance; and color acceptance. Thickener molecular weight was found to affect the paint's high-shear rheology, spatter resistance, and foaming tendency; it has no effect on low-shear rheology, scrub resistance, or color acceptance. The color acceptance testing indicated shortcomings in the commonly used hand rubup test procedure.

INTRODUCTION

Virtually all latex paint formulations in current use include a cellulosic thickener to increase the viscosity of the finished paint to an acceptable and desirable level. The established thickener materials are hydroxypropyl methylcellulose and hydroxyethylcellulose. Since each of these cellulosics is commercially available in a range of varying molecular weights, the formulator enjoys considerable choice among thickeners. But he has little guidance for the choice.

It is generally known that the higher the molecular weight of the thickener used, the less the amount of thickener needed to bring a latex paint to the desired Stormer viscosity. Also, it is generally accepted that the

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rheology of a latex paint is primarily determined by the percent solids and the thickener level in the formulation. Beyond these generalities, however, the literature offers little on how the physical properties of a paint, other than Stormer viscosity, may be affected by varying the molecular weight of the thickener used.

This study was undertaken to develop information useful in choosing between cellulosic thickeners of differing molecular weights. It identified several paint properties significantly affected by molecular weight of the thickener used. It established that paints formulated with thickeners of different molecular weights can be objectively evaluated and compared by existing test procedures.

MATERIALS

The paint used in this study was an interior flat vinyl acrylic latex, formulated as indicated in *Table* 1. As formulated, the paint had a 50% pigment volume concentration (PVC) and a density of 11.43 lb/gal. Five versions of the model formulation were prepared, all versions employing hydroxypropyl methylcellulose as a thickener, each specific thickener having a different molecular weight and hence, a different nominal viscosity.

All the thickeners used were surface-treated powders, marketed as Methocel® brand cellulose ethers, and designated as Methocel J products. These powders do not lump when added directly to nonalkaline water and, if no pH adjustment is made, hydration is delayed for up to an hour. However, once the thickener is dispersed, adjustment of the pH to 8.5 will cause hydration to begin immediately. The powders are available in nominal viscosity grades of 5,000, 12,000, 20,000, 40,000, and 75,000 cps—the values being for 2% w/w water solutions.

^{*}Dow Chemical U.S.A., Designed Products Dept., 2040 Dow Center, Midland, MI 48640.

Ingredients	lb/100 ga
Water	. 327.5
Tergitol® NP4	. 4.0
Potassium tripolyphosphate	
Tamol® SGI	. 1.5
Troysan® PMA30	. 0.5
Ethylene glycol	
Texanol®	
Camel-CARB®	. 125.0
TiPure® R900	. 175.0
ASP® 170	. 125.0
Drew® L 405	. 2.0

Table 1—Formulation for the Test Paint, An Interior Flat Vinyl Acrylic Latex (PVC, 50%; density, 11.43 lb/gal)^a

The ingredients listed above were loaded to a Cowles dissolver, in the order shown, and ground at 1200 rpm for 20 minutes. The combination was then let down as follows, decreasing the vortex speed as the let down progressed:

UCAR® 366	 	• •							•	•					•		 		285.0
Drew L 405 .																			
Methocel® J	 						 			•		 							Varied ^b
Water	 						 			•		 					 		40.0
AMP® 95	 	• •		•	•		 			•	• •	 							0.5

(a) pH of each of the five batches of test paint was held within a range of 9.0 to 9.5.(b) Variations in thickener loadings are detailed in *Table 2*.

STUDY PROCEDURES AND OBSERVATIONS

Comparative Efficiencies of Thickeners

As noted initially, cellulosic thickeners are used in latex paint formulations to increase the Stormer viscosity of the paint to an acceptable level. In this study all five versions of the test paint were formulated to essentially the same Stormer viscosity. The acceptable range for viscosity was set at 95–100 Kreb units and the Stormer viscometer was used, in the procedures set by ASTM D562-55, to confirm that test paints were within the range. As expected, and as shown in *Table* 2, the thickener loadings decreased with increasing molecular weight of the specific thickener used.

Decreasing the thickener loading of a paint formulation obviously decreases the materials cost per gallon of paint produced. Thus, if lowest-attainable cost were the

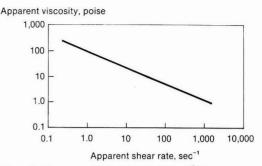


Figure 1—Rheogram of test paints in 1-1,000 sec⁻¹ shear range as developed by Haake Rotovisco Rotational Viscometer

only criterion in selecting ingredients, the thickener of highest molecular weight would be the material of choice. But real-world experience makes it advisable to determine what properties other than Stormer viscosity, if any, are affected by thickener molecular weight.

Rheology

The ideal rheological profile for a latex paint avoids extremes in both high-shear viscosity and low-shear viscosity. A viscosity that is too low at the lower shear rates means the paint is likely to sag after application and before drying; but if viscosity is not low enough in lowshear conditions, the paint may hold brush marks rather than flowing and leveling. At the same time, excessive high-shear viscosity creates brush drag and brings on arm fatigue for the painter, while too low a high-shear viscosity causes poor film build and makes overspreading beyond the recommended coverage area more likely.¹

Two series of rheological studies were made with the test paints. One series used the Haake Rotovisco Rotational Viscometer to examine paint viscosities over a shear-rate range of $1-1,000 \text{ sec}^{-1}$, simulating the low shear occurring as a paint is stirred and poured. The second series used the ICI Viscometer to measure the paint viscosities at a $10,000 \text{ sec}^{-1}$ shear rate, which simulates the high end of the shear-rate scale to represent roller, brush, and spray applications.²

Table 2–	-Thickener	Loadings
And Stormer	Viscosities	of Test Paints

Test Paint	Thickener Viscosity (nominal, cps)	Thickener Loading (Ib/100 gal)	Stormer Viscosity (Kreb units)
A	5,000	6.0	98
Β	12,000	4.8	98
С	20,000	4.2	95
D	40,000	4.0	96
Ε	75,000	3.2	95

Table 3—Apparent Viscosity of Test Paints At 10,000 sec⁻¹ Shear Rate^a

Test Paint	Thickener Viscosity (nominal, cps)	Apparent Viscosit (poise)							
Α	5,000	1.2							
В	12,000	1.05							
с	20,000	1.02							
	40,000	0.95							
	75.000	0.88							

(a) Apparent viscosities measured by ICI Viscometer.

EFFECT OF MOLECULAR WEIGHT

Figure 2—Drawdown of a test paint under low-angle lighting for comparison to standards in rating of flow and leveling characteristics

The Haake Viscometer was chosen for the low-shear studies as a convenient means for obtaining reproducible measurements of the viscosities involved. This instrument utilizes a cup-and-bob mechanism which is coupled to a recorder calibrated for the liquid being stressed. In this study, the paint sample was placed in the cup and then the cylindrical bob was inserted and rotated, at controlled speed, to induce shear in the liquid between the edge of the bob and the wall of the cup. The shear rates were recorded as apparent viscosities for plotting as a rheogram.

Figure 1 shows the rheogram developed when the five test paints were examined with the Haake Viscometer. Only one profile curve is apparent because the five formulations showed virtually identical viscosity response over the $1-1,000 \text{ sec}^{-1}$ shear-rate range. At relatively low shear rates, therefore, viscosity appears to be independent of thickener molecular weight.

The ICI Viscometer was chosen for the high-shear studies. In using the ICI Viscometer, one or two drops of the test paint are placed on a plate surface heated to 25° C (77° F), the cone is engaged to the plate and locked, and the instrument is then run for 10 seconds. After 10 seconds, the viscosity value shown on the indicator is read and recorded in poise.

Table 3 shows the high-shear viscosity values, at the $10,000 \text{ sec}^{-1}$ shear rate provided by the ICI Viscometer, of the five test formulations. The measurements indicate thickener molecular weight has significant effect on paint viscosity at the very high shear rates, with the lowest molecular weight thickener yielding the paint with the highest such viscosity.

The trend apparent in the data prompts the question: Does this trend continue, so that a latex paint formulated with a cellulosic thickener of still-lower molecular weight would have a high-shear viscosity in the range of 1.5 to 2.0 poise?

To explore this point, a batch of the test formulation was prepared with a thickener having nominal solution

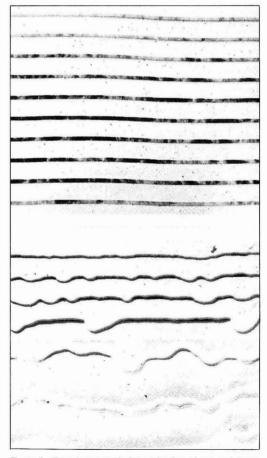


Figure 3—Drawdowns made for evaluation of sag resistance properties of a test paint

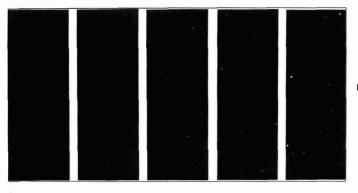


Figure 4—Spatter patterns produced with the five versions of the model paint formulation

viscosity of 1700 cps. ICI Viscometer study put the highshear viscosity of this paint at 1.2 poise—identical to that of Test Paint A. This plateauing suggests there is a limit beyond which decreasing the molecular weight of the cellulosic thickener produces no further significant increase in the high-shear viscosity of the paint.

An ICI Viscosity of 2.0 poise is usually considered the upper limit for an acceptable latex paint. The study results indicate the formulator can more closely approach this value, and still keep low-shear viscosity within an acceptable range, by using a thickener with lower, but not necessarily the lowest, molecular weight.

Flow and Leveling

The cellulosic thickeners used in latex paints solubilize by association of water molecules in hydration layers along the polymer chain constituting the thickener. The association of the water molecules occurs through hydrogen bonding of the active hydrogen in the cellulose backbone as well as in the substituent groups. Shear applied in the painting procedure disturbs the hydration layers and, at shear rates approaching those involved in

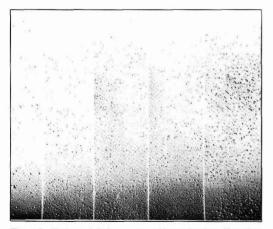


Figure 5—Test panels being compared for evaluation of foaming tendencies. For this part of the study, the flive versions of the model paint formulation were modified by removal of the defoamer agent

brush or roller applications, results in a very rapid decrease in viscosity.

Thus, at the moment paint leaves the roller, brush, or spray nozzle, disturbance of the hydration layers is maximum and viscosity is minimum. But these conditions are short-lived. With the removal of shear, the hydration layers rebuild quickly to develop a viscosity at the higher end of the low-shear range. It is this restored viscosity that limits the flow and leveling characteristics of a latex paint.

Flow and leveling relate to the tendency of a paint, when newly-applied and no longer subject to the shearing effect of the roller, brush, or spray used in application, to continue to flow under the low-shear conditions induced by gravity. It is desirable that the paint flows sufficiently to level out irregularities in the film surface, yet resist flow sufficiently to prevent runs or sags in paint applied to vertical surfaces.

The Leneta Leveling Test was used to study flow and leveling of the model paints. For this test, 10 grams of paint are applied by syringe onto a Leneta WB chart and are drawn down in a pattern of stripes. The Leneta Leveling Test Blade used for the drawdown controls paint thickness so that alternate stripes are 6 mils and 2 mils thick. The chart is air-dried in a dust-free cabinet and then placed under a low-angle light source for side-byside comparison against standards. In the comparison, the test films are rated on a 1-to-10, poor-to-excellent scale for surface irregularities and sags. *Figure* 2 shows the rating procedure.

When the films produced in these tests were compared against standards, the leveling characteristics of all five paint versions were rated as practically equal. The equality indicates that variation in thickener molecular weight has no noticeable effect on leveling characteristics of the paint.

Sag tendencies of a paint are evaluated by determining how thickly the paint can be applied before gravityinduced flow shows up as sag. The Leneta Antisag Test developed by the Leneta Co. provides a practical and reproducible method for these determinations. For sagantisag studies, a drawdown of the sample paint is made on Leneta 7B chart paper in a pattern of stripes. The stripes, separated by narrow bands of unpainted chart surface, are of equal width but progressively greater



Figure 6—Test panels coated with the model formulation provided a final comparison, and control, for the tests of foaming tendencies. The pattern seen here was induced by the roller used

thickness. After the drawdown, the chart is allowed to dry in a vertical position and with the drawdown stripes running horizontally. Thus, sag occurring anywhere along the length of any stripe shows up as intrusion into an unpainted area. The thickness, in mils, of the thickest stripe that does not sag to touch the stripe below itself is called the antisag number.

The test blade commonly used for antisag study produces drawdown stripes in thicknesses from 3 mils to 12 mils. The drawdowns of the five test formulations made with this 12-mil bar developed no noticeable sagging. To determine antisag limits, new drawdowns were made with a bar producing stripes of 12-mil to 60-mil thickness. Again, all five test paints showed the same results: sagging occurred in all five samples when paint thickness was 14 mils or more, so that each sample received the same antisag number of 12. *Figure* 3 shows the Leneta Antisag Test chart, with both sets of drawdowns, for one of the test samples.

This part of the study makes it evident that molecular weight of the thickener has no significant effect on sagantisag characteristics of a latex paint.

Spatter Resistance

Glass has done the definitive work to identify and evaluate factors influencing roller-induced spatter of latex paints.³⁻⁴ Working with thickeners of different chemical compositions, including hydroxyethyl cellulose

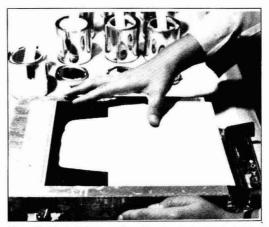


Figure 7—Typical charts produced in rubup tests of test paints in various colors

(HEC) but *not* hydroxypropyl methylcellulose, Glass observed that as thickener molecular weight increases, the amount of spatter increases.

The spatter tests done for the study reported here, while not replicating Glass's methodology, confirm that his findings also apply to hydroxypropyl methylcellulose thickeners. For this study, the spatter tests involved painting a vertical surface placed 12 inches above a black plastic panel laid flat to catch the spatter. An area 1 ft² marked on the vertical surface was coated in nine passes with a 3-inch HHR-3 roller made by the Shur-Line Manufacturing Co. A set of the spatter patterns produced with the five test formulations appears in *Figure 4*.

The visually obvious differences in the extent of the spatter patterns establish a definite correlation between thickener molecular weight and spatter. The results show that, as with thickeners of other chemical compositions, increasing the molecular weight of an hydroxypropyl methylcellulose thickener increases the spatter tendencies of a latex paint.

Foaming

Cellulosic thickeners act as mild surfactants. Thus, they contribute to the foaming tendencies which, in latex

Table 4	-Scrub Resistance of	Test Paints
As Indicated by	Gardner Scrub Test	(ASTM D2486-66T)

Test Paint	Thickener Viscosity (nominal, cps)	Number of Scrub Cycles Required To Induce Failure ^a
Α	5,000	496
B	12,000	517
С	20,000	533
D	40,000	552
Ε	75.000	523

(a) Average of four tests to failure on each formulation.

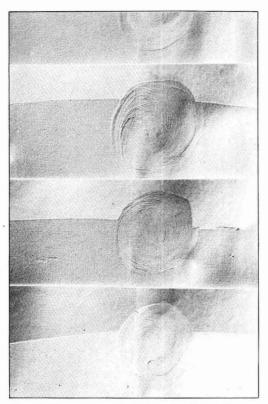


Figure 8—These rubups made with test paints illustrate the color acceptance versus color difference results noted in the initial rubups

paint formulations, are controlled by use of defoamers. For this part of the study the model paint formulation was modified by removal of the defoamer in order to make any foam-inducing effect of thickener levels more readily apparent.

Procedures for foaming tests were similar to those for spatter tests. A 1 ft^2 area was marked on a test paper and roller-painted with the modified formulation, using a 3-inch HHR-3 roller. After nine passes the painted surface was evaluated for foam.

Figure 5 presents a side-by-side comparison of foaming test results for three of the modified test paint formulations. For further comparison, and somewhat as a control, *Figure* 6 shows the foam-test results for a test paint formulation with defoamer at the specified level. (The stipple effect was caused by the roller used.)

Test results with the modified formulations indicate that increasing the thickener concentration, that is, using a thickener of lower molecular weight, increases the foaming tendency of a latex paint. But testing of the model formulations indicates established practices in use of defoamers provide the desired control regardless of thickener levels and molecular weights.

Scrub Resistance

The study included testing of the five model formulations for scrub resistance. The procedure used was the Gardner Scrub Test (ASTM D2486-66T) in which surfaces coated with the test paints, uniformly applied and thoroughly dried, are exposed to a mechanical scrubbing device. The machine provides a consistent pressure and abrasion on each scrubbing cycle and counts the to-and-fro cycles needed to scrub through the paint film.

Results of four scrub test runs on each of the five model formulations are shown in *Table* 4, the counts shown being averages of the four tests. Each of these counts, taken by itself, indicates an acceptable scrub resistance for the specific formulation and thickener loading. Taken as a group for the purpose of this study, the counts show a difference between the lowest and the highest well within the expected degree of variation of the Gardner Scrub Test, indicating that thickener molecular weight has no significant effect on scrub resistance of a latex paint.

Color Acceptance

Among desirable properties and characteristics of a latex paint, color acceptance ranks as one of the most critical. The ideal paint, tinted to a particular color or hue, dries to an identical and uniform color regardless of the method of application—roller, brush, spray. Further, an area coated partly by rolling and partly by brushing the recommended practice in painting an interior wall shows no color differences. Also, color uniformity prevails despite time lags between side-by-side or overlapping brush and roller applications, even if the first application dries before the second begins. Finally, the ideal paint provides color acceptance across the entire spectrum, with whatever color or shade of color that is involved.

The most-used procedure for evaluating color acceptance of a latex paint is known as the rubup test. The design of this test subjects the paint to two different shear forces, seeking to represent the difference in shear generated during rolling versus that occurring during brushing. Leneta 1B chart paper, which is made with the top half of the sheet primed and the lower half unprimed, is used for rubup tests. For the test, a sample of the paint is applied in a 7-mil drawdown covering both primed and unprimed areas. Immediately, using a thumb or a finger, a small area of the drawdown is rubbed, (a) taking in parts of both the primed and unprimed areas, (b) until definite resistance is felt, (c) without going through the paint so that the substrate shows. Laid flat until dry, the chart is inspected and the paint rated for color acceptance on the basis of any apparent color difference between the rubbed spot and the background. Such differences could be caused by either color flocculation or pigment flocculation within the rubbed spot. Figure 7 shows a number of typical rubup charts.

Each of the five versions of the model paint formulation was tested for color acceptance. For each of the thickener levels under comparison, two sets of samples



Figure 9—Typical panel from first series of tests evaluating color acceptance of test paint in side-by-side brush and roller applications

were prepared. Tenneco Color Trend "in-store" color dispersions (12 colors) were used to color one set; Color Corporation of American color dispersions (10 colors), the other. Rubup tests were made with each sample.

Evaluation of the rubups indicated that all five test paint formulations performed similarly. With both brands of color dispersion, in each color tested, the latex paints formulated to have five differing thickener levels had identical ratings as to color acceptance performance. Thus, it was concluded that thickener molecular weight has no effect on the color acceptance properties of a latex paint.

Color Difference

The similarity of ratings on color acceptance performance included similarity in producing color difference in some of the rubups. Specifically: All the test paints showed color differences, slight but still apparent, in rubups of samples tinted with one of the colorants—an orange. Replicating rubups of samples tinted with all colorants of that brand, the orange and others, had the same results: all versions of the model formulation generated color differences in the orange samples, and excellent color acceptance in all others. Some of these rubups are shown in *Figure* 8.

Findings in the rubup tests, viewed in light of the study's objectives, are presented.

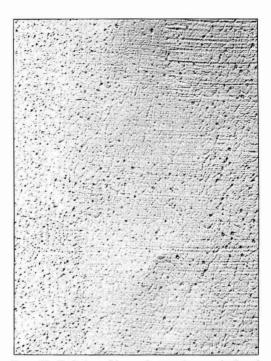


Figure 10—Typical panel from color acceptance evaluation of test paint in brush and roller applications on an undercoated surface

(1) The findings can be used in reaching conclusions as to the effect of molecular weight on thickener performance. Since the pattern of color acceptance-color difference was the same at all thickener levels, it was concluded (as stated earlier) that thickener molecular weight does not affect a latex paint's color acceptance behavior.

(2) The findings raise a question on the validity of the rubup test and, more specifically, a question as to how closely a rubup approximates real-world paint applications. This view developed from the following considerations:

(a) Two distinctly different shear rates are induced in a rubup test, but neither is measured nor is there a practical way to quantify the difference. In contrast, the ranges of shear rates induced by rolling, brushing, and spraying are known.

(b) It is intuitively known that shear induced by a thumb or finger in a rubup test will vary from person to person doing the rub; indeed, it will vary from rubup to rubup done by the same person.

Further Tests of Color Acceptance

At this point it was decided to do further testing on color acceptance. The first group of additional tests involved brush and roller applications using the model

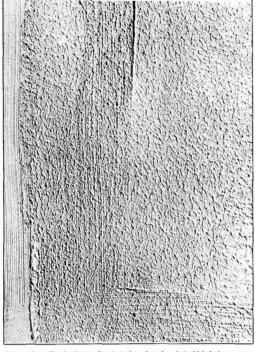


Figure 11—Typical panel painted under simulated high-temperature conditions for evaluation of test paint color acceptance performance

paints tinted with the orange colorant seen to produce color differences in rubups. The applications were made on panels of Kemglow Test Paper (gray stripe), produced by the Sherwin-Williams Co. As produced, the paper is coated and has two vertical white areas separated by a wide central stripe of gray. While each of the three test series in this group consisted of painting similar rectangular panel areas, conditions varied for each series.

Thus, the first series was done at room temperature and with uncoated panels. The perimeter areas of each test panel were painted with a brush and, after an interval, the same paint was rolled, with the inevitable slight overlap, immediately next to the previously brushed areas. From panel to panel in the series, the time interval between brushing and rolling ranged from less than a minute to 20 minutes. A typical panel from the first series is shown in *Figure* 9. When these panels were inspected, none showed any apparent color differences, except, of course, where roller-on-brush overlap had laid two thicknesses of paint film.

The second series, also at room temperature, was done with undercoated panels. The panels were undercoated one day, repainted the next in the same procedure as for the first series—brush applications on perimeter areas, followed at set intervals by roller application into the brush-painted area. *Figure*10 shows one of the panels in the second series of tests.

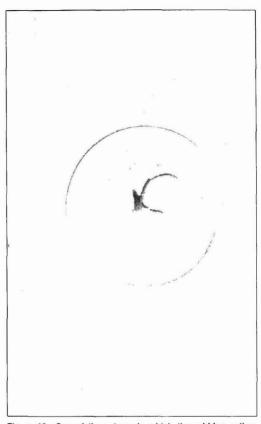


Figure 12—One of the rubups in which the rubbing action, imparted by the ICI Viscometer, induced a 10,000 sec⁻¹ shear rate. The rubup spot is at the center of the area shown

As with the first series of tests, no color differences between brushed and rolled areas were seen when the second series of panels was inspected.

The third test series simulated high-temperature conditions. All the test materials—uncoated panels, paint, brush, and roller—were conditioned to 98° F (36.7°C) and 44% RH. Then the panels were painted in the same brushing and rolling procedure as used previously. The panel shown in *Figure* 11 is typical of the third series test results. Again, none of the panels developed apparent color differences.

This group of tests concluded that, in applications closely resembling real-world practice, side-by-side and overlapping brushed-and-rolled coatings show no color difference. This is in marked contrast to the color differences between drawdowns and rubbed spots in the rubup tests.

High Shear Rate Tests

A second group of tests was done with the model paints tinted with the orange colorant identified with color differences in rubup tests. In these tests, paint samples were subjected to known high shear rates. The goal was to see what shear had to be induced for the paint to yield a detectable color difference.

The ICI Viscometer was used in the first of these tests. For this, a panel was coated with the test paint tinted with the orange colorant, and allowed to dry. Then a drop of the same paint was added to the panel surface, the panel was positioned so that the viscometer could run on the fresh drop for 20 seconds at 1500 rpm, to induce a 10,000 \sec^{-1} shear rate. Inspection of paint drops sheared this way determined that there was no color flocculation, no color difference, at this shear rate. The panel shown in *Figure* 12 is one on which this ICI Viscometer test was run.

To explore the effect of an excessively high shear rate, a Hercules High Shear Instrument was used. This instrument has a cup into which a bob can be introduced and rotated. The space between the cup's inner wall and the outer rim of the bob is limited so that, when the cup contains a test paint sample and the bob is rotated, an extremely high shear rate can be induced. For this study, the sample paint was sheared at a 40,000 sec⁻¹ rate in a 15-second run at about 3800 rpm. Drawdowns made with sheared and unsheared samples are shown side by side in *Figure* 13.

On visual inspection, any color difference between the sheared and unsheared samples is so slight as to be questionable. Thus the sheared paint, despite being subjected to four times the stress calculated to occur during brushing and rolling, developed no significant change in color acceptance properties.

Color Difference Value Study

A test done with the Hunter Tristimulus Colorimeter (Model D-25, Hunter Associates Laboratory, Inc.), using the procedures set by ASTM D2244-79, provided further insight on the rubup test procedure. This instrument simplifies color difference measurements by providing, for the color samples being studied, fixed colorimeter geometry, and fixed illumination. With these factors established by the instrument, the procedure consists of placing each painted sample in the instrument so that it is exposed to, and reflects, light from the contained source. The instrument is then operated to obtain three separate measurements: L—lightness or grayness component of the sample; a—chromaticity of the red-green components; and b—chromaticity of the yellow-blue components.

After two color-differing samples are examined with the Hunterlab instrument, the differences in the L, a, and b measurements are computed and are then used to quantify a "total color difference value," ΔE , by this calculation:

$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$

Such values were calculated for two of the color differences seen in this study: the paint sheared at 40,000 sec⁻¹ in the Hercules High Shear Instrument and the unsheared sample of the same paint (both shown in

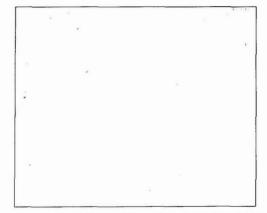


Figure 13—Color difference between these drawdowns of a test paint was determined by the use of the Hunterlab Tristimulus Colorimeter. Drawdown at left was made with paint subjected to a 40,000 sec⁻¹ shear rate by a Hercules High Shear Instrument; drawdown at right was made with unsheared sample of the same test paint

Figure 13) were found to have a total color difference value of 1.54; and the rubup test of the same paint, when the drawdown and rubbed-spot areas were measured, showed a total color difference value of 5.98.

Comparing these values, the logical conclusion is that shear applied in a hand rubup test is likely to greatly exceed shear rates occurring in actual painting applications. The excess is such as to make the rubup test questionable—and, possibly, invalid.

Other Colors, Other Latexes

Color acceptance testing of the model latex paint formulation was completed with a final group of two tests. For the first of these, the ICI Viscometer tests were repeated using all the color dispersions (other than the orange colorant so extensively used already) involved in the study. Neither the Tenneco nor the Color Corporation of America color dispersions showed any color acceptance problems in these tests.

The final consideration was evaluation of color acceptance of the model paint formulation when prepared with a different vinyl acrylic latex. Test quantities were formulated with each of two other latexes—Poly Tex 667 latex and Amsco RES[®] 3077. Tested for color acceptance, these paints showed the same excellent performance as the model formulation.

CONCLUSION REGARDING COLOR ACCEPTANCE

Intensive and wide-ranging exploration of color acceptance was not anticipated when this study began. But it seemed advisable; it proved valuable; and it led to these conclusions: (1) The hand rubup test commonly used for color acceptance evaluation is not necessarily indicative of a paint's color acceptance behavior in actual applications.

(2) Color acceptance tests comparing actual brush and roller applications are practical, uncomplicated, require little time, and can provide dependably representative samples for evaluation.

(3) Latex paints demonstrating excellent color acceptance can be formulated with hydroxypropyl methylcellulose thickeners of any molecular weight.

