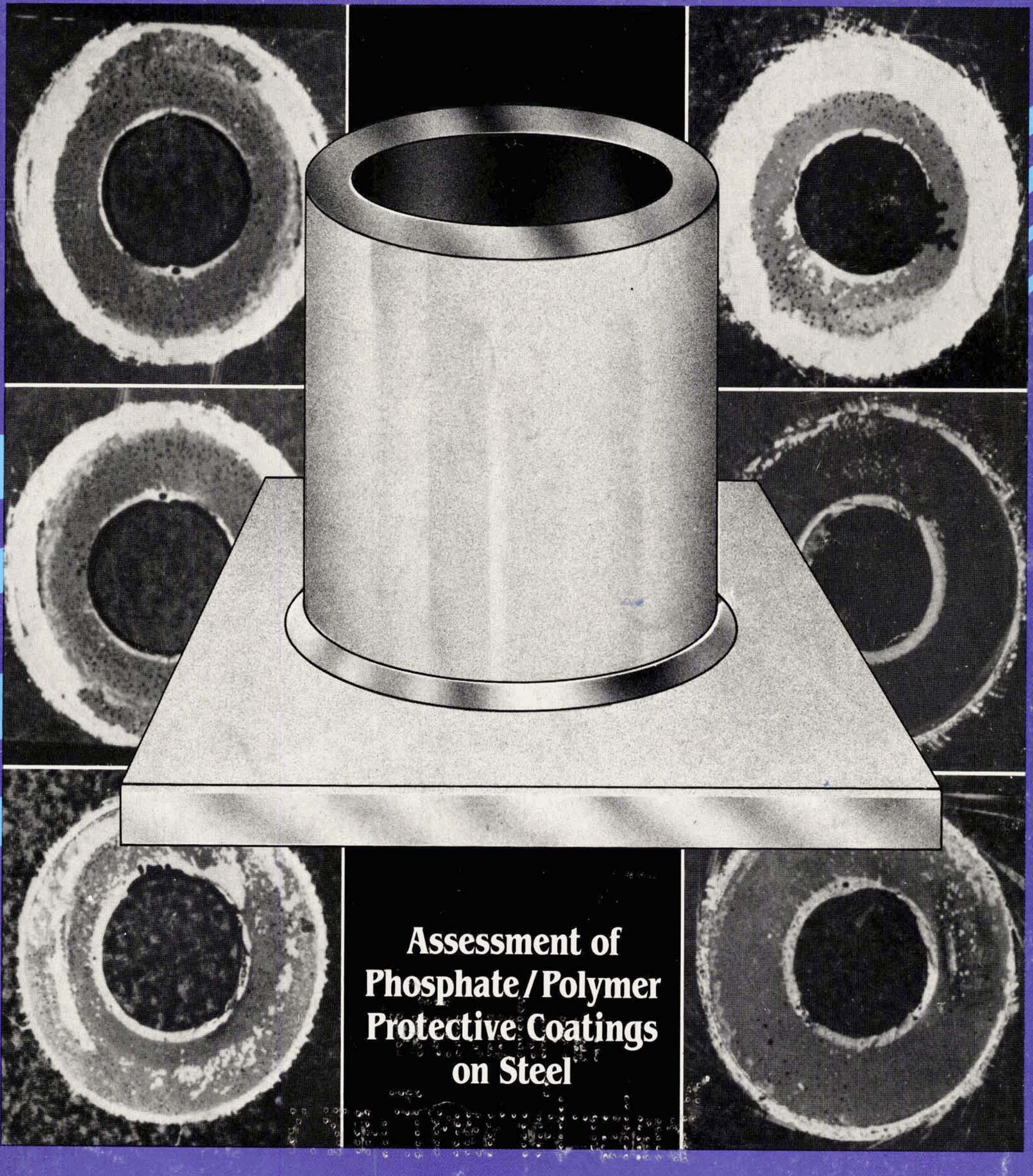
jct JOURNAL OF COATINGS TECHNOLOGY

JCTAX 59 (753) 1-116 (1987)

October 1987



Assessment of Phosphate/Polymer Protective Coatings on Steel

OCTOBER 1987

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VOLUME 59 NUMBER 753

Technical Articles

Federation News

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SOCIETY OF NATIONAL ASSOCIATION PUBLICATIONS

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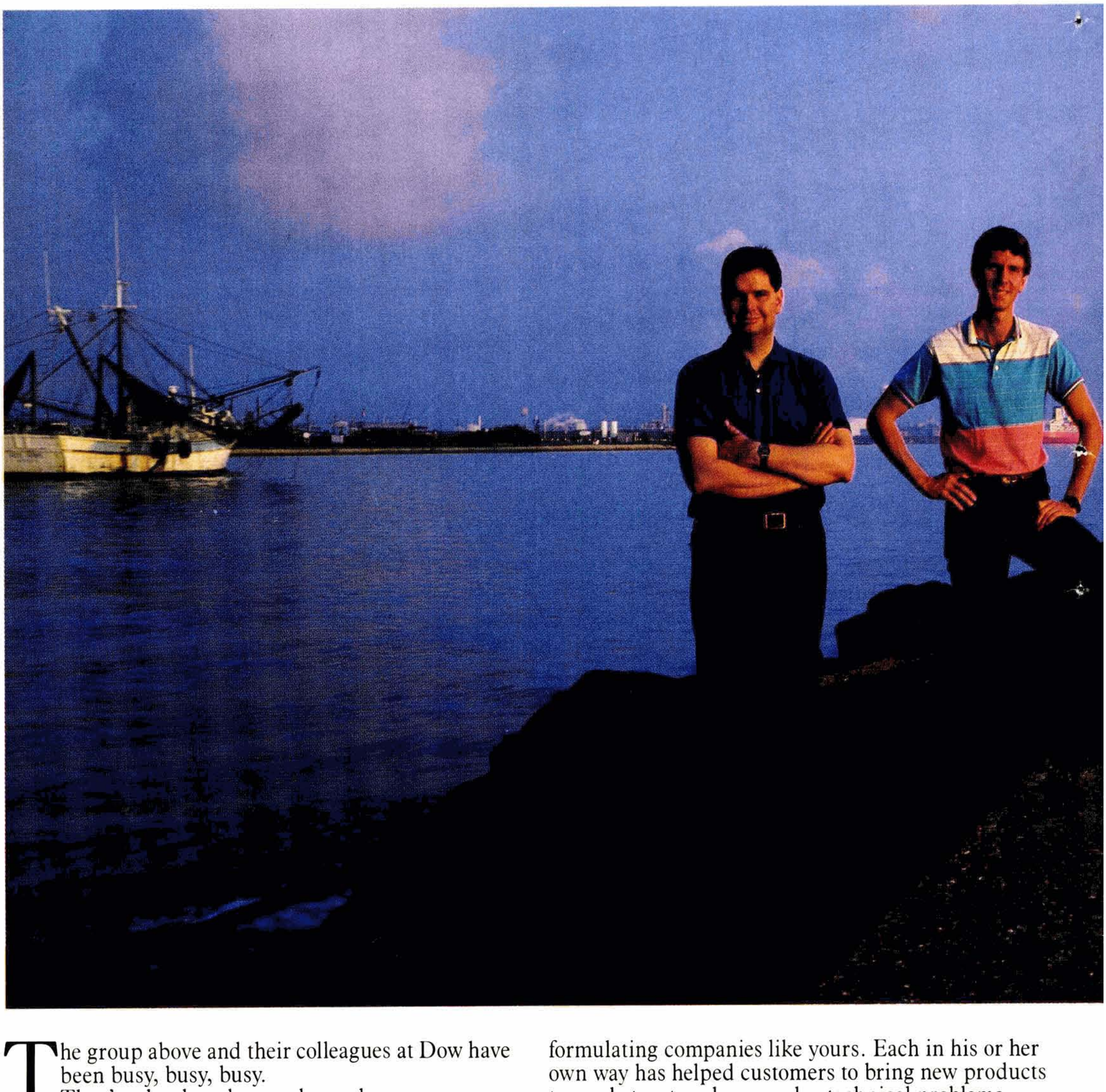
THE JOURNAL OF COATINGS TECHNOLOGY (ISSN 0361-8773) is published monthly by the Federation of Societies for Coatings Technology, 1315 Walnut St., Philadelphia, PA 19107. Phone: (215) 545-1507.

Second class postage paid at Philadelphia, PA and at additional mailing offices. POSTMASTER: Send address changes to JOURNAL OF COATINGS TECHNOLOGY, 1315 Walnut St., Philadelphia, PA 19107.

Subscriptions: U.S. and Canada—1 year, $27; 2 years, $51; 3 years, $73. Europe (Air Mail)—1 year, $55; 2 years, $107; 3 years, $157. Other countries—1 year, $40; 2 years, $77, 3 years, $112.

Thirteen degrees, 13 new mayor of Clute, Texas.



The group above and their colleagues at Dow have been busy, busy, busy.

They've developed more than a dozen new epoxy resins over the past three years, installed a new 7500 square foot laboratory in Freeport, Texas and added 13 new people to the Dow Resin Products coatings group. (You see seven of the veterans above: Henry, Tom, Chris, John, Lloyd, Dave and Dave.)

They've also lost elections, blown tennis games, and missed qualifying for the Boston Marathon, among other things. (The almost mayor of Clute, Texas is the fellow in the red shirt, Dave Hill. He lost by 61 votes.)

There are now more than 150 people in the Dow Resin Products coatings group working together with formulating companies like yours. Each in his or her own way has helped customers to bring new products to market or to solve complex technical problems.

Dave Hill, for example, has helped to bring seven epoxy resins (many for lower VOCs) to his marine and maintenance customers. He is working now on resins that will cure at lower temperatures and still yield traditional epoxy performance.

John Massingill, the chemist in the center of the photo, kept a can coating formulator from losing a one million dollar contract when its customer put on the squeeze. He did so by cutting the viscosity range of a resin in half while retaining all of the original performance characteristics.

epoxy resins, and one almost

In the coming months you will see more of Dave, John and their colleagues at Dow. They will be visiting labs, attending trade shows, and delivering papers at conferences. Wherever you see them, they are there to answer your questions, listen to your needs and respond with products and technical insights.

If you'd like, you can call right now to see how Dow epoxy resins can be put to work in coatings for automotive, marine and maintenance, powder, can and coil, and flooring. Just call toll-free 1-800-258-2436, ext. 21, Coatings, or send in the coupon.

Oh, and by the way, if you have any tips on how Dave Hill can win an election, please pass them on. He could use the help.

J1087

To: The Dow Chemical Company, Resin Products Department,
P.O. Box 1206, Midland, MI 48641-9940.

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Annual dues for Active and Associate Members of the Federation of Societies for Coatings Technology is $20.00. Of this amount, $13.50 is allocated to a membership subscription to this publication. Membership in the Federation is obtained through prior affiliation with, and payment of dues to, one of its 26 Constituent Societies. Non-member subscription rates are:

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1 Year	$27.00	$ 55.00	$ 40.00
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When available, single copies of back issues of the JOURNAL OF COATINGS TECHNOLOGY are priced as follows: $3.00 each for current calendar year issues; $4.00 each for all other issues.

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A Guide for Authors is published in each January issue.