CONCLUSIONS REGARDING MOLECULAR WEIGHT

The body of this study examined the effects of variations in thickener molecular weight on the properties of latex paints thickened with hydroxypropyl methylcellulose. The study shows such variations do have effects. The specific conclusions are: (1) thickener efficiency increases with increasing molecular weight; (2) spatter increases with increasing molecular weight; (3) foaming attributable to thickener action as a surfactant decreases with increasing molecular weight; any such foaming can be controlled with defoamers; (4) low-shear rheology is independent of molecular weight (as is indicated by Haake Rheology and substantiated by the Leneta Leveling Test); (5) high-shear viscosity decreases with increasing molecular weight; (6) scrub resistance is independent of molecular weight; (7) color acceptance is independent of molecular weight; and (8) known procedures and proven instruments are available for testing to determine how a cellulosic thickener affects a latex paint formulation.

SUMMARY

Cellulosic thickeners of various molecular weight are available for use in formulating latex paints to acceptable viscosity. It is known that the higher the molecular weight, the lower the level of thickener needed to achieve the desired viscosity. However, little has previously been published about how variations in thickener molecular weight affect the properties of the paint. This study was undertaken to determine those effects.

The model paint formulated for the study was an interior flat vinyl acrylic latex. A hydroxypropyl methylcellulose thickener available in five molecular weights, ranging from 5,000 cps to 75,000 cps in nominal viscosity, was used to prepare five versions of the model

formulation. These five versions of the model were then subjected to testing procedures selected to yield valid evaluations on significant properties.

The study found that thickener molecular weight, besides the known effect on thickener efficiency, has a detectable effect on high-shear rheology, spatter resistance, and foaming tendency of a latex paint. It also established that low-shear rheology, scrub resistance, and color acceptance properties of a latex paint are independent of thickener molecular weight.

As an ancillary finding, the study determined that the common hand rubup test is a less-reliable indication of color acceptance behavior of a latex paint than is a test of the paint in small-scale side-by-side applications with brush and roller.

ACKNOWLEDGMENT

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Trademark References

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Amsco RES Union Chemicals Div., Union Oil Co. of Calif.
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Rheological Properties of Alkyd Solutions And Their Relation with Solubility Parameters of Solvents

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The rheological properties of alkyd solutions in poorly and moderately hydrogen bonded solvents have been studied by viscometry. The data at six concentrations and at eight shear rates have been analyzed with the help of standard and proposed equations. The concentration dependence and solubility parameter dependence of several rheological parameters were obtained. This information was used for determining the solubility parameter of the alkyd.

INTRODUCTION

Alkyds form the major part of the total consumption among resins used in the paint industry. Most alkyds are thinned with solvents before use. The solvent-alkyd interactions influence the rheological properties of the resin solution. At concentrations typical to paint formulations, the unpigmented resin solutions show non-Newtonian behavior in viscosity measurements. The rheological properties data of such systems can be used for determining the interaction between resin and solvent. The present work describes the flow behavior of alkyd solutions in poorly and moderately hydrogen bonded solvents at different shear rates.

MATHEMATICAL EXPRESSIONS

Power Law Equation

The following equation represents the relation between shear stress (τ) and shear rate $(\overline{\gamma})^1$

or $r = k \overline{\gamma}^{n}$ $\log r = n \log \overline{\gamma} + \log k$ (1)

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Here, the constants k and n represent consistency and flow behavior of a system, respectively. As the relation is expressed in logarithmic terms; this equation is called the power law equation. This is found to have good fit for practical data; the values of log k and n are used for determining the other rheological parameters.

Martin's Equation

The following equation proposed by Martin² is also chosen for analyzing the results:

 $\log\left(\frac{\eta - \eta_s}{\eta_s \phi}\right) = \log\left[\eta\right] + k_1[\eta]\phi$ $\log\left(\frac{\eta_{sp}}{\phi}\right) = \log\left[\eta\right] + k_1[\eta]\phi$

or

Here, η and η_s are viscosities of solution and solvent. η_{sp} and $[\eta]$ are specific and intrinsic viscosities of the system, ϕ is the volume fraction of solute. The constant k_1 depends on the nature of the solute and solvent system. In other words it represents the interaction between the two.

Concentration Dependence of Log k

The constant k in power law equation is found to be a function concentration. However, in literature no equation is available to represent the relation. The following Erickson's equation³ describes the dependence of relative viscosity η_{γ} on weight fraction W and weight fraction dependent intrinsic viscosity $[\eta]_w$:

$$\frac{\mathbf{W}}{\ln \eta_{\gamma}} = \mathbf{k}_{\mathbf{W}} \cdot \mathbf{W} + \frac{1}{[\eta]_{\mathbf{W}}} \tag{3}$$

Taking Martin's and Erickson's equations into consideration, a relation between log k and W is proposed:

$$\log\left(\frac{k}{W}\right) = k_2 W + C \tag{4}$$

Here, the constants k_2 and C are rheological parameters, where k_2 defines the interaction of alkyd molecules with a

(2)

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Table 1-Solvents

Code No. Used for Solvents	Solvents	Solubility Parameter (cal/cc) ^{1/2}	Viscosity at 26 ± 1°C CP
Joivents	Joivents	(cal/cc)	
Poorly Hydr	ogen Bonded		
Α	. Xylene	8.80^{4}	0.6035
	. White spirit	7.82 ^a	0.7854
с	. Xylene + White spirit (1:1 v/v)	8.315	0.6670
D	. Cyclohexane	8.18 ⁴	0.8812
Moderately I	lydrogen Bonded		
Ε	. Ethyl acetate	9.104	0.4420
	. Butyl cellosolve	9.87	2.7432
	. Methyl ethyl ketone	9.274	0.4011
Н	. Cyclohexanone	9.88 ⁴	1.9932

(a) The solubility parameter of white spirit was determined by Aniline point method.^{7,8}

solvent and C is termed as a function of limiting shear stress. This equation is based on the assumption that the function of shear stress (log τ) is a limiting value when shear rate function (log $\overline{\gamma}$) is zero.

The following simple and empirical expression is also suggested for analyzing log k data from power law equation:

$$\log k = k_3 \log W + B \tag{5}$$

Here, the constants k_3 and B have the significance similar to that of constants in equation (4).

After treating the data on the flow (behavior) properties of alkyd solutions by the above equations, the rheological parameters of alkyd-solvent systems and solvency power of each solvent for the resin are calculated. The findings are also used for determining the solubility parameter of the alkyd.

EXPERIMENTAL

Materials

A commercial alkyd (66% linseed oil-pentaerythritolphthalate) having an acid value less than 10 and solid content $99 \pm 1\%$ was used for the study. The poorly and moderately hydrogen bonded solvents selected for studying rheological properties of alkyd solutions are listed in *Table* 1. All the solvents are of laboratory

Solution No.	Weight of Alkyd (g)	Weight of Solvent (g)	Weight Fraction of Alkyd W
I	250	128.5	0.6605
II	250	257.0	0.4931
III	250	300.85	0.4538
IV	250	352.4	0.4150
V	250	414.4	0.3763
VI	250	487.5	0.3389

reagent grade except white spirit. The fractions of commercial white spirit distilled at 157-167°C are used as solvent code No. B and solvent code No. C is a (1:1) mixture of A and B.

Procedure

The solutions having different weight fractions of alkyd were prepared and stored in a room conditioned at $26 \pm 1^{\circ}$ C. The concentrations of alkyd in a solvent typical to that of paint formulations are given in *Table 2*.

The viscosity properties of alkyd solutions were measured by a Brookfield RVT viscometer. The choice of spindles was so made that a spindle produces observations at rpm ranging from 0.5 to 100 and also covers the maximum number of concentrations. The determinations were first conducted in ascending order of rpm (0.5-100)and then in descending order. The observations under the two conditions indicate that alkyd solutions do not exhibit thixotropic behavior. The possible loss of solvent was controlled so that the concentration of alkyd solution does not change during the experiment.

Calculation of Shear Rate And Shear Stress

In the present work, equations previously reported⁹ were used for calculating shear rate and shear stress. The shear rate $\overline{\gamma}_1$ (sec⁻¹) for spindles No. 2–6 of Brookfield RVT model is:

$$\overline{\dot{\gamma}}_1 = \frac{2\pi \ N \ R_B}{x}$$
(6)

Here, N and R_B are the number of revolutions per second and radius of disc of spindle, respectively, and x is the height of liquid column over the disc.

The shear rate $\overline{\dot{\gamma}}_2$ (sec⁻¹) of RVT spindle No. 1 is:

$$\overline{\dot{\gamma}}_2 = \frac{2\pi N R_C R_B}{R_C^2 - R_B^2}$$
(7)

Here, R_C is the radius of the container used for test liquid.¹⁰ The dimensions of spindles, i.e., R_B and x are supplied by the manufacturers.¹⁰

The shear stress τ (dynes/cm²) is calculated by the following equation:⁹

$$\tau = \frac{7187\text{D}}{100 \times 2\pi \ \text{R}_{\text{B}}^3} \tag{8}$$

The symbol D is the dial reading and 7187 dyne.cm is full scale torque of RVT viscometer. Several workers¹¹⁻¹³ have also used other equations

Several workers¹¹⁻¹³ have also used other equations apart from the above equations for calculating shear rate and shear stress data from measurements made by using the Brookfield RVT viscometer.

RESULTS AND DISCUSSION

The viscosity data of alkyd solutions at different shear rates have been analyzed by using the Power Law equation, Martin's equation, and two other proposed equations.



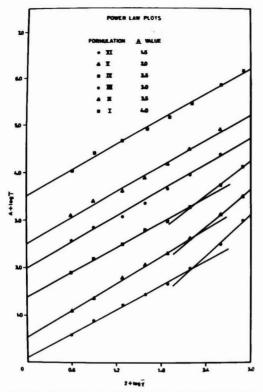


Figure 1—Shear stress vs shear rate for alkyd solutions in xylene. 'A' is an arbitrary constant used to displace lines vertically for clarity. RVT spindle used for formulation I is No. 3 and for II-VI No. 1

Power Law Equation

The doubly logarithmic data of shear stress versus shear rate for alkyd solutions in poorly and moderately hydrogen bonded solvents are plotted in *Figures* 1–4. For the sake of having better illustration of plots, the crowding of lines corresponding to individual weight fraction of alkyd has been avoided by using an arbitrary numeral represented by symbol A in each figure.

In Figures 1 and 2, the first three plots of $\log \tau$ vs. $\log \overline{\gamma}$ for alkyd solutions in poorly hydrogen bonded solvents are linear at all the shear rates. When concentrations of alkyd solutions are at weight fractions 0.415 and below, the data are better illustrated by two linear plots: one passing through points at shear rates corresponding to 0.5-20 rpm; the other passing through points corresponding to 20-100 rpm. Both plots intersect at a point very close to 0 on $\log \overline{\gamma}$ axis (i.e., $2 + \log \overline{\gamma}$). In the case of moderately hydrogen bonded solvents, this feature has been observed in alkyd solutions at even higher concentrations (*Figures* 3 and 4).

The probable reason for the relatively high increase in shear stress is that in dilute solutions the molecules may aggregate around the disc of a fast moving spindle due to

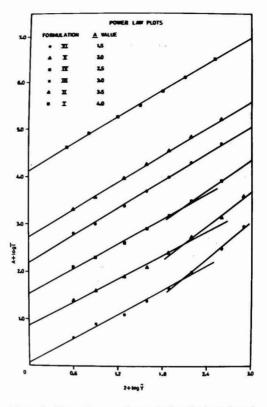


Figure 2—Shear stress vs shear rate for alkyd solutions in cyclohexane. 'A' is same as in *Figure* 1. RVT spindle used for formulation I is No. 4 and for II-VI No. 1

centrifugal forces. This may produce shear stress higher than that expected at shear rates corresponding to rpms of 20 and above. Alkyd solutions in moderately hydrogen bonded solvents greatly exhibit this effect than those in poorly hydrogen bonded solvents because of greater interaction due to structural similarity among resin and solvents. The increase in shear stress at high shear rates could also be attributed to the use of spindle No. I in experiments conducted at low concentrations of alkyd solutions. The narrow clearance between spindle cylinder and the container may contribute to this effect especially at high speeds.

Slopes and Intercepts

The values of slope 'n' and intercept 'log k' obtained from the plots (log $\tau = n \log \overline{\gamma} + \log k$) for a solution indicate the nature of its flow behavior at different shear rates and the consistency index, respectively. In low shear rate region corresponding to speeds of spindle up to 20 rpm, the values of n are less than one, i.e., 0.75–0.95. This shows that the flow behavior of solutions is pseudoplastic up to 20 rpm. On the other hand, at shear rates corresponding to 20–100 rpm, the values of n in the range

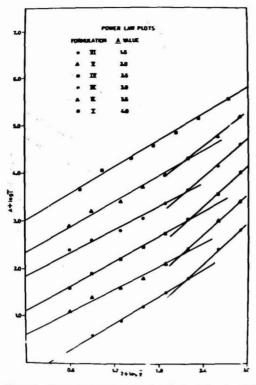


Figure 3—Shear stress vs shear rate for alkyd solutions in ethyl acetate. 'A' is same as in Figure 1. RVT spindle used for formulation I is No. 2 and for II-VI No. 1

of 1-1.5 indicate that the flow behavior of the solution is shifted from a creeping region to laminar boundary layer conditions. The deviation in values of rheological parameter 'n' suggests that flow behavior of alkyd solutions at low shear rates is like that of pseudoplastic systems and is of an apparent dilatant type at higher shear rates.^{1,14}

The constant 'n' does not show any relation with the concentration of alkyd and the nature of solvent in which the resin is dissolved. Whereas, the values of log k show a distinct relation with the concentration of alkyd in solution and also with the nature of solvent. The values of log k can be used for quantitative assessment of the solvency power of solvents for the alkyd.

Log k and Concentration In Terms of Weight Fraction W

The linear plots of log (k/W) versus W show that the proposed equation $[log(k/W) = k_2W+C]$ holds good in the treatment of rheological properties data for alkyd solutions. In plotting log (k/W) versus W in *Figure 5*, only the data obtained by using RVT spindle No. 1 is used so that the spindle constant is the same in all calculations. This choice eliminates the effect of any possible variation in shear conditions due to change of spindle constant. The slope 'k₂' and intercept 'C' of the plots are rheological

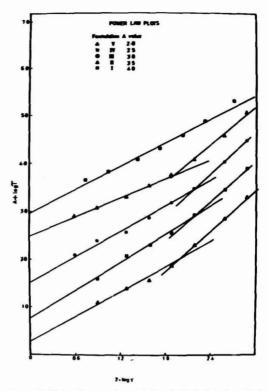


Figure 4—Shear stress vs shear rate for alkyd solutions in methyl ethyl ketone. 'A' is same as in *Figure* 1. RVT spindle used for formulation I is No. 2 and for II-V No. 1

parameters corresponding to solvent-alkyd interaction and solvency power of the solvent, respectively. These rheological parameters are in accordance with those of Martin's equation (2) where the slope represents the interaction between solute and solvent and the intercept is considered to be the function of solvency power.¹⁵

The Constant k Function of W

The plots of log k versus log W for alkyd solutions in individual solvents are linear and indicate that the constant k and W can also be expressed by a direct logarithmic relation (*Figure 6*). The constants k_3 and B of the equation (log $k = k_3 \log W + B$) also provide information similar to that obtained from the constants in equation (4).

Treatment of Results By Using Martin's Equation

Martin's equation has good application to data collected at one speed of immersion body (spindle). For convenience and simplicity, only data at two shear rates, i.e., corresponding to five and 50 rpm are analyzed by using Martin's equation; these are plotted in *Figures* 7-10.

The data at five and 50 rpm, indicate that intrinsic viscosity of each system increases with shear rate. In the

RHEOLOGICAL PROPERTIES OF ALKYD SOLUTIONS

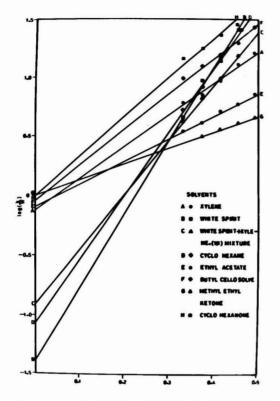


Figure 5—Log (k/W) vs weight fraction 'W' of alkyd (proposed equation (4)). Data obtained with RVT spindle No. 1

case of good solvents for alkyd, the increase in $[\eta]$ at higher shear rates is comparatively much lower than that with a relatively poor solvent. For example, when xylene is used as solvent the $[\eta]$ of solution at 50 rpm is 1.35 times that at 5 rpm (33.1 at 5 rpm and 44.6 at 50 rpm). Whereas, in the case of white spirit as solvent, it is five times, i.e., 1.14 at 5 rpm and 5.62 at 50 rpm. It can be inferred from these observations that in good solvent for alkyd stirring does not matter but it does affect the flow of alkyd in a solvent having relatively poor solvency for it.

Correlation of Results With Solubility Parameter of Solvents

RHEOLOGICAL PARAMETER k₂: The effect of solvent on rheological properties of alkyd solutions is better illustrated by plotting the data of slope k₂ and intercept 'C', obtained from plots in *Figure* 5, as function of solubility parameter δ of solvents. The plot of k₂ as function of δ passes through a minimum (*Figure* 11), indicating that solvents having their δ values in the range corresponding to the minimum of the plot would have good compatability with alkyd. In other words, a polymer dissolved in such solvents would extend its molecular segments more freely and experience minimum resistance in flow. Therefore, the solubility parameter of alkyd can be equated to δ values of solvents falling in the

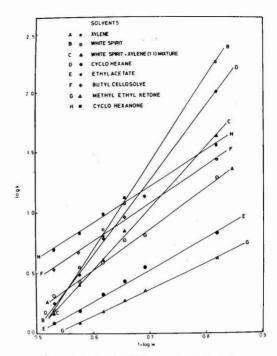


Figure 6-log k vs low W (proposed equation (5))

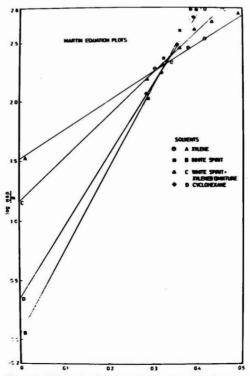


Figure 7—Volume fraction dependence of specific viscosity of alkyd in poorly hydrogen bonded solvents at 5 rpm

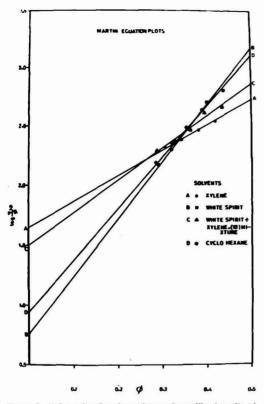


Figure 8—Volume fraction dependence of specific viscosity of alkyd in poorly hydrogen bonded solvents at 50 rpm

range of the minimum on the plot, i.e., 9.2–9.6, the midpoint of which is 9.4 $(cal/cc)^{\frac{1}{2}}$.

King¹⁶ in a series of publications has interpreted his findings in terms of solvency power of petroleum solvents for alkyds making use of viscosity and solubility parameter data. He did not consider his results in terms of interaction or rheological parameters. Whereas, Erickson³ correlated the interaction variables with δ of solvents and found that the plot of the data passed through a minimum.

The plot of the constant k_2 as function of δ of solvents can be used for determining solubility parameter of a polymeric material.

LIMITING SHEAR STRESS PLOT: The plot of intercept 'C' versus δ of solvents passes through a maximum (*Figure* 12). The significance of limiting shear stress is similar to that of the limiting (intrinsic) viscosity of polymers. It is reported that intrinsic viscosity of a system is maximum when the solute and solvent have maximum compatability and polymer segments are fully stretched in the solution.^{15,17} This situation occurs when δ values of solvent and polymer are fairly close and their physical parameters also complement each other. Hence, from the maximum of the plot in *Figure* 12, the solubility

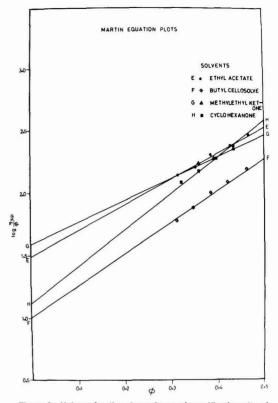


Figure 9—Volume fraction dependence of specific viscosity of alkyd in moderately hydrogen bonded solvents at 5 rpm

parameter of alkyd is equated to be 9.5 $(cal/cc)^{1/2}$. This indicates that the limiting shear stress is proportional to the solvency power of a solvent for the alkyd and can be used for determining its solubility parameter.

RHEOLOGICAL PARAMETER k₃: The plot values of the slope k₃ of equation (5), as function of solubility parameter of solvents, pass through a minimum (*Figure* 13). The value of the solubility parameter for the alkyd derived from this plot is 9.4 (cal/cc)^{1/2}.

Martin's Equation

RHEOLOGICAL PARAMETER k_1 : The plots of the slope $k_1[\eta]$ as function of δ of solvents at five and 50 rpm pass through minima (*Figure* 14). The plots at five and 50 rpm do not show any significant effects of the speed of rotation of spindle on the results. In *Figure* 15, the plots of k_1 versus δ of solvents at five and 50 rpm differ widely in magnitude from each other. This is because there is a vast difference in the values of multiplying factor $[\eta]$ at five and 50 rpm. The solubility parameter values of alkyd obtained from the minima of the plots in *Figure* 14 is 9.3 and plots in *Figure* 15 are 9.2 at five rpm and 9.3 (cal/cc)^{1/2} at 50 rpm.



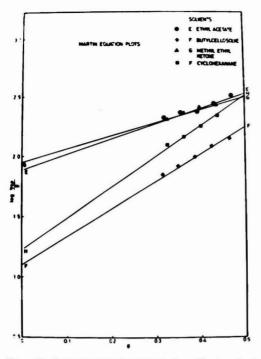


Figure 10-Volume fraction dependence of specific viscosity of alkyd in moderately hydrogen bonded solvents at 50 rpm

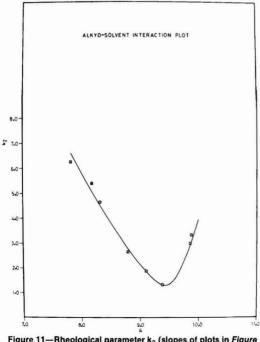


Figure 11—Rheological parameter k_2 (slopes of plots in Figure 5) vs solubility parameter of solvents

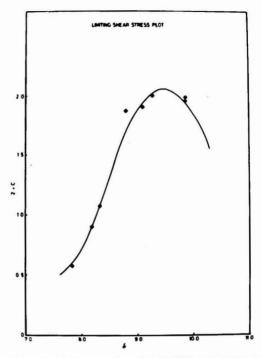
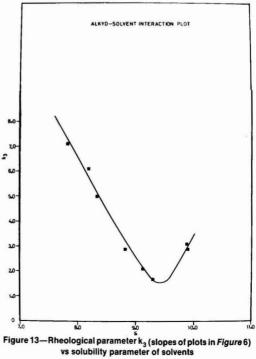


Figure 12-Limiting shear stress function 'C' (Intercepts of plots in Figure 5) vs solubility parameter of solvents



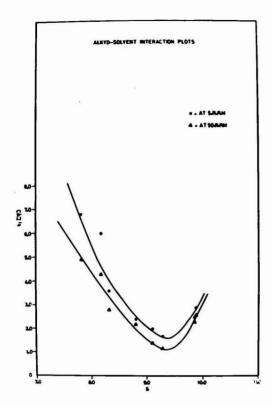


Figure 14—Rheological parameter $k_1[\eta]$ (slopes of plots in *Figures* 7–10) vs solubility parameter of solvents

INTRINSIC VISCOSITY DATA: Usually, the rate of flow of dilute polymer solution through a capillary is used for calculating intrinsic viscosity. Here, the data from Brook-field viscometer measurements are used for determining intrinsic viscosity of alkyd solutions. The intrinsic viscosity data at five and 50 rpm are plotted as a function of δ of solvents and the plots pass through maxima (*Figure* 16). The two plots being vertically far apart from each other indicate that intrinsic viscosity of alkyd solutions increase with speed of the spindle, whereas, the compatability of alkyd with solvent is not affected. The values of solubility parameter of alkyd obtained from the maxima of the plots are 9.2 at five rpm and 9.3 (cal/cc)^{1/2} at 50 rpm.

APPARENT VISCOSITY PLOT: In Figure 17, the apparent viscosity of alkyd solutions of weight fraction 0.6605 at five and 50 rpm are plotted as a function of the solubility parameter of solvents. This plot passes through a minimum and is similar to the plots of rheological parameters k_2 , k_3 , k_1 , and $k_1 [\eta]$ versus δ (Figures 11, 13–15). The value of δ for alkyd determined from it is 9.35 (cal/cc)¹⁶. The points corresponding to the solvents white spirit and cyclohexane are a little away from it. The apparent viscosity plot cannot be considered for deriving

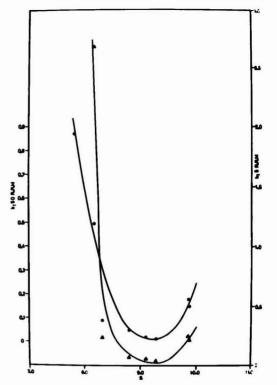


Figure 15—Rheological parameter k_1 (calculated from slopes in Figures 7–10) vs solubility parameter of solvents. Legend: $A = K_1$ at 5 rpm; $\Phi = K_1$ at 50 rpm

 δ value of a polymer as it does not include other physical parameters involved in calculations.

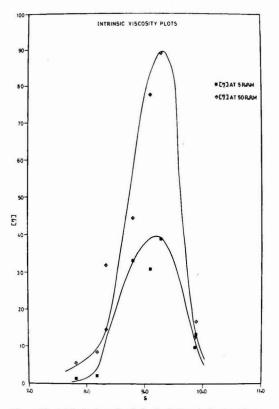
Solubility Parameter of Alkyd

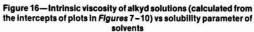
Earlier it was reported that viscosity data can be used for finding the solubility parameters of polymers or polymeric materials.^{17,18} The values of the solubility parameter of alkyd obtained from various plots do not differ much from those reported by others (*Table 3*). These findings show that the solubility parameters of alkyds and polymeric materials can be determined by using rheological parameters obtained from experiments conducted with the Brookfield RVT viscometer.

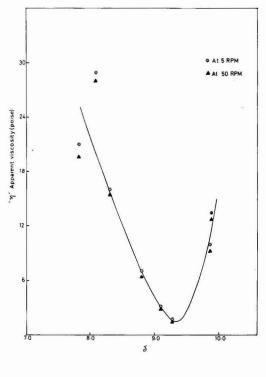
SUMMARY

(1) The findings on viscosity properties of alkyd solutions provide quantitative information about rheological parameters and solvency power of different solvents.

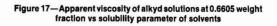
(2) The intercept 'log k' of the Power Law equation is found to be dependent on the concentration of alkyd in solutions and the solubility parameter of solvents.







Solubility parameter of solvents



Type of Alkyd	Method of Determination	δ of Alkyd (cal/cc) ^½	
δ-Values Obtained from Present W	/ork		
66% Linseed-pentaerythritol- phthalate	(1) Rheðlogical parameter k2 plot - Figure 11	9.4	
66% Linseed-pentaerythritol- phthalate	(2) Limiting shear stress plot - Figure 12	9.5	
66% Linseed-pentaerythritol- phthalate	(3) Rheological parameter k ₃ plot - Figure 13	9.4	
66% Linseed-pentaerythritol- phthalate	(4) Rheological parameter $k_1 [\eta]$ plot - Figure 14	9.3	
66% Linseed-pentaerythritol- phthalate	(5) Rheological parameter k ₁ plot - Figure 15	9.2 (at 5 rpm) 9.3 (at 50 rpm)	
66% Linseed-pentaerythritol- phthalate	(6) Intrinsic viscosity plot - Figure 16	9.2 (at 5 rpm) 9.3 (at 50 rpm)	
66% Linseed-pentaerythritol- phthalate	(7) Apparent viscosity plot - Figure 17	9.35	
δ-Values Reported by Others			
Long oil alkyd (Plexal P-65)	(1) Solubility diagram ⁴	9.4	
Long oil linseed-glycerol- phthalate	(2) Intrinsic viscosity ¹⁷	8.91	
66% Linseed-pentaerythritol- phthalate	(3) Intrinsic viscosity ¹⁸	9.4	
Long oil alkyd	(4) Solvent evaporation ¹⁹	9.4	
Linseed-glycerol-phthalate	(5) Group contribution technique ²⁰	9.39 9.27	

Table 3—Solubility Parameters of Alkyds

(3) The change in flow behavior of a system at different speeds of stirring can be predicted by the change in slopes of plots of the Power Law equation.