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

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Comment

Cornerstones and Keystones

Earlier this month the Federation celebrated its 65th Anniversary during the Annual Meeting and Paint Industries' Show in Dallas, Texas. The theme of the event was "People and Technology: Cornerstones of Progress." Those readers who attended the two and one-half days of technical programs and walked the aisles of the record-setting Paint Show came away with a greater understanding of the industry, its problems and, hopefully, more than a few solutions.

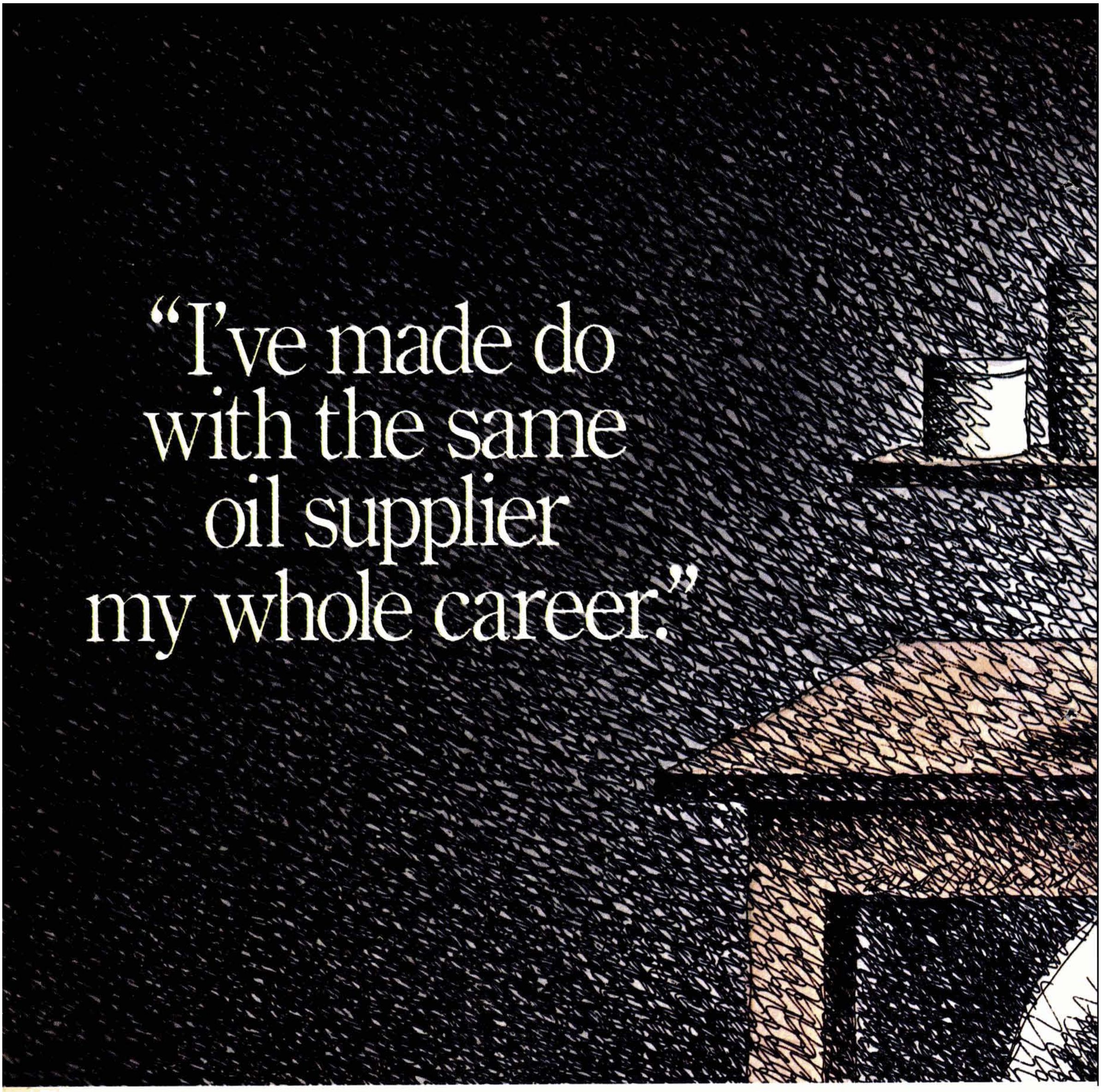
However, if for whatever reason, you decided not to attend or to send a representative, you missed the biggest and best industry forum to date. Topics ranged from compliance formulation and quality control to professional development, innovation and marketing.

To the Program Committee, speakers, and many volunteers who gave of their time and effort to make this meeting a success, the Federation offers its sincere thanks. And to the over 250 exhibiting companies and their representatives who made the 52nd Paint Show the largest in the Federation's long history, the gratitude of the FSCT membership is yours.

Just as People and Technology are the Cornerstones of the industry, so the Federation and, in particular, the Annual Meeting and Paint Show, is the Keystone which locks these two components of Progress together.

Robert F. Ziegler

Robert F. Ziegler,
Editor



See what you're missing.

If you're blindly hitched to a supplier whose line of solvents and oils is limited in hydrocarbon types, you can't see the formulating flexibility you're missing.

You never see how your supplier forces you to compensate for a limited line; the fewer options, the harder and longer you have to work. And a lack of formulating flexibility can prevent your inks, varnishes or flushes from meeting as wide a range of technical performance demands as possible.

It's high time you see what you're missing–a supplier with more hydrocarbon options. To help you get better technical and profit performance from your formulations.

Behold Exxon. Our EXX-PRINT™ line of solvents and oils for paste inks, varnishes and flushes provides an extensive scope of volatilities, solvencies, viscosities and molecular types. It offers a new standard of formulating flexibility. Making it easier than ever for you to minimize ink-related problems like picking and viscosity build.

EXX-PRINT solvents cover a wide range of hydrocarbon types. From fast evaporating cycloparaffins to tack stability-enhancing isoparaffins. From low-solvency normal paraffins to high-solvency heavy aromatics. Having this many options makes it easier and more economical to precisely customize the basic characteristics of your formulations, whether you're fine-tuning a heat-set ink's drying time or building a varnish's viscosity without increasing its resin content.

Our line of naphthenic and paraffinic oils offers you choices from a water-white process oil to a heavy black newsprint oil. Giving you the options you need for meeting your most demanding

Resin	**Betalite 248**	
Solvent	EXX-PRINT 283 D	Typical Heat-Set Ink Solvent
% Solids	50	50
Laray Viscosity (P)	110	180
Dilutability (gm/100 gm resin)	200	170
Yield Value (dyne/cm)	250	330
Shortness Ratio (yield/visc.)	2	2
Initial Tack (gm-m 400 rpm)	6	6

EXX-PRINT 283 D offers higher dilutability than a typical heat-set ink solvent at comparable initial tack.

ink performance requirements.

Here's an eye-opener: EXX-PRINT products don't require OSHA carcinogen labeling.

Furthermore, all EXX-PRINT products come with our industry-leading technical support. It starts with an expert sales force of knowledgeable professionals. Backed by an extensive ink lab that generates computer models of the effect each solvent structure has on resin solution properties. This helps you determine the optimal solvent composition for specified performance criteria without time-consuming trial and error. There are additional data on resin/solvent interactions, solvency studies, and tack/stability testing. Which brings us to another industry benefit–Exxon's toxicology lab–a service that offers you help on OSHA labeling issues.

The largest variety. The most support. The most determined commitment to serving the ink industry. Now do you see what you're missing?

Discover how having more formulating flexibility helps you meet more ink performance demands. Contact the Exxon Inks Specialists at 1-800-55-EXTRA, or write P.O. Box 60800, Houston, Texas 77205. Seeing is believing.

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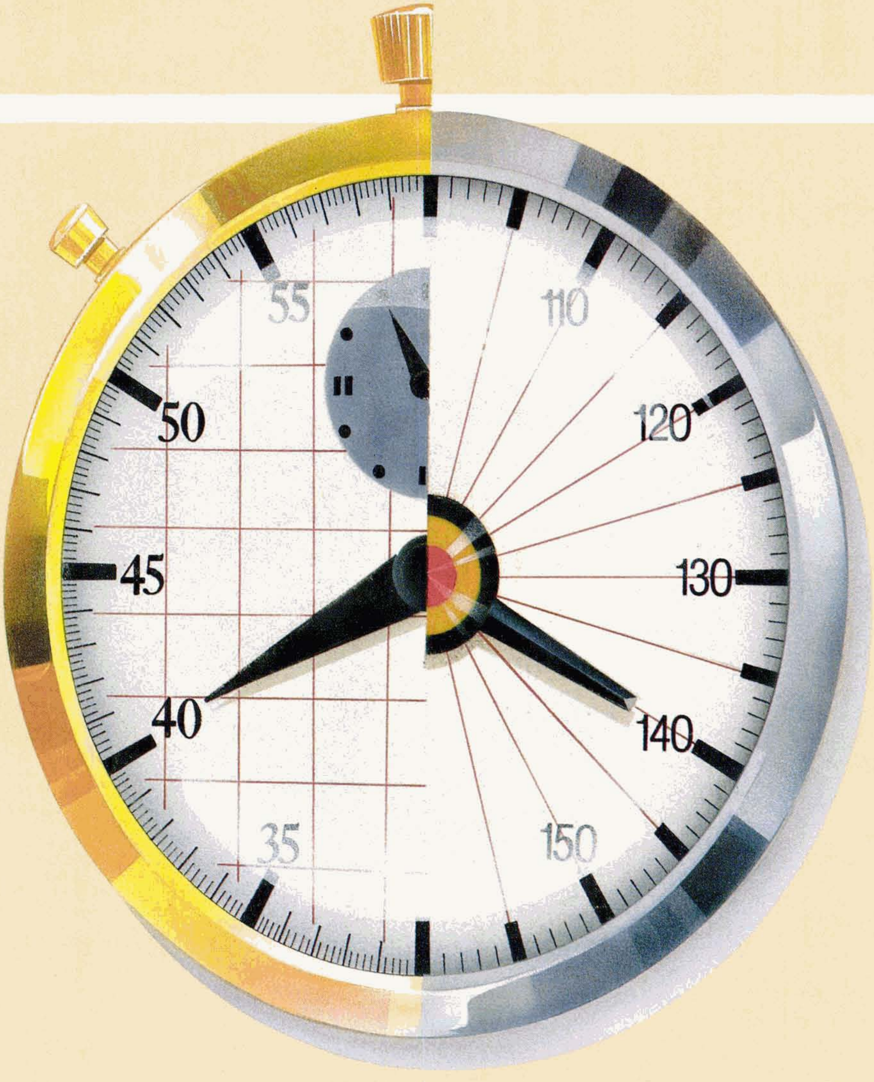

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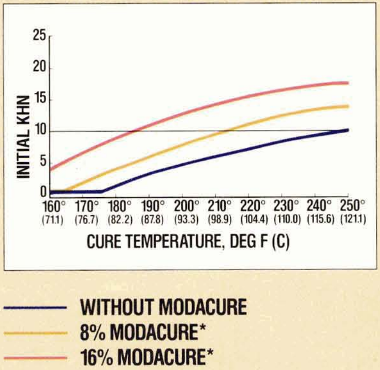

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Abstracts of Papers in This Issue

ASSESSMENT OF PHOSPHATE/POLYMER PROTECTIVE COATINGS ON STEEL. PART I: THE TORSIONAL SHEAR TEST—M.E.R. Shanahan, H. Haidara, and J. Schultz

Journal of Coatings Technology, 59, No. 753, 29 (Oct. 1987)

Conversion coatings on metallic surfaces are gaining importance in many industrial applications. One such frequent application is the treatment of cold-rolled steel sheet prior to polymer coating, aimed at improving both corrosion resistance as well as the aesthetic nature of the final product. In the automotive industry, two main types of phosphate conversion coatings are used to treat steel surfaces: zinc and iron phosphating. The former is particularly successful as far as corrosion protection is concerned, but is sometimes prone to mechanical failure, especially in shear. A test has been developed in order to apply shear stresses to such phosphated and polymer coated systems. It is a modification of the torsional shear test already well known in the context of adhesives testing. Although the maximum stress at failure gives some indication of performance, a more trustworthy and reproducible criterion emerges from this work which is based on a study of the fracture surfaces.