(4) The equations proposed for direct relation between log k and weight fraction W of alkyd are found to hold good.

(5) The analysis of data by Martin's equation provides useful information about shear dependent intrinsic viscosity.

(6) The rheological properties data for alkyd solutions can be used for determining the solubility parameter of alkyds.

ACKNOWLEDGMENT

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Surface Coloring of Glassy Polymers By Solvent Diffusion

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and

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The technique of surface coloring of polymeric glassy materials based on the swelling properties of the organic solvents is suitable for solving particular problems in decoration of plastic objects.

The depth of dye penetration and the concentration in the surface layer, and hence the coloring effect, are functions of diffusion conditions. Therefore, a knowledge of the solvent diffusion process is preliminary to the control of the surface coloring process.

In this paper a comparison between diffusion kinetics of methanol in polymethylmethacrylate samples, obtained by different processing technologies, is reported.

INTRODUCTION

The diffusion of solvents in glassy polymers cannot be described generally in terms of Fick's law.¹ If the polymer structure rearranges itself to accommodate the penetrant molecules at a rate comparable to the penetrant mobility, deviations from the limits defined by Fick's law are observed. Alfrey, et al.² have defined a limiting transport process wherein the penetrant mobility is much greater than structure rearrangement rates and therefore, do not play any part in the rate determining mechanism. Such a diffusion behavior was named as *Case II*, that is, a second limiting transport behavior in contrast to the more familiar Fickian diffusion, termed *Case I* transport.

The relative contributions of penetrant mobility and polymer relaxation are suggested by the value of the exponent, n, in the relationship between the amount of penetrant agent absorbed per unit area, M, and time, t, given by equation:

 $\mathbf{M} = \mathbf{K} \, \mathbf{t}^{\mathrm{n}} \tag{1}$

where K and n are constants. The exponent, n, varies between unity, for a purely relaxation controlled process (Case II), to $\frac{1}{2}$ for a penetrant mobility controlled process (Case I).

For purely Case II penetration, the K constant is directly the penetration velocity of the sharp advancing boundary separating the inner unperturbed core from the outer swollen gel. Moreover, in such a case, behind the advancing front, the concentration of the penetrant in the polymer is homogeneous.

For any polymer-penetrant system the type of transport behavior changes with the diffusion conditions. Particularly relevant is the increase of the penetrant mobility contribution when the experimental temperature is increased: the rate of the otherwise controlling relaxations increases enormously and at elevated temperature (approaching the polymer glass transition temperature) the relaxations at the swelling boundary are no longer slow compared with the diffusive transport of penetrant.

A solute dissolved in the penetrant agent is transported by it, if the diffusion rate of the solute in the swollen polymer is comparable to the penetrant diffusion rate in the glassy unperturbed polymer. Generally, such a condition is more easily reached in the relaxation controlled diffusion because the slow relaxation process allows a partial or total equilibration of the solute concentration behind the diffusion boundary.

If a non-volatile solute is carried by a volatile solvent, polymeric glassy materials modified in an outer shell are manufactured, once the solvent driver is removed.

Surface coloring of polymeric glassy materials using this technique³ is particularly useful as a solution to coloring problems in window decoration and in general, in plastic object ornament (i.e., polychrome goods, sheets

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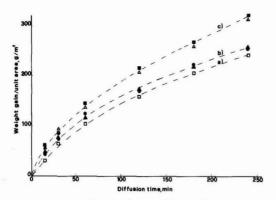


Figure 1—Sorption kinetics of methanol in PMMA at 60° C for: □ as received cast specimens; ● dried cast specimens; ▲ cast specimens annealed at 130° C; ■ dried extruded specimens; and △extruded specimens annealed at 130° C

colored on only one side, easily-made and stable pictures in plastics using suitable masks, etc.).

The depth of dye penetration and the concentration in the surface layer and hence the coloring effect are fixed by diffusion conditions (temperature, time, concentration in the diffusion bath). A deep knowledge of the solvent diffusion process is, therefore, preliminary to a control of the surface coloring, or more generally surface modification, process.

Diffusion kinetics for PMMA-alcohols systems⁴⁻⁵ and in particular for PMMA-methanol system⁷⁻⁹ were recently described over the temperature range from room temperature to the boiling point of the penetrant agent. These studies have pointed out the change in the transport behavior from being relaxation controlled at the lowest temperatures to nearly approaching Fickian behavior at the highest temperatures. In this paper a comparison between diffusion kinetics in polymer samples obtained by different processing technologies is made.

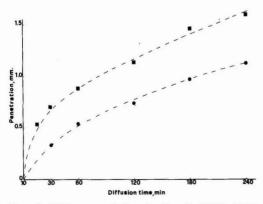


Figure 2—Thickness penetration of methanol in PMMA at 60°C for: ● dried cast specimens; and ■ dried extruded specimens

In particular, the diffusion behavior of methanol in commercial cast and extruded sheets is compared. Cast and extruded samples are different in molecular weight, in thermal history, and in amount and type of molecular orientation. For some solvent-polymer systems it has been pointed out that all these factors (molecular weight,¹⁰ thermal history,^{8,11,12} macroscopic and molecular orientation^{13,14}) have an influence on the diffusion kinetics.

To better understand the contribution of such factors in determining the observed differences in diffusional behavior in cast and extruded sheets, we have also investigated the solvent diffusion in samples annealed at a temperature higher than their glass transition temperature, by which means the preceding thermo-mechanical history was completely erased.

EXPERIMENTAL

Commercial polymethylmethacrylate cast sheets and extruded sheets were obtained from Montepolimeri S.p.A. Reagent grade methanol was obtained from Carlo Erba S.p.A. and was used without further purification in the penetration and sorption experiments.

Multiple 100 mm \times 10 mm \times 3 mm PMMA samples were immersed in methanol, maintained at 60°C (\pm 0.1°C). The individual specimens were suspended in the solvent to avoid contact with the walls of the confining vessel.

The cross section was viewed with a "Stereoscan" reflected-light microscope at $6.3 \times$ magnification.

A sharp boundary, easily detectable with the optical microscope, separated the apparently unperturbed core from the morphologically altered swollen outer layers.

The thickness of penetration was determined microscopically with a calibrated, Filar micrometer eyepiece.

All the data reported are an average of at least four measurements.

RESULTS

In Figure 1 the weight gain per unit area of PMMA in methanol at $T = 60^{\circ}$ C as a function of time has been reported. This temperature has been chosen for its technological relevance in coloring such polymeric material.³

Curve a in *Figure* I refers to cast specimens as received, while curve b refers to cast specimens previously desiccated at $T = 70^{\circ}$ C. The difference between curves a and b is due to the amount of water contained in the samples as received. In fact, taking into account this value, which is 0.7 to 0.8% by weight, curves a and b superimpose.

Curve c refers to extruded dry specimens. It can be noticed that in the extruded specimens, the diffusion is faster than in the cast ones, moreover, the exponent n of equation (1) is slightly higher (n = 0.62 for cast specimens and n = 0.66 for extruded specimens).

Similar conclusions are also reached when analyzing the thickness of penetration (Figure 2) and the increase in

SURFACE COLORING OF GLASSY POLYMERS

volume per unit area (Figure 3a) for specimens exposed to methanol.

In Figure 3b the volume gain over weight gain ratio is reported for all the materials studied. The results indicate an independence of this ratio on the diffusion time but a different value between cast and extruded specimens. In fact, in the first case this ratio is equal to 0.82 g/cm^3 and in the second case is equal to 1.00 g/cm^3 , while the specific volume of methanol is 1.25 g/cm^3 .

In order to discriminate if the different diffusional behavior in cast and extruded specimens is due to the largely different molecular weight or on the contrary to the previous thermomechanical history, the diffusion kinetics of methanol in specimens annealed above their glass transition temperature has been studied.

Hopfenberg, et al.^{11,12} have shown a relevant dependence of the diffusion kinetics of solvents in glassy polymers on their previous thermal history. These authors have pointed out that such a dependence is reduced with increasing diffusion temperature and penetrant concentration, and is completely absent for rubbery polymers. Analogous results were obtained by Thomas and Windle⁸ for the diffusion of methanol in PMMA. They have pointed out that the diffusion kinetics at 24°C is strongly dependent on the previous cooling rate across the glass transition temperature, experienced by the polymeric samples: an increase in cooling rate of three decades causes the diffusion rate to increase by nearly a factor of three. They have also shown that for such a polymer-solvent system the sorption rate at 62°C is practically independent of the previous cooling rate across the glass transition temperature. In our case, experiments performed on annealed PMMA samples have indicated the presence of higher degree of internal stresses in cast specimens which show an increase in thickness of 3.7%, while the thickness of extruded specimens increases by 2.3%, with a corresponding reduction in specimen area in both cases.

Weight gain per unit area for annealed cast and extruded specimens are also reported in *Figure* 1. The corresponding experimental points are satisfactorily correlated by curves b and c previously described. However, the volume gain per unit area, in contrast with the weight gain per unit area, is strongly dependent on the previous sample history. In fact, the experimental curves for annealed samples lie above the corresponding experimental curves for the as received samples, both for cast and extruded specimens (*Figure* 3a).

Moreover, the results reported in *Figure* 3b indicate that the volume gain in the sorption of one gram of penetrant does not change for annealed cast or extruded samples, and is equal to 1.23 g/cm^3 . In other words, the volume gain in the sorption of one gram of methanol is practically equal to the specific volume of this solvent and independent of the molecular weight of the polymer once all the frozen internal stresses are removed. On the other hand, the value of this quantity for unannealed samples deviates from the specific volume of methanol for cast specimens in which, as discussed before, the frozen internal stresses are higher.

Hence, in accordance with Thomas and Windle,⁸ there is no variation in the sorption kinetics by modifying the

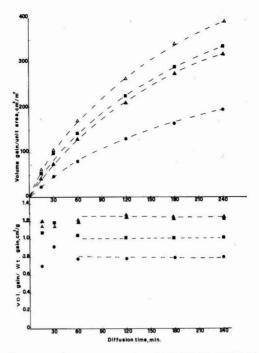


Figure 3—(a) Swelling kinetics of methanol in PMMA at 60° C. (b) Ratio volume gain over weight gain vs. diffusion time. □ as received cast specimens; ● dried cast specimens; ▲ cast specimens annealed at 130° C; ■ dried extruded specimens; and △ extruded specimens annealed at 130° C

thermomechanical history of the sample. Therefore, it is possible to conclude that the different sorption kinetics obtained in the case of cast and extruded specimens (unchanged after removal of internal stresses) can be due to the different molecular weights. The swelling kinetics are, on the contrary, influenced (in particularly reduced) when internal frozen stresses are present in the specimens, also for the higher diffusion temperature selected.

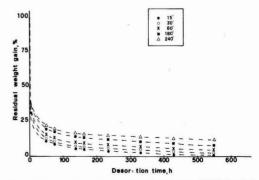


Figure 4—Desorption kinetics of methanol from PMMA, in air at 60° C. Different points indicate different diffusion times. ● 15 min; ○ 30 min; × 60 min; ■ 120 min; and △ 240 min

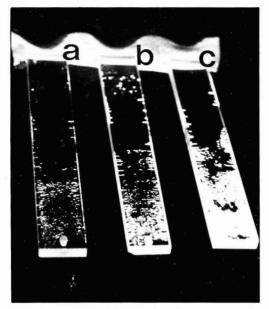


Figure 5—Dependence of the treated sample appearance on the desorption conditions. Samples with equal diffusion treatment but desiccated in an oven at 60° C for 2 hours (a), 70 hours (b), 500 hours (c)

METHANOL DESORPTION AND TREATED SAMPLES APPEARANCE

For surface coloring of plastics, the appearance of treated samples after removal of the swelling agent is critically important.

In Figure 4 the desorption curves in air at 60°C, relative to PMMA cast specimens previously immersed

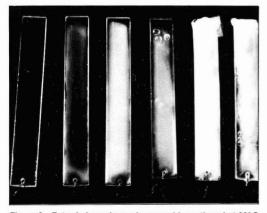


Figure 6—Extruded specimens immersed in methanol at 60° C and de-sicated in air at 60° C for 500 hours. Starting from the left side of the figure the diffusion time increase: 15, 30, 60, 120, 180, 240 min

in methanol, are reported as the residual weight increase in respect to the dry samples vs. desorption time. It can be observed that the initially high desorption rate of solvent, probably from the outer shells, becomes slower and slower when the diffusion time increases. As the solvent desorbs, the drier polymer on the surface constitutes a barrier for further diffusion of the methanol that is in lower concentrations. A residual alcohol content, also for long desorption times, is undetectable on specimen inspection but is confirmed by interferometric analyses, which allows the determination of the solvent concentration profiles inside the modified outer layer.

If the diffusion time is high enough (starting from 60 min) the cast specimens, whose desorption is described in *Figure* 4, present appearance and transparence practically equal to that of virgin samples. For short diffusion times the formation of crazing upon desorption is observed. Such a phenomenon was observed also by Thomas and Windle⁹ and was attributed to stresses developed on desorption in partially swollen samples. They observed that in these cases the birefringence in the swollen region during desorption increases greatly in magnitude, indicating the presence of biaxial tensile stresses in the surface layers.

The magnitude of these tensile stresses developed during the desorption is dependent on the degree of swelling (i.e., diffusion temperature) and on the thickness of the swollen layer.

The formation of crazing (and hence, the amount of internal stresses developed on desorption), in our case, is strongly dependent on the desorption conditions. High desorption rates (i.e., high desorption temperatures) greatly contribute to the crazing. In particular, samples with equal diffusion treatment but desiccated in an oven at 60° C for 2 hr (a), 70 hr (b), 500 hr (c), are compared in *Figure 5*.

For the extruded specimens at diffusion times higher than 10 minutes, the formation of a second sharp boundary separating a white external layer and a transparent modified layer have been observed inside the modified layer. The distance from the surface of this second boundary varies with diffusion time between 100 and 250 μ m.

Moreover, the methanol desorption at 60° C from extruded samples gives rise to the formation of large bubbles and related sample deformations (*Figure* 6).

On the contrary, if the methanol desorption is carried out at room temperature, it is possible to obtain cast and extruded colored samples without craze formation after two years from the preparation and also for short diffusion times (*Figure* 7).

In principle, it is possible to use any kind of dye soluble in the polymer and in the solvent used as driver.

Figure 7 demonstrates that different color shades are obtained by varying the immersion times in the diffusion baths; for extruded samples opaque colors are also achievable.

The thermomechanical behavior of the treated samples was characterized by the "heat distortion temperature" method (HDT, ASTM D.648). For the diffusion times

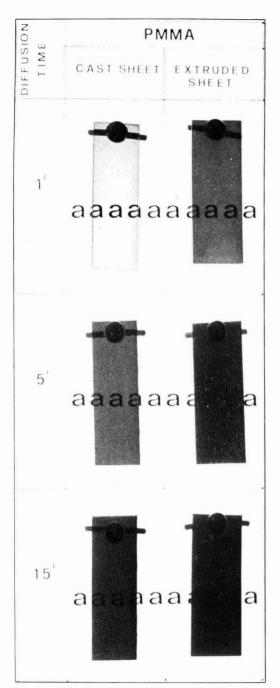


Figure 7—Case and extruded colored specimens. Different color shades are obtained varying the immersion times in the diffusion baths; for extruded samples also dull colors are achievable

used no sensible variation of the "heat distortion temperature" value for PMMA samples was evidenced.

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C. PAOLONE is working with Montedison since 1962. His interests in research have been in the fields of intermediate products for polyamides and of diffusion in polymers. He is presently engaged in new applications for plastics.

Analogously, the impact resistance of treated and untreated samples was indistinguishable.

CONCLUSIONS

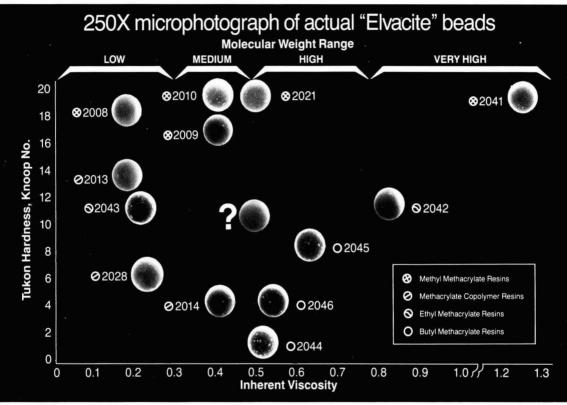
The kinetics of diffusion of methanol in PMMA at 60°C is different for cast and extruded polymeric samples. Such a difference can be attributed to the different molecular weights and not to the different thermomechanical history. However, the ratio volume gain over weight gain, after solvent diffusion, seems scarcely dependent on the polymer molecular weight, but is clearly dependent on the previous thermomechanical history.

Good results are achievable by methanol diffusion in surface coloring of PMMA. Using proper desiccation conditions, colored cast and extruded samples without defects are obtained.

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Study on the Workability of Paints

Forces Exerted During Brushing And the Rheological Characteristics of Paints

Yasuyuki Kuge Institute of Vocational Training*

The brush drag and the pressure applied on the brush were quantitatively measured under various brushing conditions using an apparatus which has a reciprocator, two-dimensional dynamometer, and brush. The theoretical equations to correlate the drag and pressure with rheological characteristics of paints were derived and the validity was tested with experiment. Equations for the drag, Fh, and pressure, Fv, were found to agree with the experimental results.

INTRODUCTION

There have been many works on brushability of paints, in which the relationship between brushability and rheological charactistics of paints has been discussed.¹⁻⁷

When the painter moves the brush on the substrate, both horizontal and vertical forces to the direction of motion are applied on the brush.

The horizontal force, Fh, which is equivalent to the brush drag, can be considered to be shearing stress along the plane of shear, necessary to move the brush at the rate of application. Thus, Fh of Newtonian fluid can be given by:

$$Fh = A S$$
$$= A \eta D$$
$$= \frac{A \eta v}{h}$$
(1)

where A is the contact area of the brush, S is the shearing stress, η is the viscosity of the fluid, D is the rate of shear, v is the brush speed, and h is the film thickness.

Mell and Finn⁴ have suggested that the upward thrust of the paint due to the "wedge effect" must equal the downward pressure of the bristles, so that the film thickness at which this equilibrium occurs will be dependent upon the rate of shear and viscosity of the paint at this shear rate, giving the equation as follows:

$$P = a \eta D / h$$
$$= \frac{a \eta v}{h^2}$$
(2)

where P is the normal pressure and a is a correcting coefficient. Since P is given as the force per unit width of the brush, the vertical force, Fv, of Newtonian fluid can be given by:

$$Fv = \frac{a \ B \ \eta \ v}{h^2} \tag{3}$$

where B is the brush width.

If these equations were in good agreement with actual brush application, they would allow the brushability to be estimated quantitatively in terms of the forces applied on the brush during application. But there have not been any experimental works to confirm them by measuring these forces.

A two-dimensional dynamometer was developed to measure the horizontal and vertical forces applied on a brush by modifying tool dynamometers which have been used in mechanical engineering for a long time.⁸⁻⁹

The forces were measured under various brushing conditions, and a correlation with the theoretically derived equations was discussed.¹⁰⁻¹¹

EXPERIMENTAL

Paints and Substrate

Polyalkyleneglycol derivatives were used since the viscosity and the thickness are not much influenced by solvent evaporation in this system. The physical constants of samples used are given in *Table 1*.

^{*} Aihara 1960, Sagamihara-shi, Kanagawa, Japan.

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Table 1—Physical Constants of Polyalkyleneglycol Derivatives Used					
Sample	Α	в	С		
Density (g/cm ³) 15°C	0.988	0.996	0.998		
Viscosity (poise) 15°C		4.30	8.13		

A polyester sheet $(270 \times 160 \times 0.2 \text{ mm})$ scuffed with a 1000 # sanding paper was used as the substrate for brushing.

Apparatus

The experimental apparatus has a reciprocator, twodimensional dynamometer, and brush (see *Figure* 1).

The reciprocator stroke length is 60 cm and stroke speed can be varied from 0 to 71.6 cm/sec. The reciprocator has an arm, which has a brush holder at the end; the brush holder can be adjusted vertically.

The dynamometer developed is shown in *Figure 2*. Horizontal and vertical forces can be measured with no cross-interference by connecting gauges into two independent Wheatstone bridges. The signal from the dynamometer is fed to a strain meter and recorder.

Four pig bristle brushes of various sizes were used as shown in *Table 2*.

Experimental

Brushing conditions are shown in *Table* 3. All experiments were carried out at room temperature $(15 \pm 1^{\circ}C)$.

CONTACT AREA DETERMINATION: The contact areas of brushes were determined using a "cut and weigh" method. A paper patch of the bristle profile under every pressing depth was cut and weighed. The weight of each patch divided by the weight of the same paper of known area, yielded the contact area (see *Table* 4).

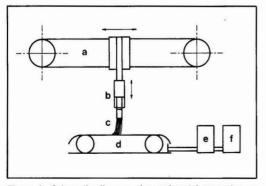


Figure 1—Schematic diagram of experimental apparatus. a: reciprocator; b: brush holder; c: brush; d: two-dimensional dynamometer; e: strain-meter; f: recorder

Table 2—Brush-size							
Brush	1	2	3	4			
Length (mm)	68	68	68	68			
Width (mm) 25		51	76	102			
Thickness (mm)	18	19	18	19			

PRESSING DEPTH PREPARATIONS: The procedure for setting the pressing depth of brush involved attaching a brush to the brush holder, letting the holder down to the zero-position where the end of bristles were almost in contact with the substrate, allowing the holder to let down by the desired depth.

THICKNESS DETERMINATION: The film thickness was calculated from the weight and density of the paint film applied.

MEASUREMENT PROCEDURE: The measurement procedure involved applying each sample to the substrate to 100 μ m thick film by a spatula, securing the substrate to the working table by means of clamps, moving the brush parallel across the substrate to measure the horizontal and vertical forces, and finally determining the film thickness. This procedure was repeated three or more times under the same condition so as to ensure the reproducibility of the measurements.

RESULTS AND DISCUSSION

Experimental Results

Typical oscillograms of the brush-strokes are shown in *Figure* 3. The horizontal, Fhobs, and vertical forces, Fvobs, measured were determined from the mean values of oscillograms and the average in the reciprostrokes; the accuracy of the reading was ± 3 grams. The average Fhobs, Fvobs, and thickness, h, of three measurements are shown in *Table* 3.

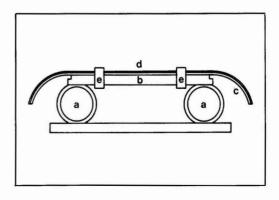


Figure 2—Two dimensional dynamometer. a: strain-gauged ring; b: working table; c: base panel of substrate; d: substrate; e: clamp

	Brushing Condition				Result	Measured	1		esult ulated		
No.	Brush No.	Pressing Depth of Brush (mm)	Contact Area of Brush A(cm ²)	Brush Width B(cm)	Viscosity of Sample η (ps)	Brush Speed v(cm/sec)	Wet Film Thickness h(×10 ⁻⁴ cm)	Fhobs (g)	Fvobs (g)	Fhcal (g)	Fvacl (g)
1	1	15	3.5	2.9	1.75	35.9	33	73	79	63	17,830
2	1	15	3.5	2.9	1.75	70.6	40	85	67	109	22,537
3	1	15	3.5	2.9	4.30	35.9	39	90	62	143	30,651
4	1	15	3.5	2.9	4.30	70.6	48	102	53	224	38,548
5	2	15	7.0	5.0	1.75	35.9	30	199	194	149	35,221
6	2	15	7.0	5.0	1.75	70.6	31	238	165	282	64,250
7	2	15	7.0	5.0	4.30	35.9	30	243	154	368	87,510
8	2	15	7.0	5.0	4.30	.70.6	39	276	128	556	101,969
9	3	10	9.5	7.3	1.75	35.9	33	143	239	183	42,146
10	3	10	9.5	7.3	1.75	70.6	37	172	208	324	66,109
11	3	10	9.5	7.3	4.30	35.9	38	188	196	391	78,285
12	3	10	9.5	7.3	4.30	70.6	42	218	155	700	128,342
13	3	15	10.6	7.5	1.75	24.5	32	171	266	146	32,735
14	3	15	10.6	7.5	1.75	35.9	33	198	274	208	45.085
15	3	15	10.6	7.5	1.75	44.0	33	213	243	250	53,255
16	3	15	10.6	7.5	1.75	53.8	33	224	234	306	65,010
17	3	15	10.6	7.5	1.75	70.6	35	236	235	386	77,742
18	3	15	10.6	7.5	4.30	24.5	32	232	237	356	78,888
19	3	15	10.6	7.5	4.30	35.9	37	257	219	456	88,185
20	3	15	10.6	7.5	4.30	44.0	36	270	199	563	109,740
21	3	15	10.6	7.5	4.30	53.8	40	288	184	631	112,570
22	3	15	10.6	7.5	4.30	70.6	40	304	173	822	145,772
23	3	15	10.6	7.5	8.13	24.5	37	284	196	582	111,352
24	3	15	10.6	7.5	8.13	35.9	44	310	170	723	117,302
25	3	15	10.6	7.5	8.13	44.0	46	323	154	848	131,440
26	3	15	10.6	7.5	8.13	53.8	51	339	137	922	127,255
27	3	15	10.6	7.5	8.13	70.6	56	360	124	1,109	140,162
28	3	20	14.7	8.1	1.75	35.9	32	305	289	301	53,028
29	3	20	14.7	8.1	1.75	70.6	33	370	234	556	91,962
30	3	20	14.7	8.1	4.30	35.9	33	391	219	702	117,380
31	3	20	14.7	8.1	4.30	70.6	39	447	177	1,178	168,305
32	4	15	18.6	10.0	1.75	35.9	32	399	279	373	62,726
33	4	15	18.6	10.0	1.75	70.6	36	444	225	653	97,746
34	4	15	18.6	10.0	4.30	35.9	34	471	208	854	133,703
35	4	15	18.6	10.0	4.30	70.6	44	520	161	1,300	157,667

Table 3—Experimental Conditions and Results

In Figure 4, the relationships between Fhobs, Fvobs, Fhobs/Fvobs, h, and the brush speed, v, are illustrated at a series of constant brush-size and pressing depth. Fhobs increased with increasing the speed v and viscosity η , while Fvobs decreased with v and η . Fhobs/Fvobs value increased with the increase in v and η . This result indicates that the direction of the force on the brush is changed with v and η . Thickness, h, gave an opposite trend to Fvobs, and increased with v and η .

The relationships between Fhobs, Fvobs, h, and the pressing depth with the same brush are shown in *Figure 5*. Fhobs increased with increasing pressing depth, speed v, and viscosity η . Fvobs gave a similar relationship, but decreased with the increase in v and η . Thickness h decreased with increasing pressing depth, and increased with v and η .

Discussion

Fhcal and Fvcal for each experiment were calculated by the equations (1) and (3) assuming a = 1. The average Fhcal and Fvcal of three measurements are given in *Table* 3.

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FHOBS — FHCAL RELATIONSHIP: Equation (1) does not seem to be applicable to practical brush application, as shown in *Table* 3. However, there was a good linear relation between Fhobs and Fhcal at a series of same brush-size and pressing depth as shown in *Figure* 6. A good correlation between Fhobs and Fhcal was found at every series (see *Table* 5), which is expressed by the following equation:

$$Fhobs = a_1 Fhcal + b_1 \tag{4}$$

Table 4—Width and Contact Area of Brush

Brush	Pressing Depth (mm)	Brush Width (mm)	Contact Area (cm ²)
No. 1	15	29	3.5
No. 2	15	50	7.0
No. 3	10	73	9.5
No. 3	15	75	10.6
No. 3	20	81	14.7
No. 4	15	100	18.6

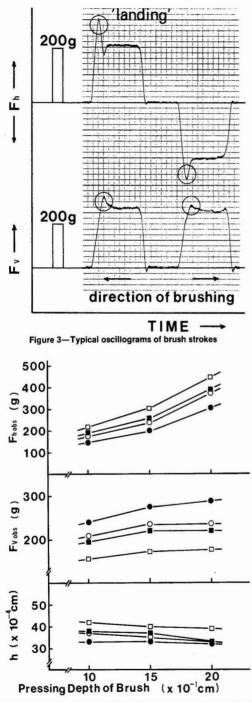


Figure 5—Dependence of Fhobs, Fvobs, h on pressing depth of brush. Brush size: No. 3; ●-Viscosity of sample: 1.75 (ps), Brush speed: 35.9 cm/sec; ○-Viscosity of sample: 1.75 (ps), Brush speed: 70.6 cm/sec; ■-Viscosity of sample: 4.30 (ps), Brush speed: 35.9 cm/sec; □-Viscosity of sample: 4.30 (ps), Brush speed: 70.6 cm/sec

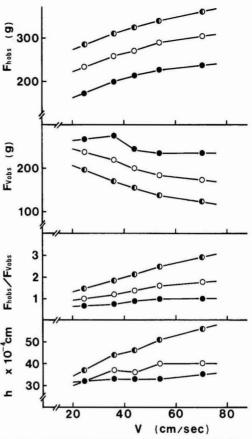


Figure 4—Dependence of Fhobs, Fvobs, Fhobs/Fvobs, h on brush speed. Brush size: No. 3; Pressing depth: 15 mm; ●-Viscosity of sample: 1.75 (ps); O-Viscosity of sample: 4.30 (ps); ●-Viscosity of sample: 8.13 (ps)

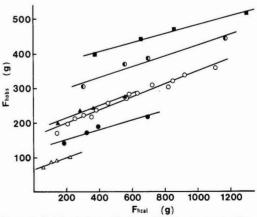


Figure 6—Relation between Fhobs and Fhcal. △-Brush size: No. 1., Pressing depth: 15 mm; ▲-Brush size: No. 2., Pressing depth: 15 mm; ●-Brush size: No. 3., Pressing depth: 10 mm; O-Brush size: No. 3., Pressing depth: 15 mm; 0-Brush size: No. 3., Pressing depth: 20 mm; ■-Brush size: No. 4., Pressing depth: 15 mm

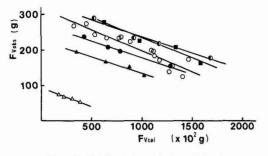


Figure 7—Relation between Fvobs and Fvcal

where $a_1 > 0$. Substitution of equation (1) for Fhcal in equation (4) results in the following expression:

$$Fhobs = \frac{a_1 A \eta v}{h} + b_1$$
 (5)

The relationship between a_1 and brush-size and pressing depth could not be found. Leakage and flow out can occur both through and at the sides of the bristles. Therefore, a_1 can be considered to be a correcting coefficient for treating actual brushing as an equivalent "plane model".

 b_1 increased with increasing pressing depth and brushsize; thus, b_1 can be equated with the horizontal component of the force required to give a certain pressing depth of brush.

FVOBS — FVCAL RELATIONSHIP: All slopes of the regression lines and correlation coefficients were negative, as shown in *Figure 7* and *Table* 6, indicating that the upward thrust calculated by using Mell-Finn equation (2) gave a contrary trend to the normal pressure measured.

However, according to the experimental results, the relationship between Fvobs and Fvcal is given by:

$$Fvobs = a_2 Fvcal + b_2$$
(6)

where $a_2 < 0$. Thus, substituting (3) into (6) gives:

$$Fvobs = \frac{a_2 B \eta v}{h^2} + b_2$$
(7)

FVOBS — FHCAL RELATIONSHIP: From the viewpoint of the deformation of the brush, the downward brush pressure was examined to estimate the variation of Fvobs.

Table 5—Correlation Between Fhobs and Fhcal

	Pressing Depth of Brush	Correlation Coefficient		ient of ion Line ation
Brush	(mm)	r ₁	a ₁	b ₁
No. 1	15	0.988	0.17	64
No. 2	15	0.979	0.18	178
No. 3	10	0.974	0.14	124
No. 3	15	0.987	0.19	163
No. 3	20	0.976	0.15	272
No. 4	15	0.995	0.13	356

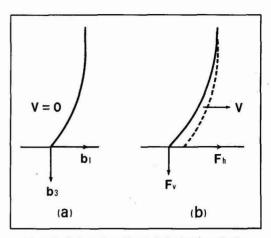


Figure 8-Deformation of brush during brush application

It is assumed that the brush is in a state of Figure 8 (a) before moving, the horizontal and vertical components of the applied force being b_1 and b_3 ; the brush will then deform to be in equilibrium with the horizontal force Fh given by equation (4) as it moves parallel across the substrate [see Figure 8 (b)].

Assuming that the deformation, as shown in Figure 8 (b), lowers the downward pressure and the decrease is approximately proportional to the varying amount of Fh, the vertical force Fv can be expressed as follows:

$$Fv = \frac{a_3 A \eta v}{h} + b_3$$
(8)

where $a_3 < 0$.

A good correlation between Fvobs and Fhcal (= A η v/h) was found, as shown in *Figure* 9 and *Table* 7. b₃ can be equated with the vertical component of the force required to give a certain pressing depth of the brush.

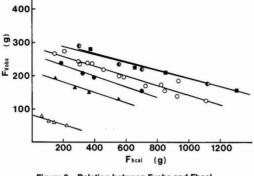
Brushing of Cassonian Fluid

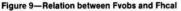
It is well known that flow characteristics of the vehiclepigment system can be represented using the Casson equation:

$$S^{\prime 2} = S_0^{\prime 2} + \eta_{\infty}^{\prime 2} D^{\prime 2}$$
(9)

Table 6-Correlation Between Fvobs and Fvcal

	Pressing Depth of Brush	Correlation Coefficient	Coeffici Regressio Equa	on Line
Brush	(mm)	r ₂	a ₂	b ₂
No. 1	15	-0.967	-0.0011	97
No. 2	15	-0.979	-0.0009	227
No. 3	10	-0.992	-0.0009	274
No. 3	15	-0.938	-0.0012	318
No. 3	20	-0.985	-0.0009	331
	15	-0.976	-0.0011	347





where S is the shearing stress, D is the rate of shear, S_o is the yield value, and η_{∞} is the residual viscosity.

Measurements of Fh and Fv were also made of the Cassonian fluids (polyalkeleneglycol derivatives pigmented with calcium carbonate) under similar brushing conditions, and measured values of Fh and Fv were compared with the calculated ones from equations (1), (3), and (9), which resulted in the following.

The horizontal force Fhc of Cassonian fluid could be given by:

Fhc =
$$\frac{a_1 A[(S_0 h)^{\frac{1}{2}} + (\eta_{\infty} v)^{\frac{1}{2}}]^2}{h} + b_1$$
(10)

The vertical force Fvc could be given using Mell-Finn equation (3):

Fvc =
$$\frac{a_2 B \left[(S_0 h)^{\frac{1}{2}} + (\eta_\infty v)^{\frac{1}{2}} \right]^2}{h^2} + b_2$$
(11)

and using equation (1) from the view point of the deformation of brush:

Fvc =
$$\frac{a_3 A \left[(S_{\alpha} h)^{\frac{1}{2}} + (\eta_{\infty} v)^{\frac{1}{2}} \right]^2}{h} + b_3$$
(12)

CONCLUSION

If the brush size, pressing depth, and initial film thickness are defined, the horizontal force Fh, and

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Table 7—Correlation Between Fvobs and Fhcal

Brush	Pressing Depth of Brush	Correlation Coefficient	Coefficient of Regression Line Equation	
	(mm)	r ₃	a ₃	b ₃
No. 1	15	-0.974	-0.16	86
No. 2	15	-0.991	-0.16	214
No. 3	10	-0.990	-0.16	262
No. 3	15	-0.964	-0.15	287
No. 3	20	-0.967	-0.12	313
No 4		-0.984	-0.12	316

vertical one Fv of Newtonian and Cassonian fluids can be expressed by the same following equations:

$$Fh = \frac{a_1 A \eta a v}{h} + b_1$$
(13)

$$Fv = \frac{a_2 B \eta a v}{h^2} + b_2 \tag{14}$$

$$Fv = \frac{a_3 A \eta a v}{h} + b_3$$
(15)

where A is the contact area of the brush, B is the brush width, v is the brush speed, h is the film thickness, ηa is the apparent viscosity of the fluid at the shear rate (= v / h), a's are correcting coefficients and b's are constants concerning the horizontal and vertical components of the initial force applied the brush to bend.

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1

Factorial Experimental Design: Determination of Hydroxyl Content In Polymers

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A statistical method for experimentation facilitated investigation of the following methods for determining hydroxyl content in polymers: an imidazole catalyzed esterification with pyromellitic dianhydride; two acid catalyzed acetylations utilizing perchloric and p-toluenesulfonic acids as catalysts; and a dibutyltin dilaurate catalyzed urethane formation with phenyl isocyanate. The statistical method involved a factorial experimental design where three independent variables were evaluated at two levels (two level three factorial design). This approach facilitated comparison of the methods for determination of hydroxyl content in polymers by efficiently optimizing reaction parameters.

INTRODUCTION

Many polymeric materials used in the coatings industry, such as polyester, polyurethane, alkyd, acrylic and epoxy resins, contain hydroxyl functionality. Accurate quantitative determination of this functional group is necessary to characterize these types of materials and to predict their coating properties. Development and evaluation of methods for quantitative analysis of this functional group have been historically achieved by employing simple, non-polymeric materials. Since previous methods were not routinely evaluated with polymeric materials, a comprehensive evaluation of various methods for the quantitative analysis of hydroxyl groups in polymers was initiated. The depth of information obtained was greatly enhanced by the use of a statistical experimental approach, a two level three factorial design. Several physical properties of polyester polymers can be related to hydroxyl content. When difunctional acids and polyols are used, molecular weight is inversely proportional to the amount of this functional group remaining, that is, the higher the molecular weight the lower the hydroxyl number. In general, as the molecular weight increases the viscosity of the polyester increases and the ability to flow decreases. In formulations containing polyesters, these physical properties have a definite influence upon the application and performance of the final product. Therefore, correlation of physical properties with the hydroxyl content of polyesters provides a means of predicting the performance of coatings.

Many hydroxyl containing polymers cure by means of crosslinking agents such as urea, melamine, or benzoquanamine-formaldhyde resins. These polymer systems include epoxides, alkyds, and acrylics. The alkoxy groups of the melamine resin react with the hydroxyl groups of the polymer backbone via a condensation reaction forming polymer-melamine resin crosslinks. The polymers are then crosslined into films by the further condensation reaction of two melamine alkoxy groups or by the formation of additional polymer-melamine crosslinks as described above. Since the stoichiometry between the hydroxyl functionality of the polymers and the crosslinkers affects the cure properties, accurate determination of the levels of this functional group in the polymer is necessary.

Various other materials used in coating formulations contain hydroxyl groups. Examples include natural or synthetic vegetable oils and raw materials such as polyols and nonionic surfactants. These materials are frequently chemically combined with polymers or other materials which have mutually reactive functional groups. To obtain the desired properties in the final products, the determination of the reactive functional groups in the starting material is crucial.

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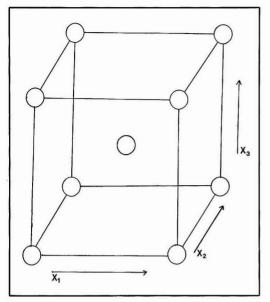


Figure 1—Three dimensional representation of two level three factorial design. Each axis represents a parameter evaluated. Two vertices on any axis represents the low and high extreme for that parameter

From the examples given it can be seen that hydroxyl containing materials are utilized frequently within the coatings industry. The ability to accurately quantify the amount of this functional group in these materials, especially in polymeric systems, would greatly enhance formulation of these polymers and their respective coatings. In addition, accurate quantitative analysis of hydroxyl groups will assist in the prediction of the coating properties of these materials. There are several methods available for the determination of hydroxyl content.¹⁻⁴ Esterifications involving carboxylic acids,⁵ acid chlorides,⁶⁻⁸ and acid anhydrides⁹⁻¹⁴ comprise the most general method for quantitative analytical purposes. The general reactions for these are illustrated by equations (1)-(3).

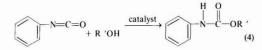
$RCOOH + R'OH \rightarrow RCOOR' + H_2O$	(1)
$RCOCI + R'OH \rightarrow RCOOR' + HCI$	(2)
$(RCO)_2O + R'OH \rightarrow RCOOR' + RCOOH$	(3)

Acid anhydrides are the most widely used reactive species for quantitative esterifications, with acetic anhydride, phthalic anhydride, and pyromellitic dianhydride being the reagents of choice. Of these anhydrides, acetic anhydride in the presence of a catalyst, has been most frequently utilized. Catalysts include bases, Lewis acids, mineral acids, and other strong acids. Pyridine is the basic catalyst of choice while sulfuric, hydrochloric, perchloric, 2,4-dinitrobenzenesulfonic, and p-toluenesulfonic acids have been used as acidic catalysts. Acid catalyzed acetylations have fairly short analysis times but are sensitive to the presence of aldehydes.

The reaction of phthalic anhydride with hydroxyl groups is not as rapid and less widely applied than the acetic anhydride method. Aldehydes do not interfere with phthalation, but this reagent fails to esterify phenols. This selectivity provides a method for determining alcohols in the presence of phenolic compounds. Pyromellitic dianhydride,^{13,14} on the other hand, combines the advantages of acetylations and phthalations without their disadvantages. Aldehydes do not interfere with this reagent; it can be used to analyze alcohols in the presence of phenols and its rate of reaction can be favorably compared with that of acetic anhydride.

Another type of method for the determination of hydroxyl content involves urethane formation via phenyl isocyanate. This reaction is usually catalyzed by organotin compounds such as stannous octoate.¹⁵ Equation (4) depicts the general reaction for this method.

Table 1—Standard Polymers						
Polymer		Туре	Theoretical Hydroxyl Number mg KOH/g sample	Theoretical Acid Value mg KOH/g sample	Theoretical % NVM	Hydroxyl Functionality Present
Standard 1		High solids polyester	88	5	80.6	1° + 2°
Standard 2		High solids polyester	72	1	79.0	1°
Standard 3		High solids polyester	24	9	80.3	1°
Standard 4		High solids polyester	31	10	78.0	1° + 2°
Standard 5		Styrenated acrylic	14		39.8	l°
Standard 6		Styrenated acrylic	40		39.9	1°
Standard 7		Styrenated acrylic	26	16	65.0	lo



This method can successfully quantify hydroxyl groups in the presence of oxirane groups, a capability the esterification reactions do not have.

Historically, most of the above-mentioned methods were evaluated using hydroxyl containing materials which would be considered non-polymeric. These materials included compounds such as aliphatic, alicyclic and aralkyl alcohols, and polyols. The hydroxyl groups on these compounds behave "ideally" compared to those found in polymeric systems. The reactivity of hydroxyl groups in a polymer can be affected by steric hindrance from the polymer itself as well as by their location along the polymer backbone. These effects are not as severe in non-polymeric materials and, therefore, the reactivity of these lower aliphatic hydroxyl groups remain relatively constant. Consequently, polymer systems react differently with the various reagents for determining hydroxyl functionality.

This paper describes the optimization and evaluation of four methods for the quantitation of hydroxyl functionality utilizing actual polymer samples. The methods choosen were: two acid catalyzed acetylations using perchloric9 and p-toluene-sulfonic acid as catalysts,¹⁰ pyromellitic dianhydride catalyzed by imidazole¹⁴ and urethane formation using phenyl isocyanate. The two acetic anhydride methods were chosen due to their wide spread use throughout industry and the short analysis time for non-polymeric materials. The imidazole catalyzed pyromellitic dianhydride method was chosen because it has enhanced reactivity and does not suffer aldehyde interferences. Since the phenyl isocyanate method can be used for determination of hydroxyl in the presence of oxirane groups as found in some epoxy resins, it was included in this investigation.

The parameters optimized were: reaction time, reaction temperature, and sample size (concentration of hydroxyl functionality). The optimization allowed a comparison of the methods for their application to a wide variety of hydroxyl containing polymers. To optimize these methods efficiently, a statistical experimental approach was employed. This approach, a two level three factorial design, minimized experimental time while maximizing data accumulation and correlation.

THEORY

Scientific research has historically employed two general classes of experimental strategies: a conventional and a statistical approach. The conventional method is characterized by examining one variable while holding all others constant. This one-factor-at-a-time experimentation is simple in concept and easily performed, though it requires many experimental levels for each factor. By necessity, this approach assumes there are no interactions of factors and that experimental error is negligible relative to the effects of the factors. The statistical approach, characterized by factorial designs, provides experimental strategies which assume that factor interactions are typical and that experimental error is not negligible relative to the effects of the factors. Not only are the number of experimental levels for each factor greatly reduced using this strategy, but the effects of several factors can be estimated simultaneously.¹⁶⁻¹⁸

Factorial experimental designs can be applied to many areas within the coatings industry. One type of factorial design, a simplex lattice, has been successfully applied to a mixture study for formulating coatings.¹⁹ This approach allowed prediction of the optimum amounts of principal components that produced specific properties. These properties maximized performance while minimizing costs. Other coatings applications suited for factorial designs are the investigation of physical and chemical properties of polymer materials as well as optimization of chemical reactions for resin formulation.

Two level factorial designs are easy to plan, perform, and analyze. These strategies are not only readily adaptable to a variety of problems, but can be used on both continuous and discrete factors. Continuous factors or independent variables are those which can assume any value over a numerical range. For experimental purposes, these factors are set at a low and a high value. Examples of continuous variables could be temperature, pressure, or concentration. Discrete factors are usually qualitative and assume only specific levels. Examples of discrete factors could be catalyst type or batch of raw materials.

Table 2—Reaction Conditions for Factorial Design^a

Method	Polymer	meq OH	Reaction Time, Min.	Reaction Temp., °C
 PMDA	Standard 1	2, 4.5, 7	10, 35, 60	25, 60, 100
PMDA	Standard 2	3, 6, 9	10, 35, 60	25, 60, 100
pTSA	Standard I	3, 6, 9	30, 75, 120	25, 35, 50
pTSA	Standard 2	3, 6, 9	30, 75, 120	25, 35, 50
HClO4	Standard 1	2, 4, 6 ^b	10, 35, 60	25, 55, 85
HC104	Standard 2	2, 4, 6 ^b	10, 35, 60	25, 55, 85

(a) Total of 24 reactions for each method-polymer combination, including four blanks. Midpoint for each parameter run in quadruplet, duplicate for parameter extremes.
 (b) From model compound data Figure 5 a and b.

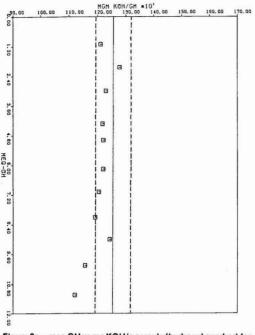


Figure 2a—meq OH vs mg KOH/g sample (hydroxyl number) for PMDA method and trimethylol propane; - theoretical hydroxyl number TMP is 1256 mg KOH/g: --- \pm 5% relative error from theoretical. Reaction run at room temperature for 60 min

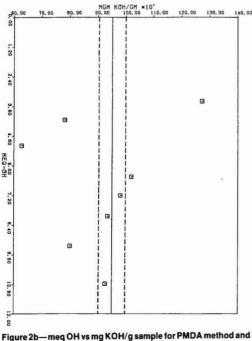


Figure 2b—meq OH vs mg KOH/g sample for PMDA method and 2,5-hexanediol; theoretical hydroxyl number 2,5-hexanediol is 951 mg KOH/g;---±5% relative error from theoretical. Reaction run at room temperature for 60 min

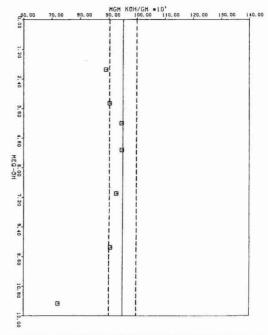


Figure 2c—meq OH vs mg KOH/g sample for PMDA method and 2,5-hexanediol; theoretical hydroxyl number 2,5-hexanediol is 951 mg KOH/g;--±5% relative error from theoretical. Reaction run at 100°C for 60 min

The three dimensional representation of a two level three factorial design is a cube (Figure 1). Each of the three axes (X1, X2, X3) represents one factor or parameter which is to be evaluated with the two vertices on any one axis dipicting the low and the high extreme. In the work described here, the factors evaluated were reaction temperature, reaction time, and hydroxyl content (sample size). The eight corners symbolize an experimental point which is run in duplicate. The center of the cube, the midpoint for each parameter, is run in quadruplicate as a check on the reproducibility of the data. Replication of each experimental point is used to reduce the effect of any random error present, while bias error is reduced by randomizing the order in which the experimental points are derived.

The results from each experimental point are utilized to generate response surface plots. These plots are actual slices of the cube at various points along one axis. The response surface plots usually contain contour lines which are statistically defined by the results from each experimental point and are unique solutions to a series of simultaneous equations which define a cubic matrix.²⁰

EXPERIMENTAL

Standard Polymers

Various high solids polyesters and styrenated acrylic polymers were synthesized under controlled conditions,

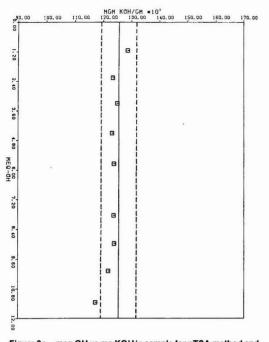


Figure 3a—meq OH vs mg KOH/g sample for pTSA method and TMP; theoretical hydroxyl number TMP is 1256 mg KOH/g; --- \pm 5% relative error from theoretical. Reaction run at 50°C for 60 min

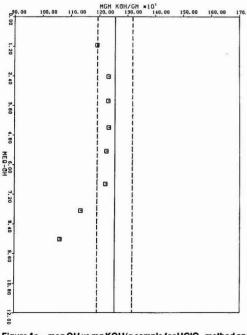


Figure 4a—meq OH vs mg KOH/g sample for HCIO₄ method and TMP; theoretical hydroxyl number TMP is 1256 mg KOH/g; ---± 5% relative error from theoretical. Reaction run at room temperature for 30 min

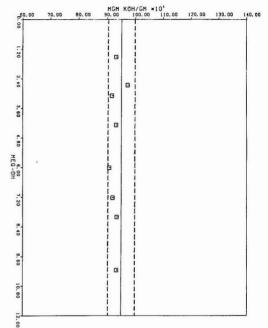


Figure 3b—meq OH vs mg KOH/g sample for pTSA and 2,5-hexanedic); theoretical hydroxyl number 2,5-hexanedici is 951 mg KOH/g;---±5% relative error from theoretical. Reaction run at 50°C for 60 min

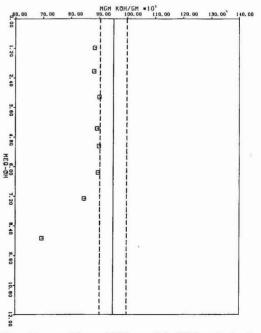


Figure 4b—meq OH vs mg KOH/g sample for HClO₄ method and 2,5-hexanediol; theoretical hydroxyl number 2,5-hexanediol is 951 mg KOH/g; --- \pm 5% relative error from theoretical. Reaction run at room temperature for 30 min

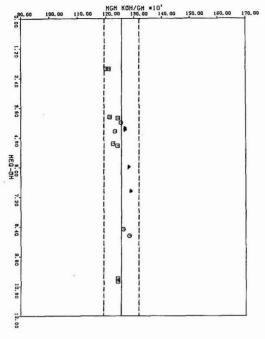


Figure 5a—meq OH vs mg KOH/g sample for phenyl isocyanate method and TMP; theoretical hydroxyl number is 1256 mg KOH/g;--- \pm 5% relative error from theoretical. \triangle Run at 20°C for 24 hours; \bigcirc Run at 20°C for 24 hours; and \square Run at 50°C for 2 hours

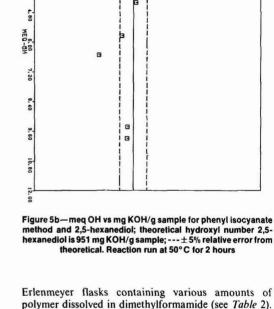
which minimized loss of starting materials. Theoretical numbers were based on the molar ratios of starting materials and extent of reaction as determined by monitoring the acid number. The type of hydroxyl groups present (i.e., primary and/or secondary) was ascertained by the type of starting materials utilized. *Table* 1 lists the theoretical hydroxyl content, the types of hydroxyl groups present and the acid values for the standard polymers.

Method A

IMIDAZOLE CATALYZED ESTERIFICATION OF PYROMELLITIC DIANHYDRIDE:

PYROMELLITIC DIANHYDRIDE REAGENT:¹⁴ Pyromellitic dianhydride (Eastman Chemical, practical grade) was heated at 160°C for 24 hours. A 0.19 M solution of pyromellitic dianhydride in dimethylformamide (Baker, reagent grade) was allowed to stir for 30-45 minutes. Imidazole (Eastman Chemical) was added to make the solution 0.31 M in this substrate and mixed for an additional 30 minutes. The final solution should be clear and reddish brown in color. Cloudy reagent is unsuitable for use and is due to a high moisture content in the pyromellitic dianhydride or the dimethylformamide. The reagent is stable for at least twenty-four hours if securely sealed.

PROCEDURE: Fifty mL of pyromellitic dianhydride reagent were pipetted into 500 mL glass stoppered



70.00

1. 20

2. 40

80,00

G

polymer dissolved in dimethylformamide (see *Table 2*). The samples were allowed to react at room and elevated temperatures for different times. Water baths and reflux condensers were utilized for reactions run at elevated temperatures. The reactions were stopped by the addition of 150 mL of deionized water and allowed to stir for 10 minutes. Reactions containing acrylic polymers were stopped by the addition of 50 mL of deionized water. The additional dimethylformamide increases the solubility of the reaction products. All samples were titrated potentically with 0.5 N methanolic KOH. Reagent blanks were treated in the same manner without the addition of sample.

MGM KOH/GM =10'

a

120.00

190.00 140.00

Method B

ACETIC ANHYDRIDE ACETYLATION CATALYZED BY P-TOLUENESULFONIC ACID;

P-TOLUENESULFONIC ACID REAGENT: A 0.53 M acetic anhydride - 0.04 M p-toluenesulfonic acid solution in ethyl acetate was prepared. The reagent was kept tightly capped and discarded after three days. All reagents were purchased from Fischer Scientific Co.

PROCEDURE: Twenty-five mL of p-toluenesulfonic acid reagent were accurately pipetted into glass stoppered 250 mL Erlenmeyer flasks containing various amounts of sample dissolved in ethyl acetate. The reaction was allowed to proceed for different lengths of time at different temperatures (see *Table 2*). The reaction was stopped by the addition of 10 mL of a 3:1 (by volume) pyridine - water mixture. The samples were swirled to insure complete mixing and allowed to stand for 15-30 minutes. Fifty mL of reagent grade tetrahydrofuran were added to each flask to help solubilize the reaction products prior to potentiometric titration with 0.5 N methanolic KOH. Reagent blanks were treated in the same manner.

Method C

ACETIC ANHYDRIDE ACETYLATION CATALYZED BY PERCHLORIC ACID:

PERCHLORIC ACID REAGENT: A 0.57 M acetic anhydride-0.04 M perchloric acid solution in ethyl acetate was prepared. The reagent was kept tightly capped and used immediately.

PROCEDURE: Twenty mL of perchloric acid reagent were accurately pipetted into 250 mL glass stoppered Erlenmeyer flasks containing various amounts of polymer dissolved in ethyl acetate. The flasks were allowed to react at room and elevated temperatures for different times (*Table* 2). Water baths and reflux condensers were utilized for reactions run at elevated temperatures. The reaction was stopped by the addition of 35 mL of a dimethylformamide-pyridine-water mixture (6:3:1 by volume) and then potentiometrically titrated with 0.5 N methanolic KOH. Reagent blanks were carried through the same procedure.

Table 3—Effect of Various	Reaction Conditions
On Phenyl Isocyanate	e Reagent Blank ^a

Time, Hours	Temp., °C	meq Isocyanate
2	20	14.67
6	20	13.60
4	45	11.04
2		12.94
6		10.55

(a) All determinations from same reagent solution.

Method D

PHENYL ISOCYANATE CATALYZED BY DIBUTYLTIN DILAURATE:

PHENYL ISOCYANATE REAGENT: A 1.0 M phenyl isocyanate (MCB chemicals) solution in tetrahydrofuran was prepared.

DIBUTYL AMINE REAGENT: A 2.0 M solution of dibutyl amine in toluene was made.