ASSESSMENT OF PHOSPHATE/POLYMER PROTECTIVE COATINGS ON STEEL. PART II: INFLUENCE OF PHOSPHATE THICKNESS—M.E.R. Shanahan, H. Haidara, and J. Schultz

Journal of Coatings Technology, 59, No. 753, 37 (Oct. 1987)

It is known that, under certain conditions, when zinc-phosphating is applied to steel sheet as a corrosion resistant protection, prior to the application of a subsequent polymer coating, mechanical resistance, particularly to shear, is poor. Reported in Part I of this paper was a torsional test developed to elucidate this phenomenon using criteria of maximum failure stress and the type of failure surface observed. The test has, in this study, been applied to two types of zinc-phosphating on steel substrates, differing only by the amount of accelerator present in the phosphatation bath and after electrodeposition of a polymer coating cured under various conditions of time and temperature. Weaker phosphate layers correspond to those with quicker deposition kinetics and greater final thicknesses. When a polymer coating is in place, the overall mechanical tenacity of the surface layers increases with curing condition severity—an effect probably related to polymer penetration within the porous crystalline layer.

ASSESSMENT OF PHOSPHATE/POLYMER PROTECTIVE COATINGS ON STEEL. PART III: INFLUENCE OF PHOSPHATE TYPE AND MORPHOLOGY—M.E.R. Shanahan, H. Haidara, and J. Schultz

Journal of Coatings Technology, 59, No. 753, 45 (Oct. 1987)

The final section of this study of the mechanical properties of phosphate and polymer coatings applied to steel is devoted to the nature and morphology of the phosphate layer. Maximum shear strength, failure surface aspects, and surface microanalysis have been used as the essential criteria for judging systems taken to failure using the previously developed torsional test. An iron-phosphate and a zinc-phosphate-based system have been compared, both in the simple state, and with polymer coatings that have undergone various stages of cure. Whereas the polymer-coated, iron-based system is more resistant to failure than its zinc-based counterpart, the roles are inversed when a polymer is not present. The vast improvement in the performance of the iron-based system would seem to be related to the presence of the polymer itself, helping to produce a compact, composite surface coating, and also to the heating cycle involved. This not only solidifies the polymer, but may facilitate a reconstruction of the phosphate layer leading to better steel/phosphate adhesion.

SOLVENT SELF-DIFFUSION IN POLYSTYRENE-SOLVENT SYSTEMS—F.D. BLUM and S. PICKUP

Journal of Coatings Technology, 59, No. 753, 53 (Oct. 1987)

Solvent self-diffusion coefficients have been measured for a variety of solvents in polystyrene solutions and gels using the pulsed-gradient, spin-echo NMR method. It is shown

that the NMR method can be used to measure diffusion coefficients in a very wide range of polymer systems and the general trends found can be used to estimate diffusion coefficients in other polymer-solvent systems. Diffusion data as a function of composition may then be used to predict the diffusion controlled drying of coatings. In semidilute to concentrated (<80 wt% polymer) solutions it is found that normalized (D/D_o) solvent self-diffusion coefficients follow a "universal" curve as a function of concentration which is independent of the temperature and type of solvent. This allows the prediction of solvent self-diffusion coefficients over a wide range of concentration provided the self-diffusion coefficients for the pure solvents (D_o) at a given temperature are known. In high solids solutions, the solvent self-diffusion coefficients were found to follow Arrhenius behavior with energies of activation increasing with increasing concentration. In polystyrene beads, the self-diffusion coefficient measurements reflected the diffusion of solvent within the bead. It was also shown that the solvent diffusion in the crosslinked system was similar to that found in noncrosslinked systems, suggesting that the crosslinks do not provide an additional impediment to diffusion.

EFFECTS OF DIFFERENT POLYOL-TERMINATED URETHANE PREPOLYMERS ON THE PROPERTIES OF THEIR CORRESPONDING CROSSLINKED FILMS— C-P. Yang and L-T. Lee

Journal of Coatings Technology, 59, No. 753, 61 (Oct. 1987)

Polyglycols (PG) and toluene diisocyanate (TDI) were reacted at various molar ratios to produce isocyanate-terminated polyurethanes and were then reacted with glycerine, pentaerythritol (PE), or trimethylol propane (TMP) to produce hydroxyl-terminated urethane prepolymers of different molecular weights. These prepolymers were mixed with an equivalent amount of blocked isocyanate in a mixture of m-cresol and naphtha to give the varnishes. Pretreated copper wires were coated and baked to give polyurethane coated magnet wires. The mechanical properties of the polyurethane crosslinked films, the change of functional groups during the crosslinking reaction, and the properties of magnet wires coated with polyurethane varnishes are discussed.

STUDY OF THE PARAMETERS AFFECTING THE EMULSION POLYMERIZATION OF VINYL ACETATE— P. Bataille, H. Bourassa, and A. Payette

Journal of Coatings Technology, 59, No. 753, 71 (Oct. 1987)

The emulsion polymerization of vinyl acetate initiated by potassium persulfate in presence of Ag(I) and other metallic ions was studied. During this study, an ionic surfactant, sodium lauryl sulfate, and Igepal® CO-970, a nonionic surfactant, were used. The samples obtained were characterized gravimetrically to obtain the reaction yield, and by GPC for the molecular weight.

It was found that silver ions in conjunction with potassium persulfate, in the presence of either surfactant, accelerates the polymerization of the vinyl acetate.

The use of any metallic salt in the presence of Igepal CO-970 improves the overall conversion.

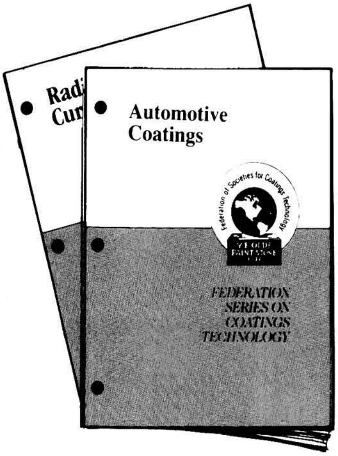
JCT DECEMBER 1987

Post-Convention Issue

On October 5-7, the Annual Meeting and Paint Industries' Show of the Federation of Societies for Coatings Technology will take place at the Dallas Convention Center. Complete coverage of this important event will be published in the December 1987 issue of the JOURNAL OF COATINGS TECHNOLOGY. Follow-up features will include articles on exhibitors, with emphasis on products and special booth features; photo displays of award-winning booths; as well as a complete review of important Annual Meeting and Paint Show happenings.

"Automotive Coatings" Monograph Now Offered In New Federation Series on Coatings Technology

The Federation has announced the recent publication of "Automotive Coatings," the eighth monograph in its continuing Series on Coatings Technology.



The 64-page publication, authored by Bruce N. McBane, features such topics as coating systems, original finish undercoats, elements of original finish topcoats, solvents and diluents, and specialty coatings. The author also focuses on application techniques, pigmentation, automotive refinishing, and coating evaluation and quality control.

Mr. McBane, now a Consultant in the area of industrial coatings development, drew from his 40 years of experience while employed by the Coatings and Resins Division of PPG Industries, Inc.

The new FSCT Series, which will total approximately 35 monographs covering the major areas of coatings technology, will serve as a valuable teaching and training resource for the industry.

The first monograph published in the Series is "Film Formation" by Zeno Wicks, Jr. This 20-page booklet includes discussions on film formation by solvent evaporation from solutions of thermoplastic polymers, from solutions of thermosetting polymers, and by coalescence of polymer particles. Attention is also given to the effect of pigmentation on film formation and testing methods.

"Introduction to Polymers and Resins," by Joseph Prane, is a guide which emphasizes the importance of polymeric materials in the coatings industry. Terminology, classification, types, mechanisms, and structures are among the many topics presented in the 36-page publication.

The subject of radiation curing is explored in the 24-page "Radiation Cured Coatings," written by J.R. Costanza, A.P. Silveri, and J.A. Vona. Emphasis is placed on the technology, equipment, and commercial applications of radiation curing. In addition, material and equipment hazards, storage and handling, personnel protection, and toxicity are presented.

"Solvents," by William Ellis, is a 32-page monograph containing information on solubility parameters, evaporation rates, solvent molecular structures, as well as terpene and oxygenated solvents. Mr. Ellis also focuses on solvents for specific resin types, solvent identification and analysis, and safety and toxicity.

The fifth monograph in the Series is "Coil Coatings," by Joseph Gaske. In addition to terminology and coating types, emphasis is placed on problems in the application and use of coil coatings, the processing of precoated coiled metal, testing, and problems in the manufacture and marketing of coil coatings. The monograph contains 20 pages.

Dr. Wicks also contributed the sixth booklet, "Corrosion Protection by Coatings" to the Series. In this 24-page publication, electrochemical corrosion, corrosion protection by intact coatings and with non-intact film, approaches to formulating corrosion protection coatings, and evaluation and testing procedures are explored.

"Mechanical Properties of Coatings," by Loren W. Hill is the most recent addition to the series. The 28-page monograph introduces the basic concepts involved with the behavior of polymeric materials which help to systematize mechanical property data. Discussion follows on physical property determinations, as well as descriptions of test methods.

Development of the Series is under the overall direction of an Advisory Board, whose members assist in selection of authors and review of manuscripts. Dr. Thomas J. Miranda, of Whirlpool Corp., and Dr. Darlene R. Brezinski, of DeSoto, Inc., are Editors of the Series.

The Series, which is prepared in an attractive 8½ × 11 inch format, designed to fit in a three-ring binder, sells for $5.00 each. Monographs may be obtained by completing the order form in this issue or by contacting Meryl Cohen, FSCT, 1315 Walnut St., Philadelphia, PA 19107.

"Coatings for Wood Substrates" Papers Now Available

Papers presented at the recent seminar on "Coatings for Wood Substrates," sponsored by the Federation of Societies for Coatings Technology, in Seattle, WA, on May 1-2, are available in limited quantities.