PROCEDURE: Fifteen mL of phenyl isocyanate reagent were accurately pipetted into 250 mL glass stoppered Erlenmeyer flasks containing various amounts of sample dissolved in tetrahydrofuran. Fifteen milligrams of catalyst, dibutyltin dilaurate, were added to each flask. The flasks were then placed in water baths, attached to reflux condensers and allowed to react for various times at different temperatures (*Table* 2). The reaction was

Table 4—Hydroxyl Numbers Determined by HCIO₄ Method on Standard 1

meq Hydroxyl Added	Reaction Temp., °C	Reaction Time, Min.	Experimental Hydroxyl Number mg KOH/g sample ^a	Theoretical Hydroxyl Number mg KOH/g sample
2	25	60	247 ± 4	88
2	100	60	255 ± 16	88
	55	35	116 ± 14^{b}	88
6	25	60	82 ± 4	88
6		60	85 ± 2	88

(b) Average of four runs.

Table 5—Hydroxyl Number Determination	by HCIO	Method on Standard 2
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meq Hydroxyl Added	Reaction Temp., °C	Reaction Time, Min.	Experimental Hydroxyl Number mg KOH/g sample ^a	Theoretical Hydroxyi Number mg KOH/g sample
2	25	60	76	72
2	100	60	104	72
4	55	35	78 ^b	72
6	25	60	68	72
6	100	60	70	72

(b) Average of four runs.

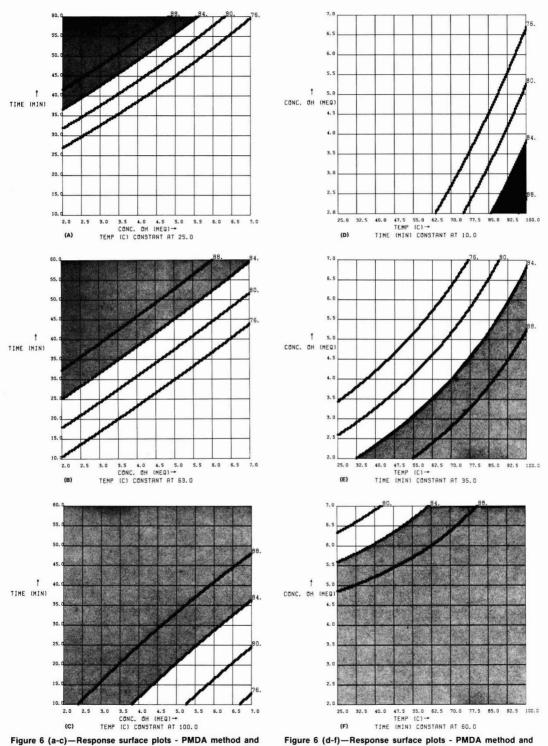


Figure 6 (a-c)—Response surface plots - PMDA method and Standard 1; contour line 88 - 88 mg KOH/g sample; contour line $84 \pm 5\%$ relative error from theoretical. Effect of temperature

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Standard 1; contour line 88 - 88 mg KOH/g sample; contour line

stopped by the addition of 10 mL of 2 M dibutyl amine reagent. The dibutyl amine reacts with unreacted phenyl isocyanate. After 15 minutes, 100 mL of methyl cellosolve were added and the samples titrated potentiometrically with 0.5 N perchloric acid in methyl cellosolve. Reagent blanks were carried through the same procedure.

CALCULATIONS: For esterification Methods A, B, and C:

Hydroxyl number =	(B - S) (0.5 N) (56.1 mg KOH/meq)	mg KOH
Hydroxyr number –	A	g sample

For the phenyl isocyanate Method D:

Hydroxyl number = $\frac{(S - B) (0.5 N) (56.1 mg KOH/meq)}{A} = \frac{mg KOH}{g sample}$

Where A =Sample size in grams B =Reagent blank titration in mL

S = Sample titration in mL

RESULTS AND DISCUSSION

Each method was originally evaluated with two model compounds to insure that analytically acceptable results could be obtained.²¹ The compounds selected were trimethylol propane which contains primary hydroxyl functionality and 2,5-hexanediol, containing secondary hydroxyl functionality. These compounds were evaluated at various sample to reagent ratios, as shown in Figures 2-5. Each method gave acceptable analytical results (\pm 5%) relative error from theoretical) over a wide range of sample sizes, with the exception of the data for the pyromellitic dianhydride method (PMDA) for 2,5hexanediol (Figure 2b). The erratic data was eliminated by elevating the reaction temperature from room temperature to 100° C (Figure 2c). The room temperature results are probably due to the lower reactivity of secondary hydroxyl groups, which was resolved by elevating the reaction temperature.

During this experimentation, problems were encountered with the phenyl isocyanate method. Inconsistent results were obtained with the model compounds when stannous octoate was utilized as the catalyst. Further investigation revealed a variable reagent blank. Based on the type of catalysts frequently utilized in urethane reactions, dibutyltin dilaurate was selected to resolve this difficulty.²²

Figures 5a and b represent the data accumulated from the model compounds using dibutyltin dilaurate as the catalyst. The 2,5-hexanediol data (Figure 5b) indicates some variability in the determined hydroxyl content. It is possible that the reaction conditions utilized did not completely compensate for the lower reactivity of the secondary hydroxyl groups contained within this compound. The results for the trimethylol propane (Figure 5a) were acceptable, indicating the method is quantitative.

The use of dibutyltin dilaurate as a catalyst did not completely eliminate the variable reagent blank. To investigate this problem, various reaction conditions were applied to reagent blanks. The results (*Table* 3) indicate that the amount of available isocyanate decreases as the reflux time and temperature are increased. This

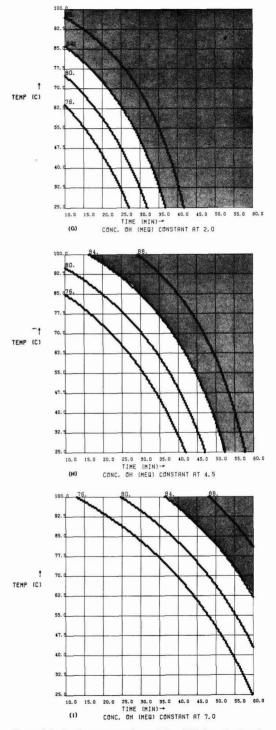


Figure 6 (g-i)—Response surface plots - PMDA method and Standard 1; contour line 88 - 88 mg KOH/g sample; contour line $84 \pm 5\%$ relative error from theoretical. Effect of sample size

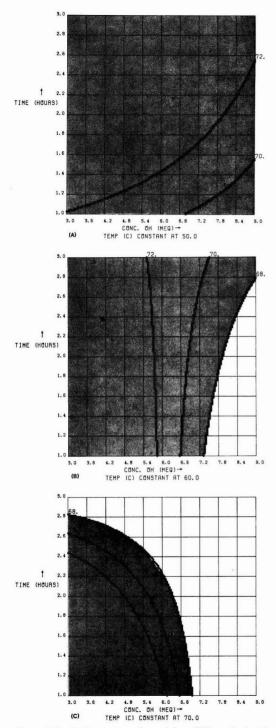


Figure 7 (a-c)—Response surface plots - pTSA method and Standard 2; effect of temperature on the reaction; contour line 72 - 72 mg KOH/g sample, contour 68 \pm 5% relative error from theoretical

74

deviation may be due to dimer (uretidione) and trimer (cyanurate) formation of the phenyl isocyanate. This method was not pursued further because of the variability in the reagent blank.

The three remaining methods were optimized utilizing the statistical design previously discussed. This approach allowed each method to be optimized with 24 determinations per standard polymer. Since two standard polymers were used for each method, a total of 48 reactions were required per method for optimization. This entailed two days of experimental time. The conventional approach of varying one factor at time would have increased experimental time by a factor of four to eight. The standard polymers, high solids polyesters, contained primary and secondary hydroxyl functionality as described in *Table* 1.

The perchloric acid catalyzed acetylation method (HClO_4) produced high and erratic results for Standard 1, a polyester containing polyethylene glycol (*Table 4*). A possible explanation for the data is the oxidation of the ether linkages by perchloric acid. Similar results have been reported when this method was applied to polypropylene and polyethylene glycol.^{1,2,4} However, adequate data was obtained for Standard 2, which contains no ether linkages (*Table 5*).

The results from the factorial design experiments for the PMDA and p-toluenesulfonic acid catalyzed acetylation (pTSA) methods were statistically analyzed to produce response surface plots such as those found in Figures 6 a-i. Each set of plots a-c, d-f, and g-i, symbolizes a series of slices through the cube at various points along one axis (parameter). The contour lines are numerically labeled with hydroxyl numbers (mg KOH/g sample) and are derived from the experimental data. These lines represent regions in the cube where particular hydroxyl numbers can statistically be achieved. Curvature of the contour lines suggests there is interaction between the two parameters being varied. As the curvature of these lines increases the interaction between the parameters increases. The distances between contour lines increase as optimum conditions are approached. This indicates a greater statistical probability of achieving acceptable data. Contour line 88 represents the theoretical hydroxyl number for this polyester (88 mg KOH/g sample) while the contour line at 84 represents 5% relative error from theoretical. The important portions of these plots are in the shaded area which delineate the regions where analytically acceptable data can be obtained.

The response surface plots (*Figures* 6 a-i) indicate the effect of temperature, time, and sample size on the esterification of the PMDA reagent with Standard 1. This polyester contains both primary and secondary hydroxyl functionality. The shaded areas in *Figures* 6 a-c increased as reaction temperature increased indicating the optimum temperature as 100° C. In *Figures* 6 d-f, where the effect of reaction time is evaluated, the shaded areas again increase as reaction times increase. The data indicates that the optimum reaction time is 60 minutes. The effect of sample size is measured in *Figures* 6 g-i, where the opposite trend is seen. As milliequivalents of hydroxyl increase, the regions where analytically accept-

	PMDA Method			pTSA Method		
Type of Hydroxyl	Sample Size meq OH	Temp °C	Time Minutes	Sample Size meq OH	Temp °C	Time Hours
1°	3-5	100	30	3-5	50-55	2
			or			
		25	60			
1° and 2°	3-5	100	60	3-5	50-55	2

Table 6—Optimized Reaction Conditions from Surface Response Curves

able data are obtained decreases. The optimum amount of hydroxyl functionality is 2-5 milliequivalents.

The response surface plots from the PMDA method utilizing Standard 2 and the pTSA method utilizing Standards 1 and 2 were evaluated in a similar manner. The optimized reaction conditions determined for the PMDA and pTSA methods are summarized in *Table* 6. The PMDA method can be used at either room temperature or 100°C for primary hydroxyl groups, while secondary hydroxyl functionality requires elevated temperature for completion of reaction. The optimum reaction conditions for the pTSA method are the same for both primary and secondary hydroxyl functionality.

Since the acetic anhydride used in the pTSA reagent is very volatile, concern was expressed regarding the possibility of its volatilization at elevated reaction temperatures. Figures 7 a-c depict the effect of increasing reaction temperature on the acetylation of Standard 2 with pTSA reagent. The theoretical hydroxyl number for this polyester is 72 mg KOH/g sample which is represented by contour line 72. Contour line 68 represents 5% relative error from the theoretical value. As temperature is increased from 50 to 70° C, the shaded areas decrease and the distances between the contour lines decrease. These trends suggest that the optimum temperature has been exceeded. An explanation for this phenomenon is loss of the acetic anhydride through the top of the reflux condensor as the solvent (ethyl acetate) refluxes. Based on this data the reaction temperature recommended for the pTSA method is 50-55°C.

The optimized conditions obtained for these methods were verified using Standards 3-7 (*Table* 1). Standards 3 and 4 are polyesters containing low hydroxyl content as well as primary and secondary hydroxyl groups. Standards 5-7 are styrenated acrylic polymers containing various amounts of hydroxyl functionality. Standard 7 also contains a significant amount of acid. This aids in determining what effect, if any, this functional group has on the determination of hydroxyl content.

The results obtained from Standards 3 and 4 are found in *Table* 7. Both methods resulted in accurate and reproducible hydroxyl numbers using the optimized reaction conditions previously attained. When dealing with polymers containing low hydroxyl numbers (below 20 mg KOH/g sample), sample sizes should not exceed 10-12 grams. Sample sizes greater than this tend to restrict the rate of esterification of the reagents with the hydroxyl functionality.

Table 8 represents the data from the acrylic Standards 5-7 using both methods. Reaction conditions indicated in the table are optimized conditions. Accurate and reproducible hydroxyl numbers were obtained in both cases. The reproducibility of the PMDA method appears to be somewhat better than that from the pTSA method as indicated by the data in *Tables* 7 and 8. This trend can be explained by the high volatility of the acetic anhydride is a solid, it is less likely to be lost during the reaction.

The effect of polymer material on the determination of hydroxyl content is indicated in *Table* 9. There are nine milliequivalents of anhydride available for esterification in the PMDA reagent while the pTSA reagent contains 13 milliequivalents. The results indicate that nonpolymeric compounds consistently give analytically acceptable results at large sample to reagent ratios (meq hydroxyl:meq anhydride). Polymeric materials, on the other hand, fluctuate between large deviations from theoretical to acceptable data at large sample to reagent ratios. This behavior can be attributed to polymers present in the reaction. In general, the larger the sample

Polymer	Type of Hydroxyl	Theoretical OH# mg KOH/g sample	PMDAOH# ^a mg KOH/g sample	Reaction Conditions	pTSA—OH# ^a mg KOH/g sample	Reaction Conditions
Standard 3	l°	24	23 ± 1	3-5 meq OH 60 min. 25°C	21 ± 1	3-5 meq OH 2 hr 50-55°C
Standard 4	1° and 2°	31	32 ± 1.5	3-5 meq OH 60 min. 100° C	31 ± 2	3-5 meq OH 2 hr 50-55°C

(a) Average of duplicate runs.

Polymer	Type of Hydroxyl	Theoretical OH# mg KOH/g sample	PMDA—OH# ^a mg KOH/g sample	Reaction Conditions	pTSA—OH# ^a mg KOH/g sample	Reaction Conditions
Standard 5	l°	14	16 ± .1	3-5 meq OH 60 min. 25°C	15 + .2	3-5 meq OH 2 hr 50-55° C
Standard 6	1°	40	40 ± .1	3-5 meq OH 60 min. 25°C	38 ± 1.8	3-5 meq OH 2 hr 50-55°C
Standard 7 ^b .	l°	23	23 ± .5	3-5 meq OH 60 min. 25° C	23 ± 2.5	3-5 meq OH 2 hr 50-55° C
(a) Average of d (b) Sample cont						

Table 8—Hydrox	cyl Number Determined b	y the PMDA and p	TSA Methods for the Acr	lic Polymer Standards
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size, the lower the probability of each hydroxyl group encountering reagent in a given reaction time. When polymeric material is present in the solution, this probability can fluctuate considerably. These fluctuations can be attributed to the thermodynamic interactions between the polymers and the solvent system. Model compounds, such as trimethylol propane and 2,5hexanediol, are not as subject to this problem. This type of an effect is negligible when the recommended reaction conditions are utilized.

SUMMARY

This work describes the investigation of four methods for determining hydroxyl content. Two methods, perchloric acid catalyzed acetic anhydride and phenyl isocyanate were not pursued because of various limitations. The applicability of the perchloric acid method is limited by the oxidation of ether linkages while the phenyl isocyanate method was plagued by an unsolvable reagent blank variability. A statistical experimental approach, a two level three factorial design, was utilized which allowed each method to be investigated in at least half the time of the conventional one-variable-at-time method. In addition, this factorial design allowed the estimation of interactions between factors. This type of approach was efficient, relatively uncomplicated to plan, and is applicable to a wide variety of investigations.

The two remaining methods, imidazole catalyzed esterification with pyromellitic dianhydride and ptoluenesulfonic acid catalyzed acetylation with acetic anhydride, were evaluated using polymer samples. These polymers were considered to be representative of the types frequently encountered in coating formulations. The reaction parameters of time, temperature, and sample size were optimized utilizing polyesters which contained primary and secondary hydroxyl groups. The optimum reaction conditions can be found in Table 6. Additional polymers analyzed utilizing these conditions and methods produced accurate and reproducible results for hydroxyl determinations. From this investigation, both the PMDA and pTSA methods can be recommended for use on a wide variety of polymeric materials for the determination of hydroxyl content. In addition, two viable methods are now available for verification of polymeric hydroxyl determination; results obtained from the method can be confirmed by utilizing the other method.

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Table 9-Comparison of Esterification Between Model Compounds and Polymer Standards

			Model Compounds ^a				Polymer Standards ^a			
Reagent	meq Anhydride Available In Reagent	meq 2,5-Hexane Diol	Deviation From Theoretical	meq Trimethylol- propane	Deviation From Theoretical	meq ^b Standard 1	Deviation From Theoretical	meq ^c Standard 2	Deviation From Theoretical	
PMDA		9 12	5% 5%	8 11	5% 5%	9 9	10% 3%	9 9	1% 29%	

(a) Optimum reaction time and temperature used.

(b) Standard 1 contains both primary and secondary hydroxyl groups

(c) Standard 2 contains only primary hydroxyl groups

work, K. K. Hesler and J. R. Lofstrom for their input concerning experimental designs, A. Tortorello for synthesizing the polymer standards, and A. M. Deihl for typing the manuscript.

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Federation of Societies for Coatings Technology

Society Meetings

BALTIMORE— VIRGINIA SECTION MAR.

"Rheological Additive"

It was announced that the current slate of officers for the 1982-83 year will maintain in their respective positions through the 1983-84 term.

"QUALITY AND CONSISTENCY FROM AN EASIER DISPERSING RHEOLOGICAL ADDITIVE" was discussed by Robert Dey, of NL Industries.

Mr. Dey reviewed the properties, advantages, and disadvantages of currently available organoclay additives for rheological control in solvent based paints and coatings.

The advantages of the new super dispersible organoclay rheological additive were then compared. Because of differences in the functionality of the exposed surfaces of the clay platelets in the new super-dispersible organoclays, the need for precise order of addition, polar activation and high shear required for older organclays is eliminated, explained Mr. Dey. The replacement of sodium ions with quaternary ammonium ions on the exposed clay platelet surface allows this easier dispersibility because the platelet structures formed are spaced at 40 angstroms, compared to conventional organoclay platelet spacing of 26 angstroms. The new clay also has a narrower distribution of platelet separation. The benefit to the paint manufacturer is more consistent results from the new organoclay in processing equipment with lower shear and higher throughput rates, said Mr. Dey.

MICHAEL J. BECKER, Secretary

C-D-I-C MAR. "Federation Visit"

Honored guests included Federation President A. Clarke Boyce, Executive-Vice-President Frank Borrelle, and Director of Meetings and Conventions Rosemary Falvey. Also in attendance were the officers of the Louisville Society: Vice-President—John Lanning, Porter Paint Co.; Secretary—W. J. Morris, of Celanese Plastic Specialties Co.; Treasurer—Edward Thomasson, of Louisville Varnish Co.; and Past-President—Phil Harbaugh, of Reliance Universal, Inc.

the Federation, explaining what it is all about, where the society fits in, and strongly emphasized the team effort within individual societies and in the leadership of the Federation. He encouraged participation in Federation activities, seminars, and research efforts. He stated that three ad hoc committees have been formed this year-one is writing the history of paint manufacturing with Joe Boatright of the Cleveland Society as the task group leader; Tom Miranda and his task group are studying reconstruction of educational activities; and Joe Bowers' task group is studying new ways of funding for the PRI.

President Boyce presented his views of

Mr. Boyce said his years in industry had taught him a lot about people, and he had a way of relating people to bone structure. He said there are a lot of wishful people always wishing things had happened differently; he calls this group the "wish bone people". Next, there are those who make a joke of everything, these are "funny bone people". Then, there are those who argue and fight about everything, these are "jaw bone people". Finally, there are the people who get the job done, these are the "back bone people"; and he said they are the kind of people who make up the Federation Societies.

Frank Borrelle covered the Spring meeting of the Federation Board of Directors to be held in Cincinnati at the Terrace Hilton Hotel. Two important items will be presented: (1) in reference to equal voting and office holding privileges for associate and active members, and (2) an increase in dues from \$15 to \$20.

A slide show about the Federation was presented. There are 6,800 members and an annual budget of \$1,300,000. Seven people serve on the Executive Committee and 23 on the Board of Directors. According to Mr. Borrelle, the money to operate is generated as follows: annual meeting and show 52%; publications 34%; memberships dues 7%; educational programs 2%; and miscellaneous 5%. The slide presentation covered the large list of publications, and Mr. Borrelle gave a brief description for each and the Society responsible for putting it together.

The upcoming seminar in Kansas City is a first to be held for the technical advisor of the Federation, and attendance was encouraged by Mr. Borrelle.

DAVID C. KINDER, Secretary

C-D-I-C APR.

"Polylsocyanates"

Ron Eritano, of Mobay Chemical Corp., presented a talk entitled, "COAT-INGS DEVELOPED AROUND THE POLY-ISOCYANATES."

Isocyanates and polyester resins have been used in the formulation of urethane coatings, explained Mr. Eritano. The basic chemical reactions in the formation of urethane polymers such as mondur and desmodur polyisocyanates resins were discussed.

Mr. Eritano explained that solvent based two component urethane coatings are based on two reactive components. Component 1 is the basis of the coating and consists of polyols, solvents, fillers, pigments, and catalyst. According to Mr. Eritano, Component 2 is the polyisocyanate resin which acts as the hardner or curing resin. In these formulations, only water free solvents such as esters, ketones, and chlorinated hydrocarbons can be used, stressed Mr. Eritano, Diluents used are tolnol and xylol. Solvents with primary or secondary hydroxyl groups are not utilized. In formulating, the isocyanate (NCO) and hydroxyl ratio is of importance in controlling the properties of the final coating.

Slides illustrated a laboratory study with a three-coat aluminum pigmented desmodur E-21 coating (7 mils) and a two-coat aluminum pigmented desmodur E-21 coating and top coated with an aliphatic urethane for a total of 7 mils along with an inorganic zinc/epoxy/urethane and an organic zinc/epoxy/urethane systems. The desmodur E-21 system performed very good, stated Mr. Eritano.

An educational program was presented by William Mirick and Robert Sharpe, of Battelle Laboratories. A film illustrated the proper paint procedures for covering asbestos ceilings.

DAVID C. KINDER, Secretary

CLEVELAND MAR.

"Microvoids in Organic Coatings"

Jerome A. Seiner, of PPG Industries, Inc., spoke on "MICROVOIDS IN ORGANIC COATINGS."

Mr. Seiner discussed several processes for placing microvoids in polymeric materials. He explained the microvoids' effects on properties of the materials, such as their effect on hiding and the changes in diffraction, refraction, and reflection.

RAYMOND PODLEWSKI, Secretary

LOS ANGELES MAR. "Past Presidents' Night"

In honor of Past Presidents' Night, President Romer Johnson recognized the following past presidents: Jan Van Zelm (1981-82); Don Jordon (1980-81); Albert Seneker (1979-80); Fred Croad (1978-79); Robert F. Koperek (1977-78); Gerald West (1976-77); Kenneth J. O'Morrow (1975-76); Robert A. McNeill (1974-75); William H. Ellis (1971-72); Duke Cromwell (1970-71); Trev Whittington (1969-70); Allan R. Yerby (1966-67); Albert M. Aronow (1964-65); Walter K. Barber (1963-64); Leonard S. Feldman (1960-61); Clyde L. Smith (1946-47); and Carl C. Howson (1930-31)

Membership Chairman Earl Smith presented a Paint Dictionary to Ruth Keirsey, the society's 600th member.

Lloyd Haanstra, Environmental Committee Chairman, reported on the February Task Force Meeting. A motion was passed at this meeting, according to Mr. Haanstra, to recommend to TRG the passing of 380 grams/liter for nonflat coatings to be extended to September 1986. The 14 exempted categories will be presented in upcoming workshops. Mr. Haanstra also reported that the funding for the Task Force will no longer be supplied by the State. Therefore, in order that the work of the Task Force be continued, industry will have to supply the necessary funds, said Mr. Haanstra.

Jim Elliott made a report explaining a mutual consent letter than can be used to explain any discrepancy in formulations when samples are picked up and tested by the Air Quality Board. This would result in a lower fine than would ordinarily be imposed, stated Mr. Elliott. He also explained that three products are being considered for exemption are not exempt to date. They are quick dry enamels, mill whites, and a special alkyd for fire and water damage.

The April Manufacturing Seminar, which will stress the use of present equipment to more adequately grind products, was discussed. Seven speakers will be present. Technical and production personnel are urged to attend.

President Johnson reported on a project to form a committee, possibly chaired by Trev Whittington, of Aseptic-Thermo Indicator, to write a textbook covering the paint course. This will be pursued at the next board meeting.

Educational Committee Chairman, James Hall reminded members of the scholarship funds available for eligible students (preferably, children of society members).

Ronald S. Bauer, of Shell Development Co., spoke on "RECENT DEVELOP-MENT IN EPOXY RESINS."

Dr. Bauer covered the recent advances being made in the area of new epoxy resin coatings which give higher performance systems, with water-based systems, high solids, and low temperature curing.

Q. What cycle of temperature/humidity/time did you use on the QUV exposure?

A. Eight hours at 50° C and 8 hours at 65° C (GM Cycle).

Q. What correlation can be made between QUV and exterior exposure on eponex vs. conventional epoxy?

A. Not very much correlation.

Q. Eponex type of resins were made to replace urethanes as a top coat. Has there been much success?

A. Eponex resins were not made to replace urethanes, but as an adjunct to our resin line. In the work done on the eponex resins it was found that gloss retention could be made that were as good as urethanes, or very close to the urethanes.

Q. Has the weatherable epoxy formulation equivalent given better or poorer color retention when compared to linear polyurethanes?

A. As far as color retention, results are about equivalent.

Q. What is the difference between epon and epoxy?

A. Epon is a trade name.

Q. Your resin eponex being the newest entry into the market place by Shell, why do you charge about \$30.00 for a gallon sample to evaluate it?

A. Samples are normally sent out free.

Q. What are the mechanism differences between kitamine H1, H2, and H3, when they react with epoxy?

A. The mechanism of reaction of epoxies with all the kitamines is essentially the same. It is the hydrolysis of the kitamine to give a primary amine to react with the epoxy to give a secondary amine and then a cured system or a tertiary amine.

MICHAEL GILDON, Secretary

NEW ENGLAND MAR.

"Microspheres in Coatings"

Environmental Chairman Thomas Manning gave an update on some new hazardous waste requirements.

"AN OVERVIEW OF MICROSPHERES IN COATINGS" was presented by William W. Loomis, of Zeeland Industries, Inc.

Mr. Loomis stated that there are different types of microspheres. There are organic microspheres which consist of hollow polymeric microspheres, and vesiculated beads types, and inorganic microspheres which consist of solid glass beads, hollow glass beads, thin walled, hollow ceramic microspheres, and thick walled hollow ceramic microspheres. All microspheres have certain properties in common, such as low vehicle demand and good flow. The many properties obtainable from specific microspheres are hiding, reduced weight, scrubbability, burnish resistance, abrasion resistance, hardness, chemical resistance, corrosion resistance, reflectance and cost reduction.

The types of architectural coatings in which common types of microspheres can be used are latex paints, said Mr. Loomis. You would try hollow polymeric microspheres, vesiculated beads, and thick walled hollow ceramic microspheres. In solvent based paints, you would try thick walled hollow ceramic microspheres. In OEM industrial baking finishes in high solids coatings, waterreducible coatings, coil coatings, general industrial finishes, and powder coatings. thick walled hollow ceramic microspheres are used. In heavy duty maintenance finishes, try solid glass beads, thin walled hollow ceramic microspheres and thick walled hollow ceramic microspheres. In marine coatings, solid glass beads, thin walled hollow ceramic microspheres, and thick walled hollow ceramic microspheres are used. In reflective coatings, try solid glass beads and thick walled hollow ceramic microspheres.

Several examples were discussed to explain the various properties that could possibly be obtained by using some of the above suggested microspheres.

CHARLES J. HOAR, Secretary

PHILADELPHIA MAR.

"Federation Officers Visit"

Honored guests attending the meeting were Federation President A. Clarke Boyce and Federation Executive Vice-President Frank Borrelle.

President Boyce discussed the Federation's availability to assist the societies. Both the societies and the Federation should work together as a team, stated Mr. Boyce. Ad hoc committee work was discussed which included: (1) the history of paint; (2) restructure of education booklets; and (3) PRI funds.