The package contains 10 of the presentations given at the seminar. Included are the following:

"Finishing Redwood Exteriors: Challenges and Opportunities"—K. Kersell, The Pacific Lumber Co., Mill Valley, CA

"Plywood Applications, Characteristics, and Finishing Recommendations"—R.A. Carlson, American Plywood Association, Tacoma, WA

"Stains for Wood Siding"—D. Williamson, Olympic Home Care Products Co., Seattle, WA

"The Mildew Problem on Coated Wood Surfaces"—M.C. McLaurin, Buckman Laboratories, Inc., Memphis, TN

"Coatings Research at the Forest Products Laboratory"—W.C. Feist, Forest Products Laboratory, Madison, WI

"Hardboard Siding—Composition and Properties: Painting Recommendations"—T.J. Rieth, Masonite Corp., Towanda, PA

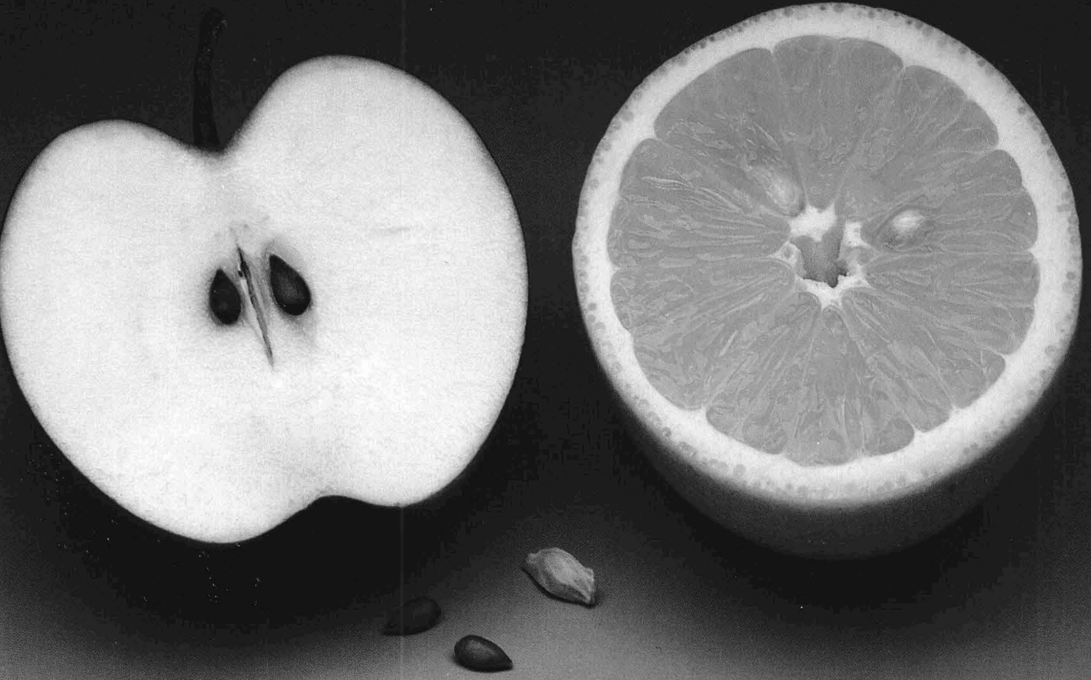
"Painting Hardboard Siding"—S. Vout, The Valspar Corp., Minneapolis, MN

"Coating Wood Furniture—Changes Are Here"—R.S. Bailey, Lilly Industrial Coatings, Inc., Indianapolis, IN

"Preservative Treatments for Wood and Successful Coating Techniques"—A.S. Ross, Koppers Company, Inc., Monroeville, PA

"Investigation of Latex Stain Blocking Primers on Wood Substrates"—F. Marschall, DPI Quality Paints, Inc., Clearwater, FL

Cost of the complete set of seminar papers is $75. To order, contact Ms. Meryl Cohen, FSCT, 1315 Walnut St., Suite 832, Philadelphia, PA 19107, (215) 545-1506.



JOURNAL OF COATINGS TECHNOLOGY

GUIDE FOR AUTHORS

GENERAL

The JOURNAL OF COATINGS TECHNOLOGY is published monthly by the Federation of Societies for Coatings Technology for its membership of approximately 7,000 in 26 Constituent Societies in the United States, Canada, Great Britain, and Mexico. The JOURNAL is devoted to the advancement of knowledge in the science and technology of surface coatings, the materials comprising such coatings, and their use and performance.

The Editors invite the submission not only of regular research papers, but also *Open Forum* comments on subjects of relevant interest, and *Letters to the Editor.* All manuscripts will be assumed to be original work and to have been unpublished elsewhere; not under consideration for such publication; not copyrighted; and to have been submitted for appropriate clearance by the organization with which the author is affiliated if such clearance is necessary. Authors are obligated to reveal any exceptions to these conditions at the time a manuscript is submitted.

The JOURNAL OF COATINGS TECHNOLOGY has first right to the publication of papers presented at the Annual Meeting of the Federation and at local or regional meetings or symposia of the Constituent Societies. ***Papers in which proprietary products or processes are promoted for commercial purposes are specifically non-acceptable for publication.***

SUBMISSION OF MANUSCRIPTS . . .

. . . for the Journal

Technical Papers: Four complete copies should be sent to the Editor, JOURNAL OF COATINGS TECHNOLOGY, 1315 Walnut St., Philadelphia, PA 19107.

If a submitted paper consists of the text of a presentation made previously to a monthly or special meeting of a Society for Coatings Technology, or to another technical group, the name of the organization and the date of the presentation should be given. If someone other than the author of the paper made the presentation, this information, too, should be noted. Papers presented to associations other than the Federation must be released by written communication before they can be considered for publication in the JOURNAL OF COATINGS TECHNOLOGY.

Papers originally composed for oral presentation may have to be revised or rewritten by the author to conform to the style suitable for written publication.

Open Forum: Three complete copies should be sent to the Open Forum Editor, at the address listed above.

The same general rules as given for technical papers should be followed in the preparation of an Open Forum manuscript. However, the subject may be informally approached. Topics may be nontechnical in nature, dealing with any aspect of the coatings industry.

Letter to the Editor: The JOURNAL will consider for publication all correspondence relevant to the coatings industry and to the contents of the JOURNAL. When a letter concerns an article appearing in the JOURNAL, the original author is usually given an opportunity to reply.

. . . by Constituent Societies For Annual Meeting Presentation

Ten complete copies of the manuscript are required for committee review. The set of copies should be addressed to the Editor at the address listed above.

. . .for Roon Foundation Award Competition

Ten complete copies of the manuscript are required, and should be submitted to the Chairman of the 1987 Roon Awards Committee, Gary Gardner, Tnemec Co., Inc., P.O. Box 1749, Kansas City, MO 64141. (For complete details, see "Roon Awards" section of the JOURNAL for January 1987.)

MANUSCRIPT PREPARATION

In general, authors are advised to use the "Handbook for Authors" published by the American Chemical Society as a guide to the preparation of manuscripts (ACS, 1155 Sixteenth St., Washington, D.C. 20036). Another excellent reference work is "How to Write and Publish a Scientific Paper" by Robert A. Day (ISI Press, 3501 Market St., University City Science Center, Philadelphia, PA 19104).

Manuscripts should be typed with double spacing on one side of 8½ × 11 inch (22 × 28 cm) paper, with at least one-inch (2.5 cm) margins on all four sides. All paragraphs should be indented five spaces, and all pages should be numbered at the top center, or upper right corner.

Title

The title should be as brief and informative as possible. Selection of titles that are key word-indexable is a helpful and recommended practice.

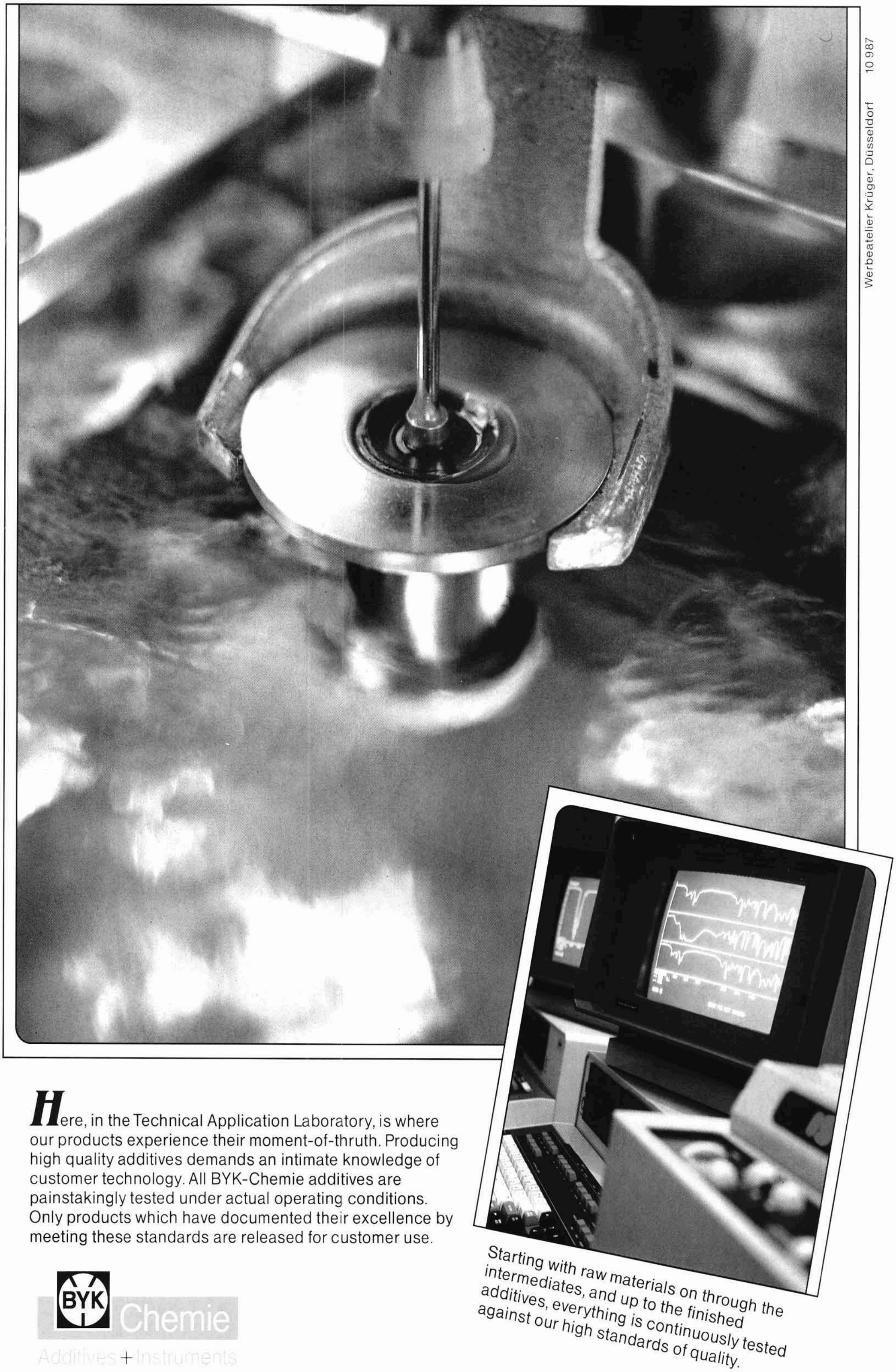
Authors' Biographies and Photographs

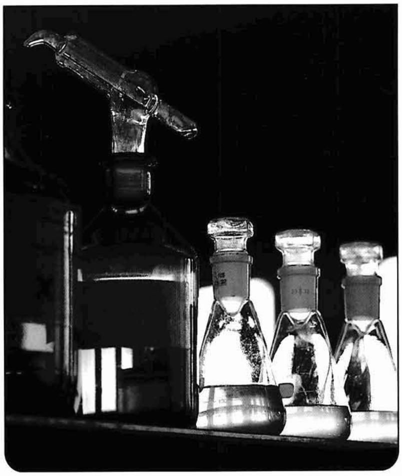
Give complete names, company or institutional affiliations, and brief biographical sketches of all authors. If available, submit a 5 × 7 inch (13 × 18 cm) black-and-white photograph with glossy or smooth high sheen surface, for each author. See later section on photographs for further details.

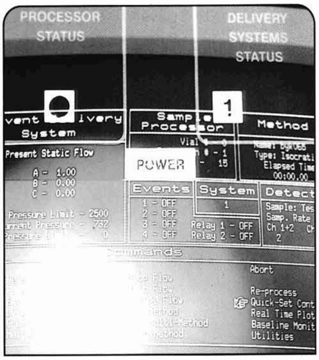
Abstracts

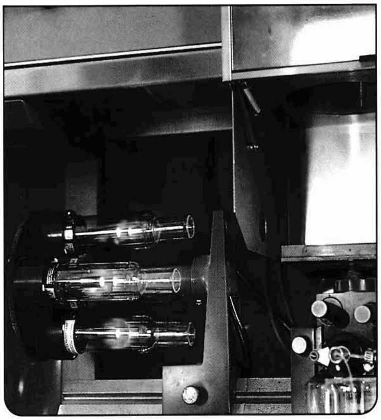
A 75–100 word abstract must be part of the manuscript, and should be a concise description of the key findings or teachings of the work described in the paper. The abstract should not repeat the title or include reference numbers, nor should it duplicate the Conclusion or Summary.

(Continued on page 19)









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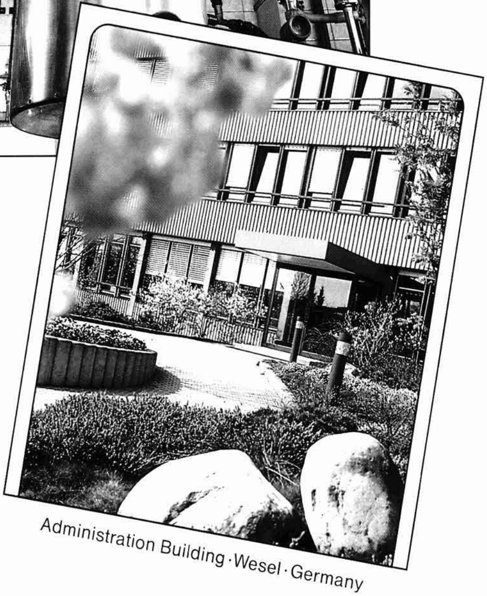

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Text

The headings and sub-headings in this Guide illustrate their use to divide the text into sections to improve readability for comprehension, and to break up typographical monotony; they may be used as a model for preparation of the text of a manuscript for publication. The text should *not* be presented as an alphanumeric outline.

Only as much review as is necessary should be given to provide an introduction to the subject; the main burden for extensive background should be placed on the list of references.

Standard scientific and technical terminology should be used to convey clear and unambiguous meaning, but the use of technical jargon or slang should be avoided. Authors should bear in mind that the JOURNAL has an international audience, for many of whom English is a second, not native, language. Use of regional idioms or colloquialisms should be avoided. The use of obscure abbreviations is also discouraged. When appropriate, abbreviations should be made in parenthesis immediately following first mention of the term in the text, and then used alone whenever necessary.

Recent issues of the JOURNAL should be consulted for desired style and technical level.

Metric System

Metric system units should be used wherever applicable with the equivalent English units shown afterwards in parentheses. The ASTM Metric Practice Guide, E 380-72 (American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103) is a convenient reference.

Tables, Graphs, and Drawings

Tables, rather than descriptive text, should be used only when they are genuinely helpful. They should be proportioned in accordance with the height and width limitations of the JOURNAL'S pages. Each table should be typed on a separate sheet, rather than included in the text, and appended to the manuscript. Each table should be numbered and have a descriptive caption. Tables should be referenced in the text (e.g., "See Table 1").

In numerical data in tables, numbers less than one should have a zero before the decimal point.

Graphs should be on good quality white or non-photographic blue-lined 8½ × 11 inch paper. Each graph should be drawn on a separate sheet, numbered, and the captions listed on a *copy* of the original graph. Graph captions and legends should also be typed on a separate sheet for typesetting.

Graphs should not be used if they merely duplicate the data given in tables, or vice versa.

Drawings should conform to the guidelines given for Graphs and should be proportioned to fit the height-to-width ratio of the JOURNAL'S pages and columns.

Photographs

All photographs should be sharp, clear, black-and-white prints no larger than 8 × 10 inches in size. Photos should be clearly labeled on the reverse side, taking care not to mar the image.

Color prints and slides are unacceptable.

When illustrations are secured from an outside source, the source must be identified and the Editor assured that permission to reprint has been granted.

Nomenclature

Whenever possible, generic names should be used in preference to trade names. When trade names must be used to avoid ambiguity, and the name is a registered trademark, the symbol R, in a circle or parentheses, should be given immediately following, and the manufacturer listed as a footnote. In general, trade names should be used only in footnotes or in an appendix, rather than in the text.

If special nomenclature is used, include a nomenclature table giving definitions and dimensions for all terms.

Nomenclature of chemical compounds should conform to the style of *Chemical Abstracts* and the IUPAC rules.

Equations

These must be typed, or written clearly, with equations numbered sequentially in parentheses to the right. If Greek letters are used, write out their names in the manuscript margin at the first point of use. Place superscripts^a and subscripts$_b$ accurately. Avoid the use of superscripts in a manner that can lead to their interpretation as exponents.

Summary

The paper should be concluded with a summary which is intelligible without reference to the main text. The summary may be more complete than the abstract, listing conclusions drawn from the text. A well written summary can serve to inspire the busy reader to turn back to the paper, to read it thoroughly.

Acknowledgment

If used, it should follow the summary.

References

These should be listed in the numerical order in which they are cited in the text, and should be placed at the end of the manuscript. Names of authors may or may not be shown in the text with reference numbers. If possible, include titles of articles referenced in the literature. The following are examples of acceptable reference citations for periodicals[1,2,3], books[4] and patents.[5]

(1) Pascal, R.H. and Reig, F.L., "Pigment Colors and Surfactant Selection," *Official* DIGEST, *36,* No. 475 (Part 1), 839 (1964).
(2) Davidson, H.R., "Use and Misuse of Computers in Color Control," JOURNAL OF COATINGS TECHNOLOGY, *54,* No. 691, 55 (1982).
(3) Stephen H.G., "Hydrogen Bonding—Key to Dispersion?," *J. Oil & Colour Chemists' Assoc., 65,* No. 5, 191 (1982).
(4) Patton, T. (Ed.), "Pigment Handbook," Vol. 1, John Wiley & Sons, Inc., New York, 1973.
(5) Henderson, W.A., Jr. and Singh, B. (to American Cyanamid Co.), U.S. Patent 4,361,518 (Nov. 30, 1982).

OTHER INFORMATION

Galley proofs will be sent to the author for checking about six weeks prior to publication.

Offprints may be purchased in quantities of 100 or more. Authors will receive price quotations. Each author will receive a complimentary copy of the JOURNAL issue in which his or her paper was published.

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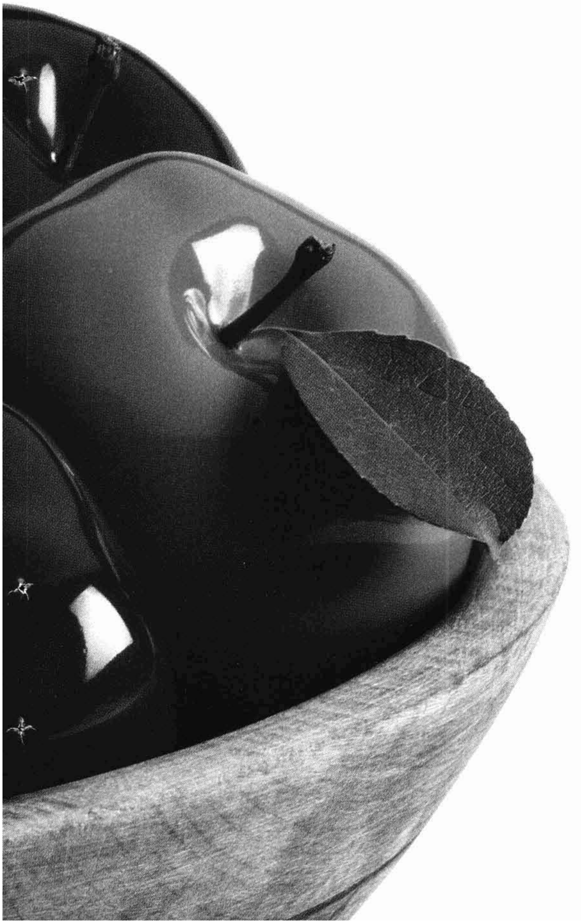

ABC's Jim McKay to Address NPCA Annual Meeting; Forum Sessions Scheduled, Oct. 26-28, in Washington, D.C.

Jim McKay, ABC sports commentator and host of the network's "Wide World of Sports," will be the featured speaker at the final breakfast session of NPCA's Centennial Annual Meeting, October 28 in Washington, D.C.

As the host of "Wide World of Sports," Mr. McKay has traveled some four and a half million miles, covering more than 100 different sports in 40 countries. He has received nine Emmy Awards, including one for his news reporting of the events surrounding the 1972 terrorist attack on Israeli athletes at the Olympic Village in Munich, West Germany. He also earned the George Polk Memorial Award for Journalism and the Officer's Cross Legion of Merit from the West German Federal Republic for his reporting of the tragedy.

Mr. McKay left his job as a reporter for the *Baltimore Sun* in 1947 to work as a writer, producer, director, newsman, sports commentator, and on-camera personality at the then, brand-new WMAR-TV in Baltimore. He joined CBS in New York in the 1950's and anchored the Masters and PGA golf championships, and covered college football, horse racing, and other sports.

Currently, Mr. McKay is the anchorman for ABC on its golf telecasts, as well as the Indy 500 and Kentucky Derby, in addition to his regular appearances on "Wide World of Sports."

He is a graduate of Loyola College in Baltimore, which awarded him a Doctorate of Humane Letters in 1981. A sports enthusiast in private as well as public, Mr. McKay enjoys golf, tennis, and skiing.

Small Paint Manufacturers To Discuss Ownership Transfer

James O. Roberts, President of Management Planning, Inc., will discuss "Transferring Ownership—Consider All the Alternatives," at the Small Paint Manufacturers Breakfast, at 7:30 a.m. on Monday, October 26. Mr. Roberts will examine the many alternatives in transferring ownership, including by sale or merger, buy/sell agreements, or passing the business on to one's children.

Management Information Forum Planned

Representatives of the computer industry, paint companies, and the Federal government will present a forum session on current management information resources and technology on Monday afternoon.