Mr. Boyce concluded his talk by thanking the society for its involvement in various projects in the past and by hoping for its future involvement.

Frank Borrelle gave a slide presentation on the objectives, services, and the benefits of the Federation and societies working together. Illustrated were how funds are obtained and disbursed. Also, programs which are available from the Federation were presented. Mr. Borrelle discussed how these programs could help the paint industry.

Concluding the talk were slides of the 1982 Federation Annual Meeting and Paint Industries Show in Washington, DC and the upcoming Montreal annual meeting and paint show in October.

WILLIAM GEORGOV, Secretary



Attending the March 16 meeting of the Piedmont Society are (left to right): Field Services Director Thomas Kocis; Piedmont Society President—Sara Robinson; and Federation President-Elect Terryl F. Johnson

PIEDMONT MAR.

"Federation Officer Visit"

Honored Federation guests included President-Elect Terryl F. Johnson and Director of Field Services Thomas Kocis.

Twenty-five year membership pins were presented to the following society members: Charles Roberts and William Younger, of DeSoto, Inc.; Lawrence Leonard, David Bouldin, and Clayton Sechler, of the Lilly Co.; William Shepard, of Reliance Universal, Inc.; Murphy Mull, of Murphy Mull Consultants; Grant Arhart, of W.C. Richards Co. of Carolina; William Warlick, of Warlick Paint Co.; and Jim Bohlen, of Sherwin-Williams Co. Other 25-year members not in attendance included: Richard Tuttle, of Guardsman, Inc.; Lee Myer, of Blue Ridge Talc Co.; Millard Firesheets, of Mobil Chemical Co.; and Linwood Carter and John Ladd, of Carolina Coatings, Inc. It was noted that Cecil Wilson, of Burks, Inc., is also eligible for a 25-year membership pin.

Awarded honorary membership in the society were: Jim Bohlen, Murphy Mull, and William Shepard.

President Sara Robinson announced that the society was looking for volunteers to serve on the new Environmental Committee as chairman and committee members.

President-Elect Johnson congratulated the society for its job in sponsoring the Polymer Chemistry course at UNC-G. He also urged the membership to participate in the Federation's seminar on the



Awarded 25-Year Membership Pins from the Piedmont Society are (left to right): Charles Roberts; Lawrence Leonard; William Younger; and Dave Boulding



25-Year Members of the Piedmont Society are (left to right): Murphy Mull; William Shepard; Grant Arhart; William Younger; Lawrence Leonard; James Bohlen; Clayton Sechler; William Warlick; and Charles Roberts

"Efficient Operation of an Up-to-Date Paint and Coatings Laboratory."

Mr. Johnson discussed the "Save the Paint Stone" project, which is being actively pursued by the Federation and the New England Society. The original Paint Stone is housed in a brick facing of the Boston Paint Stone Toy Shop not far from the waterfront, said Mr. Johnson. It is now designated a historical landmark and is under the protection of the Historic Preservation Society of Boston. Negotiation with the Society to come up with a plan to save the Stone is now underway, stated Mr. Johnson.

Thomas Kocis presented a slide-show highlighting the many activities of the Federation. Featured were: membership, which is about 6,800 (an increase of about 400 over the last five years); operating budget, which is projected at \$1,300,000 and established by the annual meeting and paint Show, sales of booth spaces, publication sales, sales of advertisements in these publications, and membership dues.

Mr. Kocis also discussed scholarship grants totalling \$18,000 and the six participating schools: University of Southern Mississippi; North Dakota State University; Kent State University; University of Detroit; University of Missouri—Rolla; and Eastern Michigan State University. Career promotional ideas are being developed to attract prospective students for the coatings industry, stated Mr. Kocis. He added that Federation Educational, Technical, and Manufacturing Committees meet annually to develop national programs to strengthen local efforts.

According to Mr. Kocis, the Corrosion Committee will sponsor a day-long seminar at the Annual Meeting and Paint Show.

An ad-hoc committee is now working to develop "A History of the Paint Industry", said Mr. Kocis. A complete manuscript, ready for publication, is tentative for 1985.

Mr. Kocis discussed the Paint Research Institute. Under the direction of Dr. Seymore Hochberg, the PRI is currently focusing on reducing the number of programs to highlight quality instead of quantity. PRI is also seeking another consortia to complement the one on mildew, and is hoping to find individuals interested in participating in this program of research on corrosion.

Mr. Kocis encouraged members to attend the Annual Meeting and Paint Show in Montreal, Canada, Slides illustrated the host city, the convention center, and the satellite hotels.

PHILIP WONG, Secretary

PITTSBURGH MAR.

"Iron Oxide Pigments"

The Educational Committee report concerning the Buhl Planetarium Science Fair was read by President-Elect Mike Gillen. It was agreed to donate the annual amount of \$150 to the science fair.

President William Cibulas made an announcement concerning the Cleveland Society's 26th Annual Technical Conference and urged attendance. He also urged the membership to attend the Federation's Seminar on the "Efficient Operation of an Up-to-Date Paint and Coatings Laboratory" to be held April 26–27. Also, Mr. Cibulas reminded the membership of \$18,000 available for 1983-84 scholarships.

"Relationships between Particle Size and Optical Properties of Iron Oxide Pigments" was presented by Rolf Odenthal, of Mobay Chemical Corp., Iron Oxide Div.

Dr. Odenthal pointed out that color shades of iron oxide pigments are related to particle size, going from vellowish to bluish over the range 0.1-0.7 µm. In masstone colors, lightness and saturation decrease as particle size increases. Curves for tint strength and opacity versus particle size pass through maxima, but peak at different particle size values. Particle size, gloss, and dispersion time show an interesting relationship for 5% PVC dispersions in a thermoset acrylic, said Dr. Odenthal. Gloss increases and haze decreases as dispersion time increases. The smaller the particle size, the greater the increase in gloss, but medium sized particles actually gave the least haze.

The difference between regular and micronized grades of iron oxides was discussed by Dr. Odenthal. Micronizing consists of dry jet milling which affects particle shape, reduces primary particle size, and reduces oil absorption by about 50%. The advantages of micronized oxides are that they impart greater gloss and lower haze than do high oil absorption grades.

Dr. Odenthal presented results of gloss retention and chalking studies based on both accelerated weathering and outdoor exposure. He found that gloss dropped off more rapidly with larger particle size iron oxides and that chalking eventually occurred. The degree of chalking was determined by applying an adhesive tape to the surface and removing loose (chalked) material with the tape, then determining the amount removed. The smaller the iron oxide particle size, the greater the degree of chalking. However, according to Dr. Odenthal, the relationship is not linear and blueish (coarser) grades chalk faster than would be expected. This is because the coarser iron oxides give higher UV transmittance than do the finer ones and binder degradation is accelerated. Chalking also varies with PVC. Starting at low PVC, chalking increases to a point ($\sim 10-15\%$ PVC), then decreases as the increased amount of pigment acts as a UV barrier and slows UV degradation. Chalking hits a minimum, then rises sharply as the PVC approaches critical. The increased UV degradation is due to the fact that the pigment no longer is completely enveloped by the vehicle, stated Dr. Odenthal.

So far. Dr. Odenthal had assumed an ideal situation with no flocculation, but that this was not really possible. In an ideal paint or dispersion, the iron oxide particles would be homogeneously scattered through the mixture with a certain distance between each particle. In reality, there would be loose agglomerates of pigment with some areas being completely empty of pigment, whereas others would be full of pigment. The agglomerates do not act merely as large particles, however. Color changes are less than one would expect from agglomerate size, although with heavy flocculation there is a definite shift toward blue.

According to Dr. Odenthal, flocculation depends on the binder system as well as the pigment. He described experiments with flat and semigloss latexes and a long oil alkyd where the degree of difference in color produced with two pigment grades varied considerably with the vehicle used. He also described some work with a series of alkvd-based red iron oxide dispersions where color as a function of PVC varied considerably. In dispersions where a high degree of flocculation occurred, regardless of pigment concentration, color intensity was low and constant (independent of PVC) above the critical region. However, where flocculation was not evident at low pigment levels, but occurred and increased with increased pigment loading, color intensity dropped in a smooth curve as PVC increased. Again, the degree of flocculation was dependent on the binder system. Therefore, optical properties of iron oxide pigments are dependent on their environments as well as particle size, concluded Dr. Odenthal.

Q. Does chalking give shifts in color? A. Yes, there is a shift when the gloss changes (index of refraction changes). For example, yellow iron oxide pigmented films shift to lighter shades.

Q. How would you choose a yellow iron oxide to minimize color shift?

A. Start out with a lighter shade. Yet this is not really a practical answer as the formulator will choose according to the color he wishes to start with, not the one that will show the least shift.

Q. You say that iron oxides tend to flocculate?

A. Yes, more so than we expected even with yellow iron oxide which normally looks good in rubouts.

Q. Is any research being carried out that is directed at producing less flocculated iron oxides?

A. Yes, the work described in this talk is merely the first step in characterizing iron oxides. We now are looking at surfactants, surface treatments, etc. to improve wetting, reduce flocculation. We are just at the beginning of the development of new pigment grades. It should be pointed out that the test procedures discussed here relate more to low PVC industrial coatings. We have not yet worked with high PVC trade sales coatings.

CLIFFORD SCHOFF, Secretary

PITTSBURGH APR.

"Solvent Option for Air Quality"

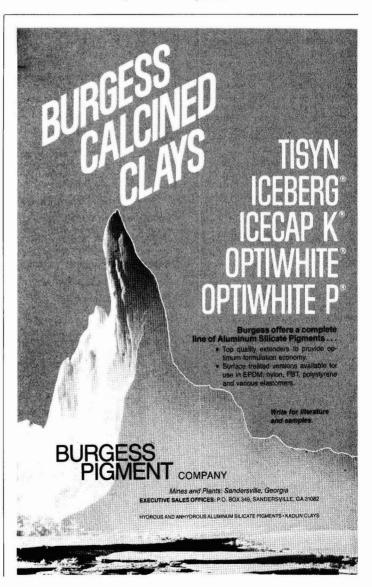
Co-chairman of the Educational Committee Patricia Dawson reported that the society had awarded two prizes in the Buhl Planetarium Science Fair. Unfortunately, only five or six projects qualified for consideration, because so few projects were actually related to coatings. Most were concerned with corrosion inhibition of paints. Ms. Dawson urged that coatings and the coatings industry be better presented to local high school students and teachers.

"THE SOLVENT OPTION FOR AIR QUALITY COMPLIANCE" was discussed by Raul Oteiza, of Dow Chemical Co., Inorganic Chemicals Department.

Mr. Oteiza discussed the use of methylene chloride and 1,1,1-trichloroethane to produce coatings that are in compliance with federal and state clean air laws and regulations. He described the current regulation system which is a consequence of the Clean Air Act. The Federal Government sets guidelines through the EPA and the states write laws implementing the guidelines, said Mr. Oteiza, Included in these laws will be quality standards for major pollutants, one of which is ozone which is produced from organic vapors by photochemical processes. Therefore, the regulation of ozone depends on the regulation of volatile organic compounds (V.O.C.'s), particularly organic solvents. It turns out that all common processing solvents undergo photochemical reactions to produce ozone. There are a few materials that do not, however, and these are classified as NON-V.O.C.'s. Three of these (methane, ethane, and Freon 113) are not useable as paint solvents, but two of them (methylene chloride and 1,1,1-trichloroethane) have considerable potential for use in paints. Mr. Oteiza pointed out that over 70% of the states exempted methylene chloride and over 80% exempted 1,1,1trichloroethane, and that these numbers were increasing.

The options that a formulator has for reduction of organic volatiles were explained by Mr. Oteiza. One possibility is engineering: absorbing or incinerating volatiles. Another possibility is new technology: waterborne, high solids, powder, and radiation cure coatings. A third option, and the one Mr. Oteiza favors, is the use of compliance solvents so that current solvent borne technology can be used. The only difference is the solvents. Partial or complete replacement of current solvents by methylene chloride and/or 1,1,1-trichloroethane is necessary under this option.

Mr. Oteiza then discussed the properties of methylene chloride (MC) and 1,1,1-trichloroethane (1,1,1-T). Some people are very wary of chlorinated solvents due to the toxicity of certain members of that family. Therefore, Mr. Oteiza addressed the question of toxicity of MC and 1,1,1-T. These two solvents are the safest of the chlorinated compounds. Data so far indicate no risk to



humans. Allowable exposures (time weighted averages) are 500 ppm for MC and 350 ppm for 1,1,1-T. For comparison, values for certain other solvents are toluene 100 ppm, MEK 200 ppm, methanol 200 ppm, and carbon tetrachloride 10 ppm. The conclusion to be drawn is that the two compliance solvents are very safe.

Mr. Oteiza mentioned the issue of whether evaporation of MC and 1,1,1-T would eventually deplete the stratospheric ozone layer which protects us from UV and other radiation. He pointed out that although these solvents showed negligible photochemical reactivity, they did not have long lives in the atmosphere. Long before they reach the stratosphere they degrade and, therefore, are not a problem.

With heat and pressure, halogenated hydrocarbons can produce corrosive HCl, particularly in the presence of freshly abraded aluminum. For this reason aluminum pumping, spraying, and handling equipment should not be used with them, stressed Mr. Oteiza. Field problems have been very rare, but it is possible to get an exotherm and high pressure leading to an explosion. Since it is not always clear whether equipment includes aluminum parts, a Halo Hydro Carbon Finishing Equipment Information Center (HHCFEIC) has been set up so that a person can find out whether his current equipment is acceptable. The number is 800-221-8300.

The cost effectiveness of chlorinated solvents, which are more expensive than conventional ones, was explained by Mr. Oteiza. He pointed out that such solvents could save money by allowing the formulator to achieve compliance without going to entirely new technology. In addition, complete replacement is rarely necessary in order to gain compliance. Other reasons for using MC and 1,1,1-T are their lack of flash points, their low toxicity, fast evaporation rates, and the good viscosity stability they impart to products. One drawback to these solvents is that they are not very good solvents for many resins. However, solvency can be achieved by using chlorinated solvents as cosolvents along with other, more active solvents, recommended Mr. Oteiza.

Chlorinated solvents are good for coatings that need to dry rapidly, stated Mr. Oteiza. For example, MC is used in flash dry traffic paints, aerosol paints, and very fast dry lacquers. 1,1,1-T is used in pipe coatings, high speed gravure inks, certain lacquers and enamels, and some wood stains and varnishes.

Mr. Oteiza concluded that use of compliance solvents allows a formulator to meet air pollution requirements and still continue to use proven solvent-borne technology. Q. What is the change of an explosion using chlorinated solvents with an aluminum pressure pot or other aluminum equipment?

A. Pressure increases the corrosivity of the solvent and, if it comes into contact with a fresh aluminum surface (through abrasion, for example), there is a rapid reaction which produces pressure from HCl and heat. This combination could lead to rupture. However, there are only two documented incidents of this type.

Q. Are there any criteria for the pressure and temperature that will cause a rupture or explosion with aluminum equipment?

A. The solvent must decompose before this can happen. Decomposition requires very high heat and pressure. A pump possibly can give this (if fresh aluminum is exposed), but the two variables of heat and pressure have to be just so for this to have any probability of happening.

Q. What are the four states where these solvents are not exempt?

A. Michigan, Kentucky, New Jersey, and Rhode Island.

Q. Do these solvents degrade to HCl? What about phosgene?

A. There is very little chance that phosgene will be produced and, if solvent is kept out of the oven flame, very little HCl will be produced.

CLIFFORD SCHOFF, Secretary

ROCKY MOUNTAIN MAR.

"Developments in Epoxy Resins"

Educational Chairman Steve Crouse, of Kwal Paints, Inc., discussed an upcoming meeting at his company to gather ideas on increasing coatings knowledge. Mr. Crouse also mentioned a steering meeting in Detroit, Ml on April 15 for the Educational Committee. A total of \$18,000 in scholarship money is available for the 1983-84 academic year, said Mr. Crouse, which is divided among six universities and is available to children of Federation members.

Paige O. Switzer, of Oil & Solvent Process Co., announced that BF1 and OSCO will present a joint briefing to Colorodo Industry on BF1's proposed Adams County Chemical Waste Treatment and Disposal Facility and on the recycling processes available for certain chemical wastes.

Dr. Ron Bauer, of Shell Development Co., presented a talk entitled, "RECENT DEVELOPMENTS IN EPOXY RESINS."

LUIS O. GARCIA, Secretary

ST. LOUIS MAR. "High Solids"

Vice-President Robert Giery discussed a tentative theme of "High Technology— Theme for the Eighties" for the June 10th joint meeting with the Kansas City Society.

Robert S. Bailey, of Lilly Industrial Coatings, spoke on "Opportunities in Coatings."

A moment of silence was observed for Marcey Gilbam, a Past-President of the St. Louis Society, who passed away March 13.

"HIGH SOLIDS" was discussed by Roger Senn, of Freeman Chemical.

Mr. Senn spoke about coating vehicles and explained Rule 66. He evaluated new air pollution rules such as VOC, powder coating, etc. A list illustrated VOC's for different industries.

Reactive diluants and water-borne coatings were discussed. Mr. Senn also listed the many high solids coating resins which included: short alkyds; medium alkyds; chain stopped alkyds; oil-free alkyds (polyesters); phenolic modified alkyds; epoxy esters; and acrylic alkyds. Formulas and formulations were discussed.

Conventional type resins, which can be cut into stronger solvents to lower viscosity, were explained.

Problems in high solids and possible solutions were also highlighted by Mr. Senn. These included: (1) telegraphing of heated metal—add some higher molecular weight material such as epoxy, acrylic, etc.; (2) recoatability—methylated melamines worse that butylated types; (3) corrosion resistance—use of manganese oxide seems to work well at 3% of pigment: (4) low gloss finishes—use enough silica to get 20% gloss or so and adjust with synthetic silica; and (5) low VOC and leveling—use small amounts of high solids medium oil alkyd.

Q. Have you tried MgO3 over steel and aluminum?

A. Yes, we have tried MgO₃ over steel and galvanized, but not over aluminum.

Q. How does high molecular weight improve telegraphing?

A. Raising average molecular weight helps to cover metal prep streaks.

Q. Has there been any further breakthroughs in air dry to reach VOC's.

A. Nothing looks new at this time but try blending in some high solids medium oil to help.

WILLIAM A. TRUSZKOWSKI, Secretary

Elections

BIRMINGHAM

Active

- GAGG, PETER Blundell Permoglaze Ltd., Birmingham, Eng.
- HURST, ERIC J.- Ciba Geigy P & A Co., Goodstrey Crewe, Eng.
- WARSON, HENRY- H. Warson (Chemical and Consultant) Ltd., Solihull, Eng.

DALLAS

Active

ANDREWS, TOM A. -- Koppers Co., Irving, TX.

DETROIT

Active

- BLOM, BRUCE E. Flint Ink Corp., Detroit, MI.
- FREEMAN, ROBERT LEE Silverlead Paint Co., Lansing, MI.
- GIERKE, MARK A. DeSoto Inc., Westland, MI.
- MASLOVICH, WALT Wyandotte Paint Products Inc., Troy, MI.
- MAUL, A. RUDOLPH DeSoto Inc., Southfield, MI.
- PERRY, KENNETH M. Glasurit America, Inc., Detroit.
- PERRY, PAMELA-Wyandotte Paint Products Inc., Troy.
- SAVAGEAU, ROBERT G. Flint Ink Corp., Detroit.
- TEUSCHER, ED--Wyandotte Paint Products Inc., Troy.

Associate

- LEMONS, DAVID M.- Union Carbide Corp., Milford, OH.
- STENDEL, PHILIP-Profit Systems, Warren, MI.

LOS ANGELES

Active

- BOLDUC, W.J.-Solvent Coating Corp., Torrance, CA.
- DIMAIO, RAY-Koppers Co., Inc., Los Angeles, CA.
- DREWS, JERRY-Koppers Co., Inc., Los Angeles.
- FENSTERMAKER, EARL Conklin-Fenstermaker, Inc., Montclair, CA.
- KAMMERER, HELMUT J. TW Graphics Group, City of Commerce, CA.

- KEIRSEY, RUTH-Dexter Hysol Div., City of Industry.
- POLLAK, RICHARD B.—Lee Pharmaceuticals, South El Monte, CA.
- WONG, KA-CHUN- Amertone Paint Corp., Long Beach, CA.

Associate

- BACKLIN, ROBERT- Tenneco Chemicals, Inc., Buena Park, CA.
- CLARK, STEVE Dow Chemical Co., Pasadena, CA.
- COPPER, ARTHUR-Cooper Drum Co., South Gate, CA.
- DANIEL, DAN G. Cooper Drum Co., South Gate.
- LOUGHREY, WAYNE F.- Ciba-Geigy Corp., Orange, CA.
- MILIANO, ROBERT-Gila River Products, Chandler, AZ.
- RASMUSSEN, THOMAS E.— Harrisons & Crosfield, Inc., Thousand Oaks, CA. SIMMONS, SPENCER L.— 3M Co., Los Angeles.

Retired

SUMERS, ROBERT W.-Orange, CA.

NEW ENGLAND

Active

MCCAULEY, PATRICK H. – The Dampney Co., Everett, MA.

Associate

- FERRI, ROBERT J.—Sun Chemical Corp., Cranston, RI.
- MALUCHNIK, DAVID T.-NL Chemicals, Duxbury, MA.

PACIFIC NORTHWEST

Associate

GARDINER, J. STEPHEN-Shell Canada Chemical Co., Vancouver, B.C., Canada. PRINCE, DAVE-Nuodex Canada Ltd., Delta, B.C., Canada.

PITTSBURGH

Active

- ABATE, KENNETH-H.H. Robertson Co., Ambridge, PA.
- ADAMS, DAVID J.-PPG Industries, Inc., Springdale, PA.
- BAKER, JOHN G.-PPG Industries, Inc., Springdale.
- GROSS, ROBERT S.-PPG Industries, Inc., Springdale.
- TRAISTER, ANDREW-PPG Industries, Inc., Springdale.

Associate

- HITCHCOCK, ROGER A.-FBC Chemical Corp., Mars, PA.
- O'KEEFE, JOHN J. JR., -- Mooney Chemicals, Inc., Cleveland, OH.

WESTERN NEW YORK

Active

KIRBY, PATRICK N.—Pratt & Lambert, Inc., Buffalo, NY. PALLADINO, WILLIAM J.—Conap, Olean, NY.

FSCT Membership Anniversaries

25-YEAR MEMBERS

Montreal

- George Campbell-Sherwin-Williams Co. of Canada Ltd.
- Richard A. Glegg-Sherwin-Williams Co. of Canada Ltd.
- James Haggerty—Maple Leaf Monarch Co. Walter Kolanitch—Sherwin-Williams Co.
- of Canada Ltd.

Piedmont

- Grant E. Arhart-W.C. Richards Co. of Carolina.
- James A. Bohlen-Sherwin-Williams Co. David L. Bouldin, Jr.-Lilly Co.

- Linwood T. Carter-Carolina Coatings, Inc.
- Millard M. Firesheets-Mobil Chemical Co.
- John A. Ladd, Jr.-Carolina Coatings, Inc.
- Lawrence S. Leonard—Lilly Co. Lee J. Meyer—Blue Ridge Talc Co.
- Murphy E. Mull-Murphy Mull Con-
- sultants.
- Charles J. Roberts-DeSoto, Inc.
- Clayton S. Sechler-Lilly Co.
- William B. Shepard-Reliance Universal, Inc.
- Richard M. Tuttle-Guardsman Chemical Coatings, Inc.
- William Warlick-Warlick Paint Co.
- William O. Younger-DeSoto, Inc.

Meetings/Education

KSU Schedules Rheology Course June 20–24

The Rheology and Coatings Laboratory of Kent State University's Chemistry Department will sponsor "Applied Rheology for Industrial Chemists" short course, June 20–24 at its campus in Kent, OH.

The session will deal with general concepts, the rheology of viscoelastic fluids, dispersions, deformable particles, gels, high solids, high molecular weight additives, and brittle materials. Also included in the session is the discussion of rheological instruments, the rheology of application by brush and roller, and the rheology encountered by liquid-to-solid conversion of coatings.

The course will include the following presentations:

Monday, June 20

"Basic Concepts of Rheology I, II, and III"—Hershel Markovitz, Carnegie-Mellon University.

"Measuring Techniques for Viscoelastic Properties of Liquids"—Raymond R. Myers.

Tuesday, June 21

"Rheology of Dispersions"—Irvin M. Krieger, Case Western Reserve University.

"Application of Rheology to Process Problems"—Joseph M. Starita, Rheometrics, Inc.

"Application of Rheology to End Use Performance Problems"—Joseph M. Starita.

"Rheology Control for High-Solids Coatings"—Clifford Schoff, PPG Industries, Inc.

Wednesday, June 22

"Brush Application of Coatings"—J. Edward Glass, North Dakota State University.

"Roll Application of Coatings"-J. Edward Glass.

"Application of Rheology in Petroleum Recovery Processes"—J. Edward Glass.

"Correlation of Film Morphology with Drying Behavior"—John W. Vanderhoff, Emulsion Polymers Institute, Lehigh University.

Thursday, June 23

"Rheology of Deformable Particles and of Gels"-Edward Bagley, U.S. Dept. of Agriculture.

"Chemorheology of Thermosets"--Richard R. Eley, Glidden Coatings & Resins Div., SCM Corp.

"Kinetic Rheology Applied to Liquidto-Solid Conversion"—Raymond R. Myers.

"Flow Fundamental in Fluid Systems of High Molecular Weight Additives"— Donald L. Hunston.

Friday, June 24

"Fracture Behavior of Brittle Materials"—Donald L. Hunston.

"Application of Rheology to Industrial Problems"--- Donald Bigg, Battelle-Columbus Laboratories.

Personnel in research and development interested in coatings, adhesives, inks, elastomers, and related areas would benefit from the course.

Additional details are available from Program Chairman Carl J. Knauss, KSU, Chemistry Dept., Kent, OH 44242.

Dallas and Houston Societies Hold Southwestern Convention

Two hundred and forty persons attended the 40th annual Southwestern Paint Convention in Dallas, April 13-15. Theme of the meeting was "Accentuate the Positive."

The technical program featured the following presentations:

(1) "Bunte Salts as Crosslinking Agents in Thermosetting Water-Borne Polymers"—Andrew Voda; and "Oligomers of Butadiene and Isoprene"—Dan Rogers. (Student papers from the University of Southern Mississippi).

(2) "Non-Pigmentary Hiding: Opacifying Polymer"—John Stauffer, of Rohm and Haas Co.; "Encapsulair Beads"— Wally Krason, of Encapsulair, Inc.

(3) "Emulsion Polymerization"—Dr. Ben Kine, of Rohm and Haas Co.

(4) "Surface Lubricity"—Warren Pushaw, of Micro Powders, Inc. (5) "Micro Computers and Paint"-William R. Coughlin, Consultant.

(6) "Waste Management Update"-Ken Walanski, of DeSoto, Inc.

(7) "Economic Forecast"—Bob Carson, of Eastman Chemical Products, Inc.

The General Chairman of the SWPC was Joseph S. Jedrusiak, of Cron Chemical Co. He was assisted by: William Wentworth (Society President), of Jones-Blair Co.; Ross Frank; William F. Holmes, of DeSoto, Inc.; Leon Everett, of Dan-Tex Paint & Coatings; Ray Marett, of Western Specialty Coatings Co.; Richard and Elsie Williamson, of Trinity Coatings Co.; Carlos E. Dorris, of Jones-Blair Co.; Joe McDaniel; Bob Pugh; and Art Adams, of Delta Solvents & Chemical Co.

The 1984 Southwestern Paint Convention will be held at the Shamrock Hilton Hotel, Houston, April 12-14.

CALL FOR PAPERS Cleveland Society's 27th Annual Technical Conference "Advances in Coatings Technology" April, 1984

The Education Committee of the Cleveland Society for Coatings Technology welcomes papers on all aspects of coatings science and engineering, including—but not limited to formulation, testing, application, and characterization.

Preliminary abstracts should be submitted by August 15, 1983, to Chairman Richard R. Eley, Glidden Coatings & Resins Div., SCM Corp., 16651 Sprague Rd., Strongsville, OH 44136.

Emulsion Polymers Course Scheduled for August 15–19

The sixth annual short course entitled, "Advances in Emulsion Polymerization and Latex Technology, will be held August 15-19 at the Schatzalp Berghotel, Davos, Switzerland.

Designed as an in-depth study of the synthesis, characterization, and properties of high polymer latexes, the course will feature a balance of theory and practical problems. Lectures will begin with introductory material and reviews and will progress through recent research results.

The session is designed for engineers and scientists who are actively involved in emulsion work as well as for those who wish to develop expertise in the area. A basic background in chemistry will be assumed. More advanced and experienced participants may elect to attend only those days in which material of specific interest is being presented. All participants will receive a set of course notes for the lectures attended.

Topics scheduled for presentation are as follows:

Monday, August 15

"Adsorption of Surfactants, Electrostatic Stabilization and Flocculation of Latexes."

"Emulsion Polymerization Mechanisms and Kinetics."

"Mixing Scale-Up in Emulsion Polymerization."

"Inverse Emulsion Polymerization."

Tuesday, August 16

"Experimental Methods for the Characterization of Latex Particle Size and Particle Size Distribution."

"Adsorption of Polymeric Molecules and Steric Stabilization of Latexes."

"Continuous Emulsion Polymerization."

"Applications of Latexes: An Overview."

Wednesday, August 17

"Surface Characterization of Latexes." "Latex Rheology."

"Preparation of Latexes by Direct Emulsification."

"Semi-Continuous Emulsion Polymerization."

Thursday, August 18

"Carboxylated Latexes: Preparation and Characterization."

"Vinyl Acetate—Butyl Acrylate Copolymers: Preparation and Characterization."

"Coating by Electrodeposition."

"A Guide for Latex Formulation."

Friday, August 19

"Core—Shell Morphology." "Latex Film Formation and Drying of Latex Films."

Course organizers include Dr. Gary W. Poehlein, Professor of Chemical Engineering, Georgia Institute of Technology; Dr. John W. Vanderhoff and Dr. Mohamed S. El Aasser, Professor of Chemistry and Professor of Chemical Engineering, respectively, and co-directors of the Emulsion Polymers Institute, Lehigh University.

Registration fee is \$575 for the entire five days or \$175 per day for any portion of the course attended.

Further information can be obtained from Dr. Gary W. Poehlein, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332.



Printing Inks Course to Be Held August 22-26 in Switzerland

The second annual short course entitled, "Physics and Chemistry of Printing Inks," will be held August 22-26 at the Schatzalp Berghotel, Davos, Switzerland.

The session is a broad study of the preparation, characterization, and application of printing inks, and the printing processes in which they are used. The subject matter comprises a balance between the underlying theory and the practical application, as well as the physical and chemical problems in the preparation and application of printing inks.

Designed for scientists, engineers, pro-

duction, technical service, and marketing personnel who wish to develop a background in printing inks and printing processes, the course will present the following topics:

Monday, August 22

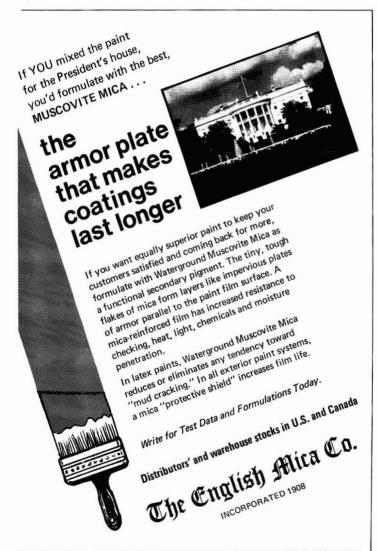
"Introduction to Printing Inks and Printing Processes."

"Identification of Prints."

"Ink Transfer and the Splitting of Ink Films."

"Preparation and Evaluation of Laboratory Films."

"Surface Chemistry of Lithography."



HIGHWAY 216 S., P. O. BOX 709, KINGS MOUNTAIN, N.C. 28086, TEL (704) 739-1321

Tuesday, August 23

"Compatibility of Polymer Vehicles and Solvents, and Formulation Using the Solubility Parameter Concept."

"Pigment-Binder Geometry in Formulation of Printing Inks."

"Stabilization of Fine Particles in Aqueous and Non-Aqueous Media."

"Dispersion of Pigments." "Pigment Flushes."

Wednesday, August 24

"Measurement of Degree of Dispersion."

"Rheology of Printing Inks."

"Measurement of Ink Viscosity and Tack."

"Composition of Printing Inks."

Thursday, August 25

"Principles of Color."

"Application of Color Theory to Print-

ing Inks."

"Computer Color-Matching."

"Types of Colorants."

"Gloss of Printed Ink Films."

Friday, August 26

"Printability." "Solventless Inks."

Fee for the course is \$575 for the entire week or \$175 per day for any part.

Further information is available from Dr. Mohamed S. El-Aasser, Dept. of Chemical Engineering, Sinclair Lab #7, Lehigh University, Bethlehem, PA 18015.

ACS Announces Color Control Seminars

Applied Color Systems, Inc., Princeton, NJ, has announced its 1983 series of three-day seminars in Color Control Technology. The first session is scheduled for June 29-July I at the Landmark Hotel, Myrtle Beach, SC. Three additional sessions are scheduled for: August 17-19 at the Wequasett Inn, Cape Cod, MA; September 17-19 in Atlantic City, NJ; and December 12-14 at the Disneyworld/Epcot Center, FL.

Conducted by Wes Coppock, ACS Director of Educational Services, the courses will cover color theory, practical application of color control in the laboratory and production environment, and the economics and cost justification of color control.

The sessions are designed to provide management and technical personnel with a fundamental knowledge of the basic problems of industrial color control.

Fee for the session is \$295.

For information, contact ACS, Inc., P.O. Box 5800, Princeton, NJ 08540.

OCCA Australia Publishes Surface Coatings Textbook

The Oil and Colour Chemists' Association Australia has prepared a textbook in two volumes covering the subject of surface coatings.

The first volume, consisting of 30 chapters, deals with raw materials and includes a paint glossary of terms. It will be available in early 1983.

The second volume discusses formulating principles, paint manufacture, and types of coatings systems in 28 chapters. It will be available in late 1983.

The volumes were written by Australian industry experts and the books have been technically edited, edited for continuity, metricated, and indexed.

Cost of the volumes is \$29.95 (Australian) [\$28.15-U.S.] per copy, plus postage.

Following is a list of chapter titles:

VOLUME 1

Past and Future of the Surface Coatings Industry

Introductory Polymer Science (Applicable to Surface Coating Technology.)

Vegetable Oils Oleoresinous Media Introduction to Alkyd Technology Manufacture of Alkyd Resins Application of Alkyd Resins Polyesters Amino Resins Phenolic Resins Polyurethane Resins Epoxy Resins Water Dispersible Epoxy Resins Silicone Resins Acrylic Solution Resins Emulsion Polymerisation Theory Emulsion Properties (1): Effect of

Monomer Composition Emulsion Properties (2): Effect of

water phase and particle size

Emulsion Properties (3): Film Formation

Emulsion Polymers: Manufacture and Testing

Applications of Emulsion Polymers (with particular emphasis on surface coatings)

Water Reducible Resins Water Soluble Resins Solvents Inorganic Pigments Titanium Dioxide Organic Pigments Extender Pigments Paint Driers Paint Additives

VOLUME II

Colour Matching (Using Computerised Techniques)

Testing and Quality Control

Substrates and Preparation

Metal Cleaning and Pretreatment Selection of Decorative Paints

Corrosion and Prevention

Corrosion and Prevention

Industrial Paint Application and Curing

Defects in Surface Coatings and Paint Removal

Analysis of Polymeric Materials in Coatings

Technical Service

Computers in the Surface Coatings Industry

Standardization, Inspection and Accreditation in Australia

The Paint Industry—Statutory Requirements

Rheology

Formulation Principles

Paint Manufacture-Pigment Grinding

Paint Manufacture—Processing Operations

Architectural Coatings

Heavy Duty Protective Coatings

Antifouling Paints

Industrial Coatings

Automotive Coatings

Lacquers

Conversion Coatings

Powder Coatings

UV Radiation Cured Coatings

Printing Inks

Components & Technical Aspects of a Paint Tinting System

For more information, or to order, please contact Mr. Brian J. Lourey, Federal President, OCCAA, c/o British Paints (Aust.) Pty Ltd., P.O. Box 43, Bankstown, N.S.W. 2200. Australia

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People

John C. Weaver, Consultant and Lecturer in Case Institute of Technology's macromolecular science department, and former Research Director of Sherwin-Williams Company's Coatings Group, was presented with the Cleveland Technical Societies' Distinguished Service Award. Dr. Weaver was recognized for his contributions to engineering, for achieving prominence in his profession, and for his civic leadership.

After earning a B.S. in Chemistry from Denison University and the M.A. and Ph.D. from the University of Cincinnatti, Dr. Weaver was a teaching assistant at the University of Cincinnati from 1930 to 1935. He then worked as a chemistry instructor until 1936, when he joined Sherwin-Williams. During his 38-year career with the company, Dr. Weaver served as a supervisor in the varnish laboratory and as coordinator of research development, technical service, manufacturing, and foreign technologies. In 1968, he was appointed research director of the coatings group, and served in that position until his retirement in 1974. He continued to serve the company as a consultant until 1979.

Dr. Weaver has been active in both the Federation of Societies for Coatings Technology and the Cleveland Society. He has served as Chairman of the Federation's Standards Committee, Research Advisory Committee, Heckel Award Committee, and the Publications Committee, serving also as Technical Editor of the Journal of Paint Technology. In 1969, Dr. Weaver was honored with the Federation's George Baugh Heckel Award.

A member of the American Society for Testing and Materials, Dr. Weaver has been active on its Committee D-1 on Paint and Paint Materials, serving as both its Chairman (1946-1960) and its Vice-Chairman (1960-present). He has also been involved in Committee D-1's varnish subcommittee and its miscellaneous subcommittees, and has served as its liaison via ANSI to ISO/TC 35 on Paint and Paint Materials. Dr. Weaver received ASTM's Award of Merit.

Cleveland Technical Societies also awarded Scott E. Rickert, Associate Professor of macromolecular science at Case Western Reserve University, with its Technical Achievement Award, for his outstanding performance, creative ability, and technical competence.

Dr. Rickert is a member of the Cleveland Society and was presented with its Outstanding Speaker Award.









J.C. Weaver

M.F. Dante

T.G. Brown

R.C. Albrecht

Dr. Mark F. Dante has joined Hempel Technology, Inc., Houston, TX, as a Senior Research Associate. He will be responsible for research in industrial and marine coatings and report directly to General Manager, Dr. C. Malcolm Hendry.

Prior to joining Hempel, Dr. Dante was employed for 28 years in coatings research specializing in the application of epoxy resins and solvents in the coatings industry. During this time, his work has resulted in several patents and publications.

Dr. Dante is a member of the Houston Society and serves as its Educational and Publicity Chairman. He is also a member of the American Chemical Society and serves as an adjunct instructor in the Technical Education Division of Houston Community College.

PPG Industries, Inc. has named **Roger M. Christenson** and **Marco Wismer** as charter members of the company's newly established Technical Collegium in recognition of their outstanding, sustained contributions to the firm's success. Mr. Christenson serves as Director of Research, Coatings & Resins Div., and Mr. Wismer is Vice-President of Research and Development, Coatings & Resins Div. Messrs. Christenson and Wismer are Pittsburgh Society members.

Also, the firm's Coatings and Resins Div., has appointed **Samuel Porter** to the position of Manager, Original Finish Automotive Coatings, in its Research and Development Department, Allison Park, PA.

Roger F. La Londe has been promoted to the newly created position of Assistant to the international sales manager for the Bareco specialty waxes group of Petrolite Corp. Mr. La Londe recently served as a sales representative for the firm. Zeelan Industries, Inc., St. Paul, MN, has appointed **Thomas G. Brown** as President. Mr. Brown has over 20 years experience in the pigment, chemical, and coatings industries. He joined Zeelan in 1982 as Director of Sales. Prior to that appointment, he held a variety of administrative, marketing, and research management positions with Engelhard Minerals and Chemical, Corp., M.A. Bruder & Sons, Inc., and E.I. duPont de Nemours & Co., Inc. Mr. Brown is a member of the New York Society.

Ace Hardware Corp., Oak Brook, IL, has made several new appointments for its first paint manufacturing center now under construction in Matteson, IL.

Rudolph C. Albrecht has been named Technical Director. Prior to his appointment, Mr. Albrecht was employed by The Enterprise Co. as Supervisor of Trade Coatings. He has also served as Supervisor of Special Products at Standard T. Chemical Co. from 1962–77; as Chief Chemist at Seymour of Sycamore from 1959–62; and as Paint Formulator at United Wallpaper from 1957-59.

Mr. Albrecht has been active in the Federation of Societies for Coatings Technology and currently serves on its Board of Directors. He has also been active in the Chicago Society, serving in the offices of Treasurer through President from 1974-78. He has served the society in its technical, educational, program, nominating, and executive committees. Mr. Albrecht was awarded the Society's Outstanding Service Award in 1980.

Other appointments announced were: John J. Hanacek was named Research Chemist; Steve A. Hodges was appointed an R&D Chemist; and David H. Stewart was named Field Engineer.

Zeno W. Wicks, Jr., of NDSU, to Retire; Frank N. Jones Named as Replacement

Dr. Zeno W. Wicks, Jr., Chair of the Department of Polymers and Coatings at North Dakota State University, Fargo,



ND, since July 1972, will retire effective June 30. He was appointed Professor Emeritus of Polymers and Coatings effective July I. Dr. Wicks came

to NDSU with nearly 30 years of industrial experience, including

positions as director of research and vice-president, technical of Inmont Corp., Clifton, NJ. At Inmont, in addition to management activities, Dr. Wicks participated in and directed many studies of polymers, coatings, printing inks, adhe-

sives, and textile colorants. In 1975, 1978, 1980, and 1982, Dr. Wicks was presented with the Roon Award by the Federation of Societies for Coatings Technology for his outstanding technical papers. He was also honored as the 1981 Fargo Chamber of Commerce Distinguished Professor, receiving a \$5,000 cash award.

In his research pursuits under some \$60,000 in annual industrial grants, Dr. Wicks focused efforts on research related to new types of coatings such as watersoluble industrial baking enamels, ultraviolet cure coatings, low-temperature cure coatings, and high solids coatings. These efforts were directed largely at reducing energy consumption, solvent emissions, and emission of possibly toxic by-products into the atmosphere—all major problems for the industrial coatings industry.

Dr. Wicks served as Visiting Lecturer at the University of Stuttgart, Germany in 1975 and 1978, at the University of Tokyo in 1977, and in China last summer for five weeks.

Following his retirement, Dr. Wicks will serve as a consultant and lecturer for four months in China. He will lecture at the Ministry of Chemical Industries, attended by researchers in paint and allied industries from throughout China, and also at Lanzhou at the Paint and Coatings Industries Research Institute.

On his return to the U.S., Dr. Wicks hopes to resume studies at an archeological dig in northern New Mexico. He hopes to do additional work on developing coatings that can be used to better preserve archeological artifacts.

Dr. Wicks earned an A.B. in chemistry in 1941 at Oberlin College and his Ph.D. in chemistry in 1944 at the University of Illinois. He is an Educator/Student Member of the Northwestern Society.

Replacing Dr. Zeno Wicks as Chair of the Polymers and Coatings Department of North Dakota State University is **Dr**.

Frank N. Jones, Manager of the Chemical Products Research Department at Cargill, Inc., Minneapolis, MN.

Since 1979, Dr. Jones has served in his current post at Cargill, leading a team of 30 chem-

ists and engineers involved in research and development of polymers for use in



F.N. Jones

Premier Mill Corp., New York, NY, has appointed **Edward Simal** & Associates, Inc., Atlanta, GA, to cover North and South Carolina, Georgia, Alabama, eastern Mississippi, and eastern Tennessee. Mr. Simal is a Southern Society

member. In addition, Cypress Color and Chemical, Inc., Westwood, MA will represent Premier in the New England area and eastern New York. Responsible for the sale of Premier's products is **Robert Marderosian**. He is a member of the New England Society.

John Boehle, Jr., has been appointed Vice-President, Pigments Department, Ciba-Geigy Corp., Hawthorne, NY. Dr. Boehle most recently served as Vice-President of the firm's applied plastics department.

Also announced by the corporation was the appointment of Michael Stadelmaier to the position of Sales Service Coordinator for the Coatings, Radiation Curing, and Photography Group within the Additives Department. He will be headquartered in Ardsley, NY.

The election of **Peter E. Flood** as President of The Flood Co., Hudson, OH, has been announced by **Earl Flood**, former President, who has become board Chairman. Other management changes include the appointment of **Jake Clark** as Secretary/Treasurer and the retirement of **Warner/Clark**, former Treasurer. coatings and plastics, polyurethane elastomers, and seed protectants.

Before joining Cargill, Dr. Jones served from 1963-73 as a staff chemist for the duPont Central Research Department, Wilmington, DE. From 1968-73 he served as a chemist and research supervisor working on acrylic polymers, automotive coatings, powder coatings, and pigment dispersion stabilizers. He joined Celanese Corp., Louisville, KY, in 1973 as research and development manager.

Dr. Jones earned a B.S. Degree in Chemistry from Oberlin College in 1958, and a ph.D. in organic chemistry from Duke University in 1961.

Dr. Jones is a member of the Northwestern Society. He is also a member of the National Paint and Coatings Association and the American Chemical Society.

At the annual stockholders meeting of Southern Protective Products, Atlanta, GA, a vote was taken to change the company's name to Superior Sealants, Inc. Serving as President of the firm is Larry Hecht.

Regina A. Haley has been appointed Vice-President of Marketing of Finnaren & Haley, Inc., Ardmore, PA. Ms. Haley is the first woman to hold the position of vice-president in the firm's 70-year history. In this position, Ms. Haley will develop marketing strategies and plans, and will direct all internal and external advertising, marketing, and public relations efforts for the company's 18 stores. In addition, she will act as Marketing Consultant to the company's 70 dealers.

Walter A. Bauer has been named Corporate Sales and Marketing Director for the AZS Corp., Atlanta, GA.

Jack E. Phelan was promoted to the position of Division Vice-President, Corporate Sales, for Nalco Chemical Co., Oak Brook, IL. As a 28-year employee of Nalco, Mr. Phelan has served as a District Representative in the Waste Treatment Group, Area Manager and District Manager in the Metal Industry Chemicals Group, Industry Manager for the steel industry, and Executive Account Manager in Corporate Sales. Prior to his promotion, he served as Regional Sales Manager in the Water Treatment Chemicals Group. Southern Coatings, Inc., Sumter, SC, has announced the promotion of Jerry Jones to Technical Director and Chris Hall, to Group Leader of Specialty and Compliance Coatings in its Product Finishes Div.

Also, joining the company as chemists are Joe Mamayek, serving as Group Leader of Baking Finishes, and Tim Taylor, appointed Group Leader of Air Dry Coatings.

M&T Chemicals, Inc., Woodbridge, NJ, has announced the promotion of William M. Kraus to the position of Vice-President, Chemicals, responsible for the management of the plastic additives, bio & fine, and industrial chemicals divisions.

Gary L. Solgere has been appointed a Sales Engineer by the driQuik Industrial Div., Dry Clime Lamp Corp., Greensburg, IN. Tom Brown has joined Red Spot Paint & Varnish Co., Evansville, IN, as Manager of Manufacturing. He replaces Ed Jones, who has retired after 40 years of service with the firm.

English China Clays America (ECC America), Atlanta, GA, has announced the following appointments. **Dr. Robert A. Burns** has been named Vice-President, Marketing. **Kenneth J. Hartman** has been appointed Vice-President, Sales. Dr. Burns and Mr. Hartman will be responsible for the worldwide marketing and sales activities, respectively, for ECC America and its subsidiaries, Anglo-American Clays Corp. and Southern Clay Products, Inc.

Illinois Bronze Paint Co., Lake Zurich, IL, has announced the following appointments. Lawrence A. De Marino has been named Director of Marketing and Ellen Metz has been appointed National Accounts Manager.

CALL FOR PAPERS ASTM Committee E-12 Symposium "Review and Evaluation of Appearance: Methods and Techniques" May 23, 1984 Montreal, Canada

A call for papers is being made by the ASTM Committee E-12 on Appearance of Materials for its symposium entitled, "Review and Evaluation of Appearance: Methods and Techniques," scheduled for May 23, 1984 in Montreal, Canada.

The purpose of the symposium is to provide a forum for researchers, quality control personnel, buyers, sellers, and standards writers to present recent developments in how to measure or specify the appearance attributes of materials. Appropriate topics for contributed papers include the following:

- The psychophysical attributes of appearance, distinctions between the physical attributes, and the most recent work by the CIE in these areas.
- Effort toward standardizing the conditions of observation and measurement.
- Methodology in choosing standards for appearance specification, i.e. chromatic or geometric attributes or materials classification.
- Research in color, color difference, gloss, image clarity, haze, turbidity, opacity (hiding power), reflectance, retroflectance, conspicuity, transmittance, transparency, yellowness, whiteness, spectrophotometry and goniophotometry.
- Applications involving opaque, light-transmitting, metallic, fluorescent, retroflecting and other materials. Also, other related subjects.
- Papers are expected to emphasize either new research or practical applications in the field of appearance measurement. The program will consist of an invited keynote speaker and sessions relating to the above topics.

A special technical publication on the symposium proceedings is anticipated by ASTM.

Prospective authors are requested to submit a title, a 300–500 word abstract, and an ASTM Paper Submittal Form by August 1, 1983 to Kathy Greene, Manager, ASTM Publications Div., Acquisition & Review, ASTM, 1916 Race St. Philadelphia, PA, 19103.

Applied Color Systems, Inc., Princeton, NJ, has named Norman Uress as Assistant Manager of Marketing.

Ted L. Disabato has been named Director of Marketing for Chemical Coatings Div. of The Sherwin-Williams Co., Chicago, IL.

Obituary

Donald A. Metz, 69, former President and co-founder of Van Horn, Metz & Co., Inc., Conshohocken, PA, died March 16.

Mr. Metz, who was active in the Philadelphia Society for Coatings Technology and was a past Executive Committee member of the Philadelphia Paint and Coatings Association, joined H.M. Van Horn in 1949 as co-founder of Van Horn, Metz & Co., Inc. In 1961, he was elected president of the firm and served in that position until his retirement in 1972.

Following graduation from Yale University with a B.S. Degree from the Sheffield Scientific School, Mr. Metz was associated with Winthrop Chemical Co. before leaving to take up his commission in the Army in 1941. After his release from the army, Mr. Metz joined the Zapon Div. of Atlas Powder Co., serving as Manager of Technical Services.

Mr. Metz is an original member of the Gallows Birds and also served as Chairman of the Board of Meadowbrook Inventions, Bernardsville, NJ.

He is survived by his wife, Mildred Garnett Metz, a son, three daughters, and five grandchildren.

Dr. Abraham Learner, 78, Past-President of the Birmingham Paint, Varnish & Lacquer Club, died February 16. A 50year member of the Club, Dr. Learner served as its President in 1934-35 and again in 1940-41. He was awarded Honorary Membership to the Club in 1947.

A graduate of Newcastle University with a B.Sc. Degree in Chemistry and of Birmingham University with a Ph.D., Dr. Learner had been associated with the British Cyanide Co. (later to become British Industrial Plastics); had formed Learners' Paints Ltd.; and had founded Nu Fabrics Pty. Co. Ltd.

Literature

Acrylate

A new functional caprolactone acrylate, developed specifically for acrylic and other copolymers that are formulated into higher solids, crosslinked industrial coatings used for automotive topcoats, appliance finishes, and general metal coatings, is featured in recent literature. For further information, contact Union Carbide Corp., Specialty Polymers and Composites Div., Dept. M1553, Danbury, CT 06817.

Pigments

Literature is available featuring four new high performance pigments for automotive and other coatings applications. Information is provided on: Irgazin Orange 5R, a red shade azomethine nickel complex pigment which exhibits excellent lightfastness in automotive exterior coatings; Irgazin Orange 3GL, a high strength, bright, clean isindoline pigment which exhibits excellent lightfastness at high or low chroma levels in both tints and metallic coatings; Irgazin Yellow 3R, a red shade isoindolinone cobalt complex pigment which has excellent lightfastness and heat stability; and Irgazin Yellow 4GT, an azomethine cobalt complex pigment, which exhibits high strength, transparency, and exceptional weatherability in both tints and metallic finishes. For additional information, contact CIBA-GEIGY Corp., Pigments Dept., 3 Skyline Dr., Hawthorne, NY 10532.

Additive

An improved version of Claytone PS^R rheological control additive for unsaturated resin applications is the subject of new literature. For information, contact Southern Clay Products, P.O. Box 44, Gonzales, TX 78629.

Vinyl Acetate

A revised handbook of technical information for VAMTM vinyl acetate is now available. The updated, 24-page "VAM Vinyl Acetate Monomer Handbook" features health and safety information and describes specifications, properties, applications, polymerization and chemical reactions, and shipping characteristics. Uses of the vinyl acetate are also listed. For a copy of the handbook, contact U.S.I. Chemicals Co., Technical Literature Dept., 99 Park Ave., New York, NY 10016.

Pigments

A new, exterior-grade, non-metallic, metallic luster pigment, recommended for product finishes where a decorative, metallic luster pigment, recommended for product finishes where a decorative, metallic effect must have the durability and resistance to withstand long-term outdoor exposure to weathering, is featured in recent literature. Additional information can be obtained from The Mearl Corp., 41 E. 42nd St., New York, NY 10017.

Valves

A comprehensive 400-page engineering catalog covering a full line of plastic valves in sizes ¹/₄" thru 8" (ball, diaphragm, butterfly, process control, yglobe, metering, solenoid valves, line strainers, and piping systems) is available. Included are data, charts, and tables featuring physical properties of plastic materials and their chemical resistance. Information is provided on installation practices and specifics of different valve types. For a copy, contact +GF+ Plastic Systems, Inc., 2882 Dow Ave., Tustin, CA 92680.

Alkyd

An oil free water-reducible alkyd for production of high quality industrial coatings is covered in a recently published bulletin. Provided are the physical properties, chemical resistance, and formulation properties of the resin. Contact Reichhold Chemicals, Inc., 525 N. Broadway, White Plains, NY 10603 for bulletin SC-121.

Polyols

A 24-page booklet describing specification polyols for urethane and nonurethane applications has been recently issued. "Specification Polyols" discusses four polyol types which cover both polyether and capped diols and triols. Application uses are discussed as are urethane chemistry, formulation principles, and the function of prepolymers. Typical physical properties are listed in table form as are solubility data and physical properties. Storage and handling information and shipping data are also provided. Copies of the booklet designated SUI-209, are available from Union Carbide Corp., Urethane Intermediates, Dept. K1476, Danbury, CT 06817.

Instruments Catalog

A new, comprehensive handbook of paint testing, corrosion control, and laboratory instruments is now available. Its 590 pages cover over 3,000 items and include complete descriptions, prices, instructions for use, and specification references. Some of the catagories covered include: adhesion, impaction, abrasion, film application, color and gloss, grind fineness, film thickness, flashpoint, gelation, hardness hiding power, milling and mixing, pH measurement, refraction, and surface profile. Some of the instruments described include: paint thickness gages, Zahn viscosity cups, viscometers, paint testers, Setaflash flash point testers, pH meters, colorimeters, mills and mixers, safety cans and equipment, hardness testers, hiding power instruments, wet film applicators, and adhesion testers. A copy of the catalog is available from Paul N. Gardner Co., Inc., 218 Commercial Blvd., Suite 205, Lauderdale by-the-Sea, FL 33308.

Defoamer

Roller foam, a familiar application problem, is eliminated from latex paints and other aqueous systems by Troykyd 777, a liquid defoamer, which is featured in new literature. Detailed technical information is provided as are the defoamer's advantages such as high efficiency, excellent retention, and minimal adverse effects on flow, color acceptance, and other properties. Information is available from Troy Chemical Corp., One Avenue L, Newark, NJ 07105.

Urethane Coatings

A 12-page brochure detailing the performance characteristics of aliphatic urethane coatings, and offering guidelines in selecting the appropriate aliphatic urethane coating system to meet specific environmental and performance requirements has been recently published. "Aliphatic Urethane Coatings-Performance Data Guide" is fully illustrated with color photos of applications and details the performance characteristics of two of the most common aliphatic urethanes: acrylic and polyester. Charts compare the overall cost-effectiveness, life expectancy, and performance characteristics of three aliphatic urethane coatings with eight other generic high-performance coating systems. The literature is available from Mobay Chemical Corp., Plastics and Coatings Div., Penn Lincoln Parkway W., Pittsburgh, PA 15202.