Thomas M. Jewell, President of Research Data Access Corp., Greenville, PA, will address the topic of computerized information management and give an audiovisual presentation on the history, present, and future of computers and their role in helping executives manage the information they are exposed to on a daily basis.

Benjamin Belcher, Jr., of Benjamin Moore & Co.; Merritt Marcus, of Marcus Paint Co.; and Jerome Crowley, of the O'Brien Corp., will discuss current MIC activities in the areas of government data, the "Management Guide to Profitability in the Paint Industry: A New Cost Manual for Decision Making," and other MIC programs and communications designed to benefit industry members.

Attendees will also hear presentations on management information available to the industry from government sources. Speakers include Robert Marske, of the Bureau of the Census; Dixon Tranum, of the Federal Reserve Board; and Vincent Kamenicky, of the U.S. Department of Commerce. Exhibits will include material furnished by the Bureau of Labor Statistics, International Trade Commission, International Trade Administration, Bureau of Economic Analysis, and Bureau of Standards.

Product Liability Impact On the Coatings Industry

"The Real World of Product Liability" as it impacts on the paint and coatings industry will be the topic of a forum session on Tuesday morning. The forum will feature discussions by David Gallagher, Assistant Counsel, for PPG Industries, Inc., and Sara Holtz, Counsel, The Clorox Co.

A half-hour videotape dramatization, "When Products Harm," cosponsored by the American Corporate Counsels Association, will be shown.

Topics for discussion may include: the roles of industry standards, the NPCA Labeling Guide, and state and federal regulation; in-company product safety committees; company record retention programs; making sure it is your product; voluntary safety measures; punitive damages; the effect of marketing claims; effects of remedial actions; alternate formulations; what to do when a complaint is received; and the role of the insurance carrier.

The program, geared to the paint and coatings manufacturer's perspective, will encourage audience participation.

Betz Industrial Opens Conversion Coating Lab

Betz Industrial, Trevose, PA, has opened its new conversion coating lab facilities at its corporate research and development center in Trevose. The new laboratories are equipped to simulate realistic environmental, weathering, and use conditions for the development and enhancement of state-of-the-art conversion coating technologies, including cleaning, phosphating, sealing, and paint adhesion.

Highlighting the facilities is a seven-stage spray washer through which metal panels move on a conveyor line and into a steam-dry oven. The panels can be exposed to a variety of different conditions, such as temperatures, speeds, etc., to test effects of different variables. The laboratories are also equipped to robotically paint phosphated panels and conduct performance testing for paint adhesion and corrosion inhibition.

Included among the laboratory's new equipment are an ED dip tank for electrodepositing paint; a gas-fired and electric oven to cure paints; various chambers to test corrosion inhibition under different environmental conditions; a gravelometer for chipping tests; and an impact tester to evaluate paint adhesion under specific impact levels, as well as bend tests to determine adhesion after metal forming.

A 15-foot long, multitiered outside weather rack provides the closest means possible to simulate real weather and environmental conditions.

The facility is equipped to test both wet and dry paint spray booth treatment processes. Painting can be done manually or robotically. Sludge removal testing is performed with the use of a belt filter.

a celebration . . .

The Members of the

Federation of Societies for Coatings Technology

Extend Their Heartiest Congratulations

To the

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On the Occasion of

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To the Coatings Manufacturing Industry

October 1987

100
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National Paint &
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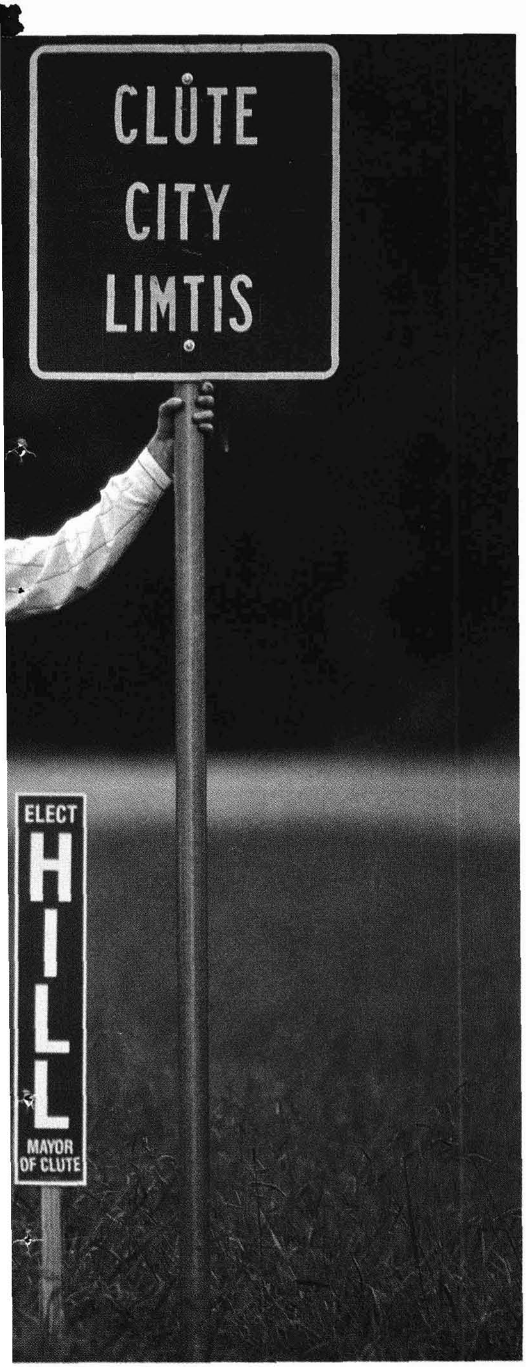
Dave Hill has helped to devel his marine and maintenance cust mayor of Clute, Texas.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DSET Labs Forms COSTAR I Weathering Program

DSET Laboratories, Inc., Phoenix, AZ, recently announced the formation of a multiclient weathering research program to stimulate the development of new tools to design, conduct, and analyze the results of weathering and exposure test programs. The COSTAR I (Correlation of Solar and Thermal Aspects of Weathering) program is designed to provide the participating sponsors the benefits of pooled resources.

COSTAR I will extend and broaden the scope of DSET's pioneering weathering program, which has already developed ultraviolet/total solar radiation regression models.

Major emphasis will be placed on the use of kinetic factors that account for the influence of sample temperature on exposure test results. Using thermally adjusted UV irradiance values in conjunction with the refined and expanded UV-sky models, participants will be able to develop lifetime prediction models for their products based on any geographic region in the world for which climatic data are available.

The program will address the correlation between accelerated and natural exposures, and higher acceleration factors.

DSET has two exposure test sites, one hear Homestead in southern Florida and one in New River, AZ, just north of Phoenix. The New River Laboratories are equipped with controlled environment chambers; EMMAQUA®, QUV, SUNTEST, and Xenotest 1200 accelerated weathering devices; absolute spectral colorimetry; color difference and multiangle gloss instruments; UV-VIS-NIR spectrophotometry (with differential and derivative spectroscopy capabilities); IR and FIR spectrophotometry (including FIR spectroreflectometry and IRS capabilities); scanning electron microscopy; and tensile/elongation test facilities. In addition, DSET operates a complete solar and solar ultraviolet radiometry laboratory.

An annual participation fee for each year of the planned four year research program will be charged.

For more information on COSTAR I, contact DSET Laboratories, Inc., Box 1850, Black Canyon Stage I, Phoenix, AZ 85029.

NL Chemicals Converting Ghent TiO_2 Plant; Chloride Process to Eliminate Waste Acid Disposal

NL Chemicals has announced plans to convert its Ghent, Belgium, titanium dioxide plant from the sulphate process to the chloride process at a cost of more than $50 million.

The current practice of disposing of waste acid into the North Sea will cease and sulphur dioxide emissions to the atmosphere will be reduced by 90% when the conversion is completed in late 1989.

In 1985, NL converted much of its Leverkusen, West Germany, plant to the chloride process, with the remaining sulphate process wastes being handled via waste acid concentration. Deep sea disposal is expected to be eliminated from this construction site by 1989.

The construction of an advanced waste acid concentration facility at NL's Nordenham, West Germany, plant is expected to eliminate 50% of North Sea waste acid disposal by the year's end. The balance is scheduled for elimination in late 1989.

NL Chemicals has produced titanium dioxide at Langerbrugge for 30 years. The present sulfate unit has an annual capacity of 40,000 metric tons. Construction time for the new plant is estimated at two years, and start up is scheduled for late 1989. When the new chloride plant is in production, the sulphate plant will be closed. Thus, the total titanium dioxide capacity at Ghent will remain unchanged.

ASTM Forms New Section: Wear Testing of Coatings

A new section on Wear Testing of Coatings has been formed within ASTM Subcommittee B08.10, Test Methods for Coatings. This new section is to liaison with ASTM Committee G-2, on Wear and Erosion, in identifying and developing appropriate wear tests for coatings.

The tests are needed because several performance requirements for wear on thin coatings, 0.5-250 microns, are being considered for new coating specifications. Among the coatings are alloys of metal matrix composite coatings and thermal spray coatings. Testing for the wear performance of these coatings requires wear scars of less than four microns at the conclusion of the test.

The next meeting of the Wear Test and Coatings group takes place this month at the ASTM Headquarters in Philadelphia, PA. The topic of discussion will be a draft document of a Guide for the Testing of Wear of Coatings. The guide will focus on the characterization of four types of wear: adhesive, abrasion, fretting, and erosion. Also intended for inclusion in the Guide are the test methods for coatings which can be used to evaluate their performance.

Additional work will be planned to collect test methods used by industry and to develop a plan to demonstrate their precision and accuracy. Methods which demonstrate good precision and accuracy will be submitted to Committee G-2 for their analysis and eventual balloting into an ASTM standard test method.

For more information, contact Phillip Stapleton, Stapleton Co., 1350 W. 12th St., Long Beach, CA 90813.

Glidden, Shinto Paint Co. Sign Licensing Agreement

The Glidden Co., an ICI Company, Cleveland, OH, and the Shinto Paint Co., Osaka, Japan, jointly announced the signing of a technical licensing agreement, naming Glidden the North American licensee of Shinto's cathodic electrodeposition (CED) coatings.

The agreement makes Glidden the only company in the United States to manufacture and supply the CED technology which is approved and used in Japan. Shinto, a coatings supplier to the automotive and appliance industries in Japan, is a licensee of Glidden's powder coatings.

Glidden plans to target the CED coatings for application on components such as wheels and under-the-hood parts.

The Glidden Co. was acquired by Imperial Chemical Industries (ICI) PLC, London, England in 1986.

KaiserTech Seeks Interest In Harshaw/Filtrol Partnership

KaiserTech Limited, Oakland, CA, has announced its intention to purchase Chevron Corp.'s half interest in the Harshaw/Filtrol Partnership. Terms of the purchase are not being disclosed. Harshaw/Filtrol, a specialty chemicals and high-technology company headquartered in Cleveland, OH, is managed by Kaiser Chemicals, a division of KaiserTech's Kaiser Aluminum & Chemical Corporation subsidiary.

The partnership was formed in 1983 by combining the assets and operations of Harshaw Chemical Co. (then owned by Gulf Oil) and Kaiser's Filtrol Corp. By acquiring Gulf in 1985, Chevron became a partner with Kaiser in Harshaw/Filtrol.

In 1986, Harshaw/Filtrol had an annual revenue of about $264 million. The company has approximately 2100 employees at 19 facilities worldwide.

The partnership agreements include an option for Kaiser to buy the Harshaw stock from Chevron at any time prior to July 26, 1989.

Test panels painted with high-solids acrylic white enamel using different solvents.

◄ Exxate 600 formulation

MAK ► formulation

Exxate® solvent outshines MAK in high-solids baked coatings

Recently, a series of highly controlled, repeatable tests was conducted to compare the performance of Exxon Chemical's Exxate 600 solvent with methyl amyl ketone (MAK) when used as both tail and polymerization solvents in otherwise identical high-solids acrylic coatings. Among the coating properties evaluated were appearance and durability. Here's how the two solvents compared.

Appearance

As shown by the reflections in the photograph above, the Exxate 600 coating has better gloss than the MAK coating. The 20° gloss measurements were: Exxate 600 = 80, MAK = 62. In a color comparison of clear coatings, the Exxate 600 coating scored an excellent Pt-Co color of 40; the MAK coating measured 150 Pt-Co.

Durability

The test panels in the photograph also demonstrate the superior durability imparted by Exxate 600. In direct-impact tests, the Exxate 600 coating withstood an impact of almost 120 inch-pounds without breaking; the MAK coating withstood less than 80 inch-pounds.

Exxate solvents have much more to offer than just superior coating properties. Here are some other key performance comparisons.

VOC emissions

When Exxate 600 is used as both the tail and polymerization solvents instead of MAK, higher solids content can be achieved, resulting in lower volatile organic compound (VOC) emissions. Also, all Exxate solvents are Rule 66 exempt.

Electrical resistance

Exxate solvents offer a unique combination of the high solvent power of an oxygenated solvent plus the high electrical resistance of the hydrocarbon solvents typically used in low-solids coatings. That means you can now formulate high-solids coatings that meet EPA standards and still get the transfer efficiencies made possible by high resistivity. Exxate 600 has a resistance of more than 20 Ransburg Megohms compared to MAK's resistance of about 1.0 Ransburg Megohm.

Cost

Coatings formulated with Exxate 600—with superior properties, lower VOC's and improved transfer efficiencies—*cost about the same* as coatings formulated with MAK.

Full technical details

Complete details on how the comparative tests were conducted and on these and other properties of Exxate solvents—wetting action, evaporation rate, solvent release rate, chain transfer activity, oxidative stability, water miscibility, and more—are yours for the asking.

Call your sales representative or contact Jackie Reynolds at Exxon Chemical Company, P.O. Box 3272, Houston, Texas 77253-3272. Phone 713/870-6377.

EXXON
CHEMICALS

Assessment of Phosphate/Polymer Protective Coatings on Steel
Part I: The Torsional Shear Test

M.E.R. Shanahan, H. Haidara, and J. Schultz[†]
Centre de Recherches sur la Physico-Chimie des Surfaces Solides*

Conversion coatings on metallic surfaces are gaining importance in many industrial applications. One such frequent application is the treatment of cold-rolled steel sheet prior to polymer coating, aimed at improving both corrosion resistance as well as the aesthetic nature of the final product. In the automotive industry, two main types of phosphate conversion coatings are used to treat steel surfaces: zinc and iron phosphating. The former is particularly successful as far as corrosion protection is concerned, but is sometimes prone to mechanical failure, especially in shear. A test has been developed in order to apply shear stresses to such phosphated and polymer coated systems. It is a modification of the torsional shear test already well known in the context of adhesives testing. Although the maximum stress at failure gives some indication of performance, a more trustworthy and reproducible criterion emerges from this work which is based on a study of the fracture surfaces.

INTRODUCTION

Over the last decade or so, a great development has taken place in the field of conversion treatments for metal surfaces. This development is directly related to both the economic needs and the increasing demand for special surface treatments in technology. Several aspects are clearly important in this general domain, but the present study is devoted essentially to the phosphate treatment of steel sheet destined to improve corrosion resistance. Such treated steel is generally coated with one or more polymeric layers both to improve further resistance to environmental attack and to render more aesthetic the appearance of the finished product. We have therefore studied essentially steel/phosphate/polymer systems in the present work, although results from experiments using incomplete systems (e.g., without any polymer coating) have proved useful in the interpretation.

As far as the automotive industry is concerned, two major types of phosphate treatment are used, these being generally termed iron and zinc phosphating. The latter tends to present better corrosion resistance but its mechanical resistance, particularly in shear, is at times questionable. Consequently, an essential part of the present study was the development of mechanical tests capable of applying shear stresses as pure as possible to the interfacial steel/phosphate/polymer zone.

The great range of test methods developed to study the adhesion and/or cohesion properties of polymer layers and films on various types of substrate is a consequence of the complexity of the problem.[1-9] Among the shear tests commonly used for rigid polymers, probably the most frequently employed is that of the single lap joint. Although relatively easy to use experimentally, the lap joint suffers from complex internal stress distributions and can therefore be difficult to interpret. In the present work, two tests have been developed. One of these, based on the displacement of a metal block bonded to steel sheet and parallel to the interface has already been described.[10] The work reported here, however, involves the adaptation of an already well known test, that of torsional shear.[11-12] Apart from the evaluation of maximum stress at failure as a criterion of resistance, a second aspect, the facies of failure, also will be seen to be of great value.

---

*24 Ave. du Président Kennedy, 68200 Mulhouse, France.

[†] Laboratoire de Recherches sur la Physico-Chimie des Interfaces de l'École Nationale Supérieure de Chimie de Mulhouse, 3 Rue Alfred Werner, 68093 Mulhouse Cedex, France.

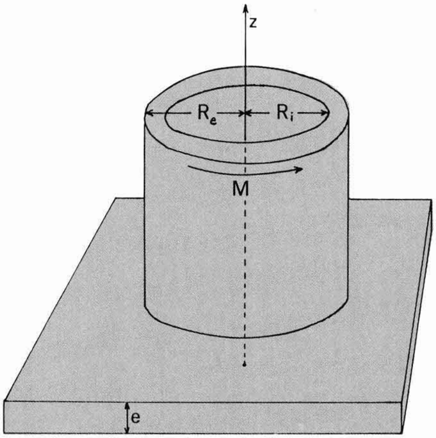

Figure 1—Cylinder bonded to flat plate

EXPERIMENTAL

The torsional test is well established and has been used for many years to evaluate the mechanical properties in shear of adhesives.[11,12] In this form, two hollow circular cylinders are bonded together and a torsional couple, M, applied to the system. If the external and internal radii of the cylinders are R_e and R_i, respectively, it can be readily shown that the maximum elastic shear stress, τ_{max}, is developed at the outer radius and is given by:

$$\tau_{max} = 2\,M\,R_e/\pi\,(R_e^4\text{-}R_i^4) \qquad (1)$$

The analysis leading to equation (1) assumes R_e and R_i to be similar, otherwise certain radial stresses due to Poisson's ratio effects will be generated.

This same method can be used to investigate the surface coating resistance of sheet material by bonding a cylinder to a flat expanse of the sheet (see *Figure* 1). The essential outcome of the analysis is the same as for the two-cylinder test but a correction factor should be incorporated into equation (1) to account for a slight resistance to rotation coming from the sheet material outside the confines of the bonded cylinder.[13] The corrected value of the maximum shear stress, τ_{max}*, is then given by:

$$\tau_{max}^* = \tau_{max} \cdot z/e = 2\,M\,R_e\,z/\pi e\,(R_e^4\text{-}R_i^4) \qquad (2)$$

where e represents the overall thickness of the sheet/coating system and z represents the distance from the far side of the sheet (i.e., without cylinder) to the locus of failure. For the present work this correction is found in all cases to be less than 5% and is therefore neglected.

Guided by a choice of material found in the literature,[14] aluminum cylinders of specification AU4G were machined to obtain R_e and R_i of 10 mm and 5 mm, respectively. Before bonding, the aluminum cylinders underwent the following preparation:

—light abrasion with 600 grade emery paper;
—compressed air jet to eliminate abrasion debris;
—trichloroethylene extraction for ca. 1 hr;
—drying for 10 min at 60°C;
—sulfo-chromic (H_2O - 30 parts, H_2SO_4 - 10 parts, $K_2\,Cr_2\,O_7$ - 1 part) attack for 20 min at 65°C;
—immersion in cold water for ca. 20 min; and
—final drying at 65°C.

The polymer covered steel plates used here were prepared by very light abrasion of the region to be bonded, followed by methanol rinsing and drying at 60°C for 20 min. Assembly of the cylinder/plate systems was effected as quickly as possible using an epoxy adhesive; Araldite™ AW 106/HV 953 U (CIBA-GEIGY) cured for 30 min at 100°C and at a pressure of 0.15 MPa. After assembly, the glue fillets surrounding the cylinder were carefully removed with a scalpel for most of the tests. The completed assemblies were mounted in the apparatus shown in *Figure* 2. Using a system of pulleys, the apparatus transformed the linear motion imposed by the jaws of a tensile testing machine into a rotational movement applying a couple to the bonded cylinder. Maximum shear stress is calculated from equation (1). Tests were effected at room temperature.

The work presented here involves two types of systems: cold-rolled steel covered directly with an electrodeposited polymer, and cold-rolled steel, zinc-phosphated and then polymer treated. In our nomenclature, Z represents zinc-phosphating and T represents no phosphate layer. Electrodeposition is represented by E. The zinc-phosphating employed corresponded to a layer of 2.3 gm/m^2 and the polymer was a "corona cataphoresis"—a black polymer electrochemically deposited on the substrate with the latter acting as cathode. The curing conditions were 160°C for 10 min, represented by 0, and 190°C for 13 min, represented

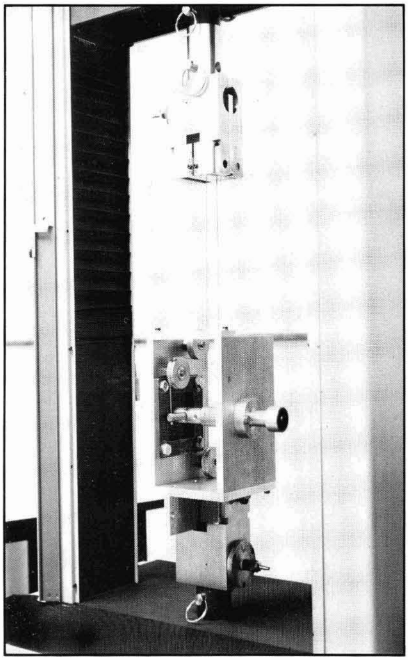

Figure 2—Torsional shear apparatus mounted in tensile tester

by 1. Thus with this nomenclature, the systems considered here are E0/Z, E1/T, and E1/Z.