Catalysts

A 20-page brochure is available which features a complete line of homogeneous catalysts. This revised and updated brochure, with over 95 references, reviews the applications of soluble organometallics and transition metal salts which are useful in single phase reactions. Free copies are available from ALFA Products, Thiokol/Ventron Div., 152 Andover St., Danvers, MA 01923.

Emulsion

An improved emulsion product containing 45% minimum solids which is suited for automotive rust preventive applications as well as for other industrial coating uses is the subject of new literature. Advantages and application uses of SACI-445W emulsion are detailed. For information, write Witco Chemical Corp., Sonneborn Div., 520 Madison Ave., New York, NY 10022.

Polyester Polyol

Literature is available featuring a new 100% solids polyester polyol, designed for high reactivity and fast cure. Recommended uses and advantages are listed. Information can be obtained from King Industries, Inc., P.O. Box 588, Science Rd., Norwalk, CT 06852.

Mixers

A new, 24-page catalog is available which features a complete line of portable mixers. Over 200 models are listed as are related items. Free copies of the catalog are available from Indco, Inc., P.O. Box 589, New Albany, NY 47150.

Wood and Tall Oil Products

A comprehensive line of wood and tall oil naval stores products are covered in a new bulletin. The eight-page publication covers various rosins, pine oils and tars, tall oil products, rubber and paper chemicals, resins, hydrocarbon resins, as well as special terpene chemicals, terpene hydrocarbons, and gloss oils. Literature is available from Reichhold Chemicals, Inc., 525 N. Broadway, White Plains, NY 10603.

Additive

Literature is available introducing NiPar S-20 which provides low viscosity with relatively high content of isopropyl alcohol in the solvent blend when formulating lacquers. Write Angus Chemical Co., 2211 Sanders Rd., P.O. Box 3037, Northbrook, IL 60062.

Published Proceedings Of 1982 FATIPEC Congress

The Proceedings of the 1982 FATIPEC Congress, held in Liege Belgium, is available from: ATIPIC, Square Marie Louise 49, B-1040 Brussels, Belgium.

The Proceedings consist of four volumes totalling 1,360 pages. The papers are in published in English, French, or German. The price is 4,700 Belgian francs (about \$98).

Checks should be made payable to: Account 210-0235175 of ATIPIC, and mailed to: Societe Generale de Banque, Agence Dilbeek, Chssee de Ninove 84, B-1770 Dilbeek, Belgium.

Measurement System

Color, brightness, and opacity measurements in the pulp and paper, textile, paint, plastics, and printing industries can be performed faster with the new ElrephoMat System, which is introduced in recent literature. Described are the system's new features including automatic sequencing, automatic data processing, digital display, and on-line operation. Analysis and application uses are discussed as well as the range of measurements which can be performed. For further information, contact Carl Zeiss Canada Ltd., Klaus Wagener, 45 Valleybrook Dr., Don Mills, Ont., M3B 256

Urethane System

Literature is available announcing the release of a new high solids two component urethane system: Spenkel P81-A1-75 is the aromatic urethane component, and Polyol 8100 is the polyol second component. Formulation properties and application uses are featured. Contact Spencer Kellogg, Div. of Textron, Inc., 120 Delaware Ave., Buffalo, NY 14240.

Reactive Diluent

Literature is available discussing V-Pyrol/RC vinylpyrrolidone reactive diluent, which is used in radiation curable coatings/inks for many types of products and has been improved with respect to quality and packaging. Discussed is the assay of this liquid vinyl monomer which has been increased to a minimum of 99% and the moisture content which has been decreased to 0.1%. Information is provided on the elimination of its trace pinkish coloration. Storage and handling data is also included. For a copy of the data sheet and technical brochure #2302-081R, write GAF Corp., Acetylene Chemicals, 140 W. 51st St., New York, NY 10020.

Corrosion Inhibitors

Literature is introducing a new line of phosphate-base corrosion inhibitors for open recirculating cooling water systems. For further information, contact Hercules Incorporated, 910 Market St., Wilmington, DE 19899.

Vehicle

A new pre-neutralized water-reducible exterior vehicle for air-dry wood coatings, stains, and light-duty concrete compounds is the subject of recent literature. Recommended applications and formulations are listed. For further information, write Cargill, Inc., Research Dept., P.O. Box 9300, Minneapolis, MN 55440.

Polyurethane

A new VOC-complying two-component polyurethane high solids finish is introduced in recently published literature. Information is provided on its application uses and performance properties. Further information can be obtained from G.L. Holmberg, Chemical Coatings Div., Sherwin-Williams Co., 11541 S. Champlain Ave., Chicago, IL 60628.

Pigment

An 18-page brochure on a new anitcorrosive pigment that is an effective, environmentally acceptable alternative to lead and chromate pigments, is available. Technical data on the low oil absorption, fine partical size pigment are featured as are demonstrations of its high performance in a variety of solvent and water based paint systems. For a copy of the brochure, contact NL Industries, P.O. Box 700, Hightstown, NJ 08520.

Polymer

A new, low-priced, easy to handle, amide imide polymer used to make topcoat enamels for high temperature applications is the subject of recent literature. The polymers' unique advantages are listed as are its application uses. For additional information, request bulletin AM-3 from Amoco Chemicals Corp., Dept. ADV-JCT, Mail Code 4002, 200 E. Randolph Dr., Chicago, IL 60601.

Airless Sprayer

Literature is available featuring a new powerful airless sprayer, designed for one or two-gun production painting. For more information, contact The O'Brien Corp., Dept. TC, 450 E. Grand Ave., S. San Francisco, CA 94080.

Solvents

A technical brochure on the use of CHLOROTHENE^R SM and AERO-THENE^R MM solvents in the coatings industry is now available. The 10-page brochure provides information on the physical properties of the solvents and addresses health and safety considerations. Benefits of using the solvents are also discussed. For a copy of the literature, contact Susan King, Inquiry Correspondent, Inorganic Chemicals Dept., Dow Chemical U.S.A., 1703 S. Saginaw Rd., Midland, MI 48640.

Alkyl Alkanolamines

An 18-page booklet describing alkyl alkanolamines for textiles, pharmaceuticals, household and personal care specialties, corrosion inhibitors, gas treating, coatings, and other diverse applications has been recently issued. The literature explains the chemistry of alkyl alkanolamines and lists 11 generic alkyl alkanolamines-four that serve as general intermediates and seven developed for special applications. An applications section discusses the use of alkyl alkanolamines in the production of textile processing chemicals such as emulsifying agents, lubricants, and coatings. Physical properties are listed in a table and graphs illustrate temperature relationships with vapor pressure, apparent specific gravity and viscosity, and liquid-vapor equilibria. Detailed specifications are also presented, as are shipping data, health effects, and storage and handling data. Copies of, "Alkyl Alkanolamines," F-47185A, is available from Union Carbide Corp., Specialty Chemicals Div., Dept. K3446, Danbury, CT 06817.

Mill

A 16-page brochure describing the features and advantages of a new C-MILL SYSTEM-Computerized Milling System is now available. The literature discusses how the system incorporates advanced high-speed dispersion and particle reduction equipment with full computer control capabilities. Contact Morehouse Industries, Inc., P.O. Box 3620, Fullerton, CA 92632 for additional information

Colorimeters

A new brochure that describes a complete line of tristimulus colorimeters is now available. The literature provides general information on the colorimeters and explains their use for a wide variety of color and appearance measurements. For a copy of the brochure, write Hunter-Lab, 11495 Sunset Hills Rd., Reston, VA 22090.

Instruments

A catalog is available featuring a complete line of electronic analytical and precision balances, balance accessories, peripherals, and precision instruments. This 34-page, full-color catalog describes the instruments available and their unique features. Also, a fold-out section gives a summary of all models with specifications and data interfacing information. Contact, Mettler Instrument Corp., A.F. Bremble, Jr., Box 71, Hightstown, NJ 08520, for more information.

Polvols

An eight-page booklet on polyols for high-performance coatings, adhesives, and elastomers has been issued. The booklet describes the polyols and lists their special features. Applications are noted as are the polyols' availability in eight molecular weight grades. Additional information is provided on FDA status, health effects, shipping, storage, and handling. Copies of "TONE Polyols for High Performance Coatings, Adhesives, and Elastomers", designated F-49593, can be obtained from Union Carbide Corp., Specialty Polymers and Composites Div., Dept. M1553, Danbury, CT 06817.

Polymers

Literature is available featuring two new polymers, one that permits the formulation of a roof mastic with superior water ponding resistance and one with improved adhesion to polyurethane foam. Rhoplex^R EC-1895 emulsion, designed for the production of a roof coating for flat roofs with potential water ponding problems, and Rhoplex EC-1791, formulated for elastomeric roof mastics with superior wet and dry adhesion to both high and low density polyurethane roofing systems, are fully discussed and detailed. Form llations and application advantages are also listed. For information, contact Rohm and Haas Co., Marketing Services Dept., Independence Mall West, Philadelphia, PA 19105.

Emulsion

A high-performance pressure-sensitive emulsion combining aggressive peel and tack properties with excellent shear resistance is introduced in new literature. Suitable applications for the emulsion are discussed as are its useful advantages. For information, contact Air Products and Chemicals, Inc., Polymer Chemicals, Box 538, Allentown, PA 18105.

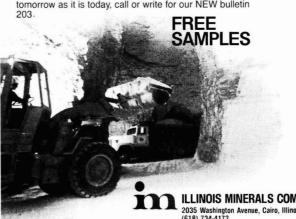
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Evaporation Mechanism Data Questioned

TO THE EDITOR:

"Comparative Solvent Evaporative Mechanisms for Conventional and High Solids Coatings" by William H. Ellis represents an excellent piece of experimental research. There are clearly two mechanisms operating to control the evaporation of organic solvents from the coatings studied. They are not those stated in the article, however. There is no internal diffusion resistance of significance to be found in these systems until after 90% of the solvent has evaporated. if then. This can be demonstrated by studies of evaporation from the same system at different film thicknesses. Unfortunately the author does not mention film thicknesses.

I suspect from earlier work that the two stages found by Ellis are those primarily controlled by heat transport initially and later on by mass transport with a falling surface concentration. We have definitely found this for waterreducible coatings, and the same behavior can also be seen in the data for evaporation from solvent systems, although no specific mention of this is made.

> C.M. HANSEN, Horsholm, Denmark

Mr. Ellis responds:

Usually, I would be flattered and pleased that Charles Hansen was interested enough to comment on my paper because I have often admired his fine research. In this case, however, I cannot agree completely with his unsupported, dogmatic pronouncements.

We have ample evidence over a period of many years to demonstrate that heat transport is not a significant factor in first-stage evaporation measurements with our instrument. The instrument provides sufficient heat input to compensate for heat of vaporization and to maintain isothermal conditions. If temperature changes were a factor, evaporation rates would not correlate so well with vapor pressure measurements; nor would the initial evaporation rates with and without solute be equal because the solution has greater film thickness and greater heat capacity than the neat solvent. Finally, we did run experiments at different film thickness but did not dwell on this topic because it proved insignificant.

Our disagreement on the second-stage mechanism may be partly, but not entirely, semantics. Diffusion is the migration of a solvent molecule through the film to and through the surface where vaporization can occur. Clearly, the resin affects this process in most cases long before 90% of the solvent has evaporated. Furthermore, the analogy between solvent and water-based systems is not valid because their structures are quite different.

> W.H. ELLIS, El Segundo, CA

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National High Blood Pressure Education Program, National Heart, Lung, and Blood Institute, U.S. Department of Health and Human Services

Coming Events

FEDERATION MEETINGS

(Oct. 12-14)—61st Annual Meeting and 48th Paint Industries' Show. Place Bonaventure, Montreal, Quebec, Canada. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

1984

(May 17-18)—Spring Meetings. Society Officers on 17th; Board of Directors on 18th. Galt House, Louisville, KY. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

(Oct. 24-26)—62nd Annual Meeting and 49th Paint Industries' Show. Conrad Hilton Hotel, Chicago, IL. (FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107).

SPECIAL SOCIETY MEETINGS

1984

(Mar. 7-9)—Southern Society for Coatings Technology. Fiftieth Anniversary Meeting. Surfside Hotel, Clearwater, FL. (James E. Geiger, Sun Coatings, Inc., 12295 75th St., N., Largo, FL 33543).

(Apr.)—Cleveland Society for Coatings Technology 27th Annual Technical Conference, "Advances in Coatings Technology. (Richard R. Eley, Glidden Coatings & Resins Div. SCM Corp., 16651 Sprague Rd., Strongsville, OH 44136).

(Apr. 12–14)—Southwestern Paint Convention of Dallas and Houston Societies. Shamrock Hilton Hotel, Houston, TX.

1985

(Feb. 26-Mar. 1)—Western Coatings Societies Symposium and Show. Disneyland Hotel, Anaheim, CA.

OTHER ORGANIZATIONS

(June 15–18)—Oil & Colour Chemists' Association Biennial Conference on "The Efficient Use of Surface Coatings." Viking Hotel, York, England. (R.H. Hamblin, Director and Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, HAO 2SF, England).

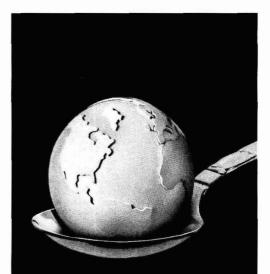
(June 19–22) — Dry Color Manufacturers' Association Annual Meeting. The Greenbrier, White Sulphur Springs, WV. (P.J. Lehr, DCMA, Suite 100, 117 N. 19th St., Arlington, VA 22209).

(June 20-24)—"Advances in Color Technology" Course. Rochester Institute of Technology, Rochester, NY. (Val Johnson, T&E Center Seminar Coordinator, Rochester Institute of Technology, P.O. Box 9887, Rochester, NY 14623).

(June 20–24)—"Applied Rheology for Industrial Chemists" Short Course. Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(June 20–24)—"Films and Coatings for Technology" Short Course. Palace Hotel, Zandvoort, The Netherlands. (The Continuing Education Institute, Europe [CEI-Europe], Gustav Adolfsgatan 9, S-582 20 Linkoping, Sweden).

(June 20-24)—"Printing Inks and Printing Processes" Short Course. Lehigh University, Bethlehem, PA. (Dr. Mohamed S. El-Aasser, Dept. of Chemical Engineering, Sinclair Lab #7, Lehigh University, Bethlehem, PA 18015).



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For technical literature and detailed information write: Jerry Walter, **Sherwin-Williams, Chemical Division,** 501 Murray Road, St. Barnard, Cincinnati, Ohio 45217.



(June 26–27)—ASTM Committee E-12 on Appearance of Materials Meeting. Hyatt Regency Nashville, TN. (Bob Morgan, ASTM, 1916 Race St., Philadelphia, PA 19103).

(June 26–29)—ASTM Committee D-1 on Paint and Related Coatings and Materials Meeting. Hyatt Regency Nashville, Nashville, TN. (Phil Lively, ASTM, 1916 Race St., Philadelphia, PA 19103).

(June 26–30)—70th Annual Technical Conference and Exhibit, "SUR/FIN '83," sponsored by the American Electroplaters' Society. Indiana Convention-Exposition Center, Indianapolis, IN. (*Co-Chairmen:* Paul Johnson, Guide Div., GMC, 2915 Pendleton, Anderson, IN 46011; Richard Watson, Benchmark, Inc., 5256 N. Tacoma Ave., Suite 9, Indianapolis, IN 46220).

(July 25–29)—"Microbiological Degradation" sponsored by the Gordon Research Conferences. Brewster Academy, Wolfeboro, NH. (Alexander M. Cruickshank, Director, Gordon Research Conferences, University of Rhode Island, Kingston, RI 02881).

(July 29-31)—Oil & Colour Chemists' Association, New Zealand Div., 21st Annual Convention. International Hotel, Rotorua, New Zealand. (OCCA, 1983 Convention, P.O. Box 5192, Auckland, New Zealand).

(June 29-July 1)—"Color Control Technology" Seminar sponsored by Applied Color Systems, Inc. Landmark Hotel, Myrtle Beach, SC. (ACS, Inc., P.O. Box 5800, Princeton, NJ 08540).

(Aug. 8–12)—Fourth International Conference on the Organometallic and Coordination of Germanium, Tin, and Lead. McGill University, Montreal, Que., Canada. (Prof. M. Onyszchuk, McGill University, Dept. of Chemistry, 801 Sherbrooke St. West., Montreal, Que., Canada H3A 2K6).

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4114 N. Ravenswood Ave., Chicago, Illinois 60613 U.S.A. Phone 1 312 327-4520 Telex 25-4328 (Aug. 8–12)—"Physics and Chemistry of Coatings and Films" sponsored by the Gordon Research Conferences. Plymouth State College, Plymouth, NH. (Alexander M. Cruickshank, Director, Gordon Research Conferences, University of Rhode Island, Kingston, RI 02881).

(Aug. 15–19)—"Advances in Emulsion Polymerization and Latex Technology" Short Course. Schatzalp Berghotel, Davos, Switzerland. (Dr. Gary Poehlein, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332).

(Aug. 17–19)—"Color Control Technology" Seminar sponsored by Applied Color Systems, Inc. Wequasett Inn, Cape Cod, MA. (ACS, Inc., P.O. Box 5800, Princeton, NJ 08540).

(Aug. 22–26)—"Physics and Chemistry of Printing Inks" Short Course. Davos, Switzerland. (Dr. Mohamed S. El-Aasser, Lehigh University, Dept. of Chemical Engineering, Sinclair Lab #7, Bethlehem, PA 18015).

(Aug. 26–30)—International Colour Association's Forsius Symposium on Colour Systems. Kungalv, Sweden. (Fred W. Billmeyer, Jr., Dept. of Chemistry, MRC 217, Rensselaer Polytechnic Institute, Troy, NY 12181).

(Aug. 31-Sept. 8)—20th Commission Internationale de l'Eclairage Congress. RAI Congress Center, Amsterdam, The Netherlands. (U.S. National Committee, CIE, c/o National Bureau of Standards, Washington, D.C. 20034).

(Sept. 17–19)—"Color Control Technology" Seminar sponsored by Applied Color Systems, Inc. Atlantic City, NJ. (ACS, Inc., P.O. Box 5800, Princeton, NJ 08540).

(Sept. 18–20)—Canadian Paint and Coatings Association's 71st Annual Convention. Hyatt Regency Hotel, Montreal, Quebec, Canada. (CPCA, 515 St. Catherine St. W., Suite 825, Montreal, Quebec H3B 1B4, Canada).

(Sept. 27–28)—Pulp Chemicals Association's 11th International Naval Stores Conference and Technical Symposium. Westin Peachtree Plaza Hotel, Atlanta, GA. (Pulp Chemicals Association, 60 E. 42nd St., New York, NY 10165).

(Sept. 27–29)—"Radiation Curing in the Printing and Converting Industry" Workshop sponsored by the Association for Finishing Processes of the Society of Manufacturing Engineers (AFP/SME). Hershey Hotel, Philadelphia, PA. (Donna Theisen, Administrator, Technical Activities Dept., SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121).

(Sept. 27–30)—National Coil Coaters Association Fall Technical Meeting. Hyatt Regency O'Hare, Chicago, IL. (NCCA, 1900 Arch St., Philadelphia, PA 19103).

(Sept. 28-Oct. 1)—Oil & Colour Chemists' Association's Silver Jubilee Convention and Exhibition. Southern Cross Hotel, Melbourne, Australia. (O.C.C.A.A., 1983 Pacific Coatings Convention, C/- Tioxide Australis Pty. Ltd., Private Bag 13, Ascot Vale, Victoria, 3032, Australia).

(Oct. 5–6)—"Finishing of Plastics for Functional and Decorative Purposes" Technical Conference. Decorating Division of the Society of Plastics Engineers. Hyatt Regency, Cherry Hill, NJ. (Ed Stumpek, General Electric, One Plastics Ave., Pittsfield, MA 01201).

(Oct. 11–13)—"Finishing '83" sponsored by the Association for Finishing Processes of the Society of Manufacturing Engineers. Cincinnati Convention Center, Cincinnati, OH. (Susan Buhr, AFP/SME Administrator, One SME Dr., P.O. Box 930, Dearborn, MI 48128).

(Oct. 30–Nov. 2)—National Paint & Coatings Association 96th Annual Meeting. Bonaventure Hotel, Los Angeles, CA. (Karen Welch, NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(Nov. 2–3)—3rd World Congress on Coatings Systems for Bridges and Steel Structures. Breckenridge Concourse Hotel, St. Louis, MO. (John A. Gordon, Jr., Conference Director, Arts & Sciences, Continuing Education, University of Missouri-Rolla, Rolla, MO. 65401).

(Nov. 2–4)—"Fundamentals of Adhesion: Theory, Practice, and Applications" Short Course. State University of New York, New Paltz, NY. (Dr. Angelos V. Patsis, Chemistry Dept., SUNY, New Paltz, NY 12561).

(Nov. 8-9)—Resins and Pigments Exhibition. Europa Hotel, London, England. (Polymers, Paints & Colour Journal, Queensway House, Redhill, Surrey, RH1 1QS, England).

(Nov. 11-13)-36th National Decorating Products Show. McCormick Place, Chicago, IL. (NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Dec. 7-8)-"Painting Public Structures: Practices and Economics" Symposium sponsored by the Steel Structures Painting Council. Cocoa Beach, FL. (Dr. Harold Hower, SSPC, 4400 Fifth Ave., Pittsburgh, PA 15213).

(Dec. 12-14)-"Color Control Technology" Seminar sponsored by Applied Color Systems, Inc. Disneyworld/Epcot Center, FL. (ACS, Inc., P.O. Box 5800, Princeton, NJ 08540).

1984

(Feb. 12-15)-Inter-Society Color Council Conference. Colonial Williamsburg Lodge. Williamsburg, VA. (Fred W. Billmeyer, Dept. of Chemistry, Rensselaer Polytechnic Institute, Troy, NY 12181).

(Feb. 12-16)-14th Australian Polymer Symposium sponsored by the Polymer Div. of the Royal Australian Chemical Institute. Old Ballarat Motor Inn, Ballarat, Australia. (Dr. G.B. Guise, RACI Polymer Div., P.O. Box 224, Belmont, Vic., 3216, Australia) -

(Apr. 8-10)-Inter-Society Color Council Annual Meeting. Michigan Inn, Southfield, MI. (Fred W. Billmeyer, Dept. of Chemistry, Rensselaer Polytechnic Institute, Troy, NY 12181).

(Apr. 12-15)-"FARBE 84". Munich Trade Fair Centre, Munich, West Germany. (Kallman Associates, Five Maple Court, Ridgewood, NJ 07450).

(Apr. 24-25)-Electrocoat/84 Conference, sponsored by Products Finishing Magazine. Drawbridge Inn, Cincinnati, OH. (Anne Porter, Products Finishing, 6600 Clough Pike, Cincinnati, OH 45244).

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'Humbug' from Killman

Finding their way to Humbug's Vermont nest were letters from Walter B. Maass, Howard Jerome, Sid Lauren, and Maureen Lein. A thanks to my faithful friends for their contributions.

I was very pleased to get the following thoughts from sceptics for sceptics, sent in by my non-sceptic friend of many years, Walter Maass.

Thoughts for Sceptics Waiter B. Maass

Let's face it, we live in a sceptical era. Statesmen, politicians, generals, men of letters seem to raise very little enthusiasm. The days when middle-aged ladies climbed on chairs to get a better view of the presidential candidate or literally jumped into the air seem very far away. Nowadays, people do not even cry when their congressman dies. What little passionate feelings are left, are spent on baseballplayers or moviestars. Even opera singers and rock musicians raise only a moderate degree of ecstacy. The prevailing atmosphere is polluted with doubt and mistrust.

Therefore, let us turn to those great minds who shared that attitude and listen to the comment on their times.

Domitian, Roman Emperor, (81-96 AD): "The lot of princes is an unhappy one. When they uncover a conspiracy, no one believes them, unless they are killed."

Galgacus, British chieftain (84 AD): "They make a desert and call it peace."

John Knox, Scottish reformer (1561): "The monstrous regiment of women!"

Henry IV, King of France (1594-1610) to a courtier: "I know you are not worthy of your office but my nephew asked me to appoint you."

Cardinal Richelieu (1642): "Secrecy is the soul of statesmanship."

Michel de Montaigne, French philosopher (1533-1592): "Men are most apt to believe what they least understand."

William Shakespeare: "The first thing we do, let us kill all the lawyers!" (Henry IV)

"The empty vessel makes the greatest sound." (Henry V)

"Oh, how wretched is that poor man that hangs on princes' favors! (Henry VIII)

A. de Voltaire (1694-1778) French man of letters: "Every great event has been a capital misfortune."

Thomas Jefferson: (1743-1826): "That government is best, which governs least."

L.A. de Saint Just (1793) French politician: "No one can govern without guilt."

Georges Clemenceau, French Prime Minister (1919), after being slightly wounded by a would-be assassin:

"After four years of war there are still some Frenchmen left who do not know how to shoot."—"All cemeteries are filled with irreplacable people."

Mark Twain: (1894): "If you pick up a starving dog and make him prosperous, he will not bite you. This is the principal difference between a dog and a man."

Winston Churchill (1950): "When you have to kill a man it costs nothing to be polite."

Ernest Hemingway (1929), American writer: "I was always embarassed by the words sacred, glorious and sacrifice."

Bernard L. Montgomery (1950) Fieldmarshal. "I do not think that I would make a good politician."

Lyndon B. Johnson, on becoming President of the USA (1963): "This is a sad time for all people."

Howard Jerome has sent in some "gems" accumulated over the years. Here are a few:

- Law of sub-committee formation: If you leave the room, you're elected.
- Assumption is the mother of all screw-ups.
- To err is human, but to really foul things up requires a computer.
- If you hit two keys on the typewriter, the one you don't want hits the paper.
- If it's green or it wriggles, it's biology. If it stinks, it's chemistry. If it doesn't work, it's physics.
- Ziegler's Law: Any technical problem can be overcome given enough time and money. Jerome's Corollary: You are never given enough time or money.

Sid Lauren recalls a story of his freshman chemistry days when his instructor asked—

"Can you describe the nature and properties of $H_2S?"$

"H₂S is hydrogen sulfide," the student halting replied. "It is a colorless gas, and — uh - uh - it has a pleasant odor."

"A pleasant odor?" the instructor asked, with some irritation.

"Well, I like it" answered the student (who was not definitely identified as Sid!).

De Gustibus

Trivia (but true) from Maureen Lein-

Since 1971, any money lost through bribery has been tax deductible, according to the IRS "Official Taxpayers' Guide." "Bribes and kickbacks to government officials are deductible unless the individual has been convicted of making the bribe, or has entered a plea of guilty or nolo contendere."

-Herb Hillman



Color-matching Aptitude Test Set was created by the Inter-Society Color Council and sponsored by the Federation. It is in world-wide use as a means for estimating color-matching skill. The 1978 edition contains minor refinements over the previous editions (1944, 1953, 1964), and these bring it closer to the original ISCC plan, making it a still more successful tool for evaluating color-matching skill.

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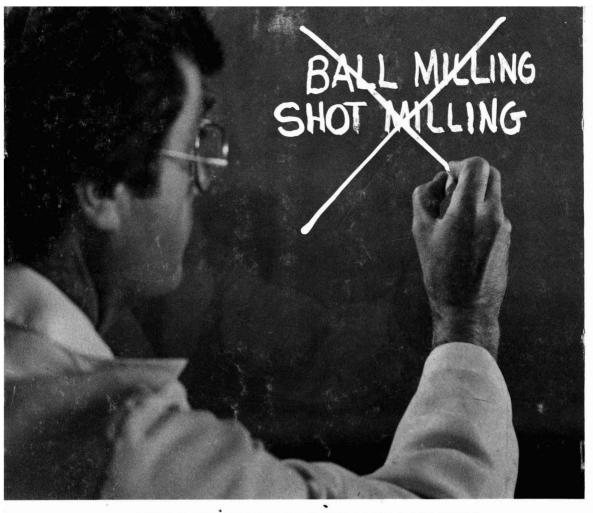
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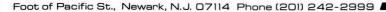
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