Table 1—Data Related to Maximum Shear Stress at Failure And Failure Surfaces of Three Coated Steel Systems

System	Test Rate (mm. min^{-1})	τ_{max} (MPa)	Failure Surface
E1/T	0.01	37.4 ± 0.6	50-100% Interfacial Polymer/steel
E1/Z	0.01	39.0 ± 0.5	15-30% Interfacial Polymer/phosphate
E0/Z	270	52.3 ± 1.6	80-90% Interfacial Polymer/phosphate
E1/Z	270	54.5 ± 0.5	15-30% Interfacial Polymer/phosphate

RESULTS AND DISCUSSION

Table 1 represents a summary of experimental results obtained from torsional tests of the systems E0/Z, E1/T, and E1/Z and at test rates (defined by the linear deformation rate at the outer cylinder radius) of 0.01 and 270 mm. min^{-1}. Apart from presenting mean figures for maximum shear stress at failure, we give, in the last column, assessments of the type of failure surface observed. These surfaces were effected by planimetry. Clearly there is a significant increase in failure stress corresponding to a vastly increased test rate (to be expected for systems involving polymer fracture), but stress differences between systems at a given test rate are very small, if not insignificant. However, the results clearly show that failure surfaces very definitely depend on the type of surface treatment. In the cases of E1/T and E0/Z, failure is essentially at the weak junction between the steel itself (or phosphate coating) and the insufficiently adherent coating of polymer. *Figure* 3 shows the cylinder and the steel sheet failure surfaces for a typical case of failure for system type E1/T. The predominant failure zone is clearly such that the major part of the electrodeposit is torn from its steel substrate. Results for E0/Z are similar. However, the E1/Z failure surfaces were found to be consistently mixed, with something like only 30% of the failure taking place at the polymer/phosphate interface, the remainder being essentially within the polymer and therefore of a cohesive nature. *Figure* 4 is a schematic representation of a typical E1/Z failure. Due to its high degree of reproducibility, we have attempted to account for the steps involved in this type of failure. There are essentially four zones of failure as denoted in *Figure* 4.

Zone (i)

This zone of failure is either in the electrodeposit (E) or the electrodeposit/adhesive (A) interface. Its existence depends on the test-piece preparation. It is an edge effect in which the initiation of failure occurs (highest stress). If the inevitably present fillet of adhesive around the circumference of the cylinder at the cylinder/sheet interface after crosslinking is left intact, zone (i) rarely appears. Nevertheless, the overriding majority of experiments was conducted with the fillet removed by scalpel blade. When the fillet is cut, the cutting blade exerts a force perpendicular to the plate surface such that there is probably a tensile stress applied to the adhesive/electrodeposit interface. This tensile stress will tend to weaken the interface and the nearby material, and corresponds to the suspected

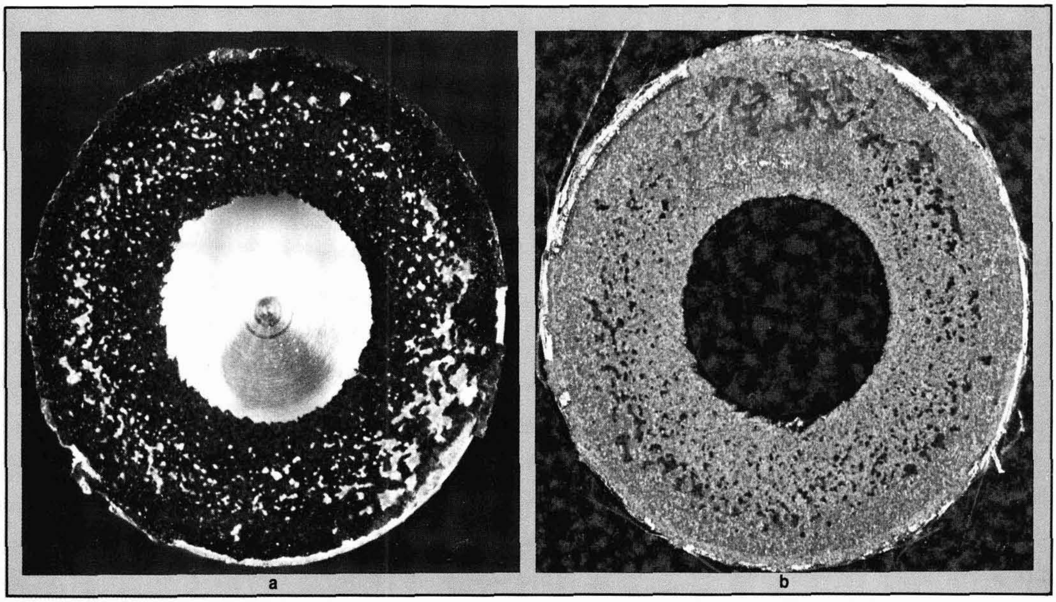

Figure 3—Typical failure surfaces for the E1/T system: (a) cylinder; (b) steel sheet

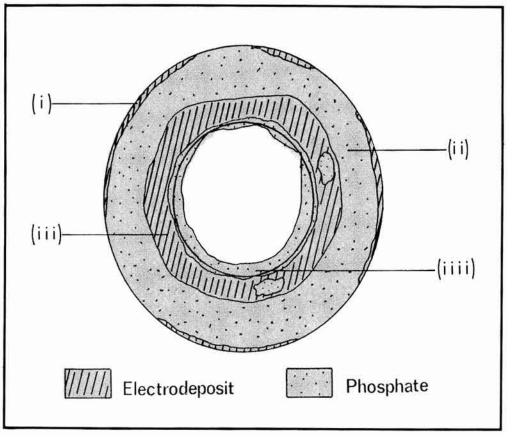

Figure 4—Schematic representation of the four zones of E1/Z failure as found on the sheet

cause of the interfacial adhesive/electrodeposit or cohesive electrodeposit failures observed. Experiments performed with and without adhesive fillet tend to suggest that there is no overall effect on the other failure zones. The locus of failure in zone (i), and the other zones, is shown schematically in *Figure* 5.

Zone (ii)

Zone (ii) is of more significance. It corresponds to failure taking place essentially at the phosphate (P)/electrodeposit interface; the majority of the polymer being retained on the aluminum cylinder after failure. The interpretation is simply that in this zone, and for the given conditions of temperature, stress, and strain rate of all the interfaces present, the weakest corresponds to that of the phosphate/electrodeposit. The area covered by this zone, as we shall see, depends on the coating/substrate adhesion and the viscoelastic properties of the polymer.

Zone (iii)

This zone corresponds to a changeover. In this area, failure is essentially interfacial adhesive/electrodeposit or within the electrodeposit. The sudden increase in stress conditions, resulting from the reduction of effective radius of mechanical attachment of the annulus, involves a drop in the dissipative characteristics of the polymer phase (cf., effects of shock). The polymer will thus tend to exhibit a rather fragile, or brittle, behavior instead of the more frequently observed tough aspect. Under these conditions, the adhesive/electrodeposit polymer system probably becomes less resistant than the phosphate/electrodeposit interface.

Zone (iiii)

This again is an edge effect of little significance. The final separation between the cylinder and the plate will take place under catastrophic conditions. In addition, there is an inevitable fillet of adhesive near this region. Zone (iiii) corresponds to a perturbation and is of little interest in our failure analysis.

Transition Phases

Clearly zones (i) and (iiii) are of no great use, but the relative proportions taken by zone (ii) and zone (iii) type failure can be used as a useful and reproducible criterion of substrate/coating resistance to shear. The relative surface covered by zone (ii) depends on the characteristics of the conversion phosphate layer on the steel and on the conditions of treatment of the electrodeposit. These traits determine the equilibrium between the phosphate/electrodeposit interface and the polymeric phase, or more specifically, the transition between failure at the former and failure in the latter. For *relatively* low stress conditions, such as those obtained when the failure front is near the external radius of the cylinder, the polymer system (electrodeposit and adhesive) is capable of large deformations without failure and it is thus the mineral or phosphate layer which is more prone to failure. However, at higher levels of stress, whereas the mineral components present virtually identical resistance characteristics (elasticity), the polymer components are less able to absorb the shock and thus correspond to the weaker zone (viscoelasticity). The overall result is a failure path which originally was in the mineral layer, changing over and continuing preferentially in the polymer layer. The nearer this transition takes place to the outer radius, the more resistant the steel/phosphate structure. This hypothesis seems to be borne out by the experiment (both results presented here and those to be presented later). It is generally known that the steel/polymer system (E1/T) presents poor adhesive properties as does the E0/Z system for reasons of undercure of the polymer. The percentage of zone (ii) failure is very high. The E1/Z system, although far from being the best studied, is clearly vastly superior with zone (ii), representing only ca. 30% of the failure surface.

Figure 6 is a photograph obtained by scanning electron microscopy of the transition from zone (ii) to zone (iii)

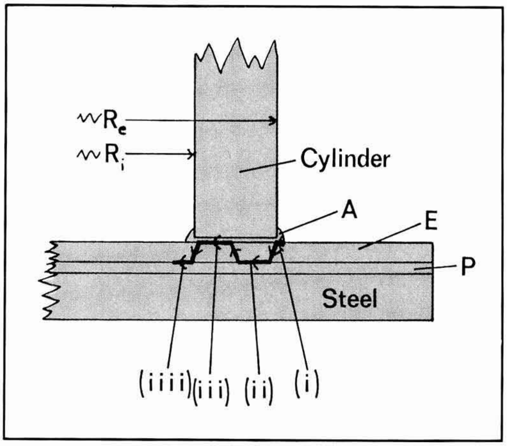

Figure 5—Schematic representation of locus of failure following the four zones

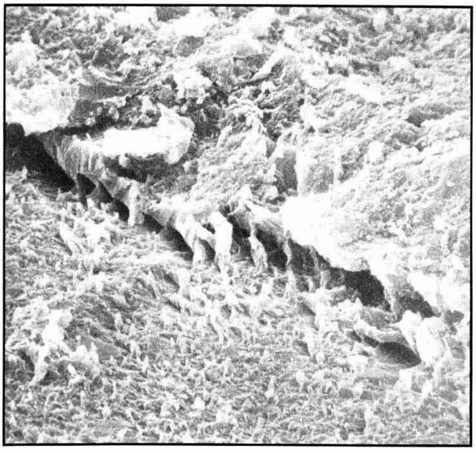

Figure 6—Scanning electron microscopy photograph of the transition from zone (ii) to zone (iii) failure on the steel sheet (see text)

type failure and clearly shows the step taken by the failure locus. This transition is accompanied by plastic deformation of the polymer phase related to sudden increase in local stresses.[15] There is clearly some formation of fibrils in the polymer phase.

CONCLUSION

A torsional test has been developed to characterize the relative resistance of polymer and phosphate protective coatings on steel sheet under conditions of shear. The obvious criterion for performance would seem to be the maximum stress at failure. Although failure stresses are quite clearly related to test rates, they give relatively little information on the relative quality of the substrate/coating adhesion of different systems. A far better and reproducible criterion for coating performance is based on the aspect of the failure surfaces. An exterior zone of the annular interface usually fails at the phosphate/polymer interface. The smaller this zone is, the better are the shear resistance characteristics of the surface layers. This fact is related to the essentially elastic and therefore rate-independent nature of the mineral layers, and the basically viscoelastic, rate-dependent qualities of the polymer components. Under *relatively* low stress conditions, the mineral layer tends to be less resistant to shear, whereas under higher stress conditions, failure takes place preferentially within the polymer components.

ACKNOWLEDGMENTS

The authors are indebted to the Régie Renault for both their financial support and permission to publish this work.

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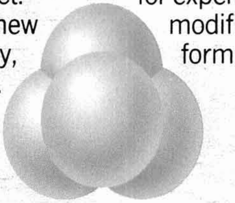

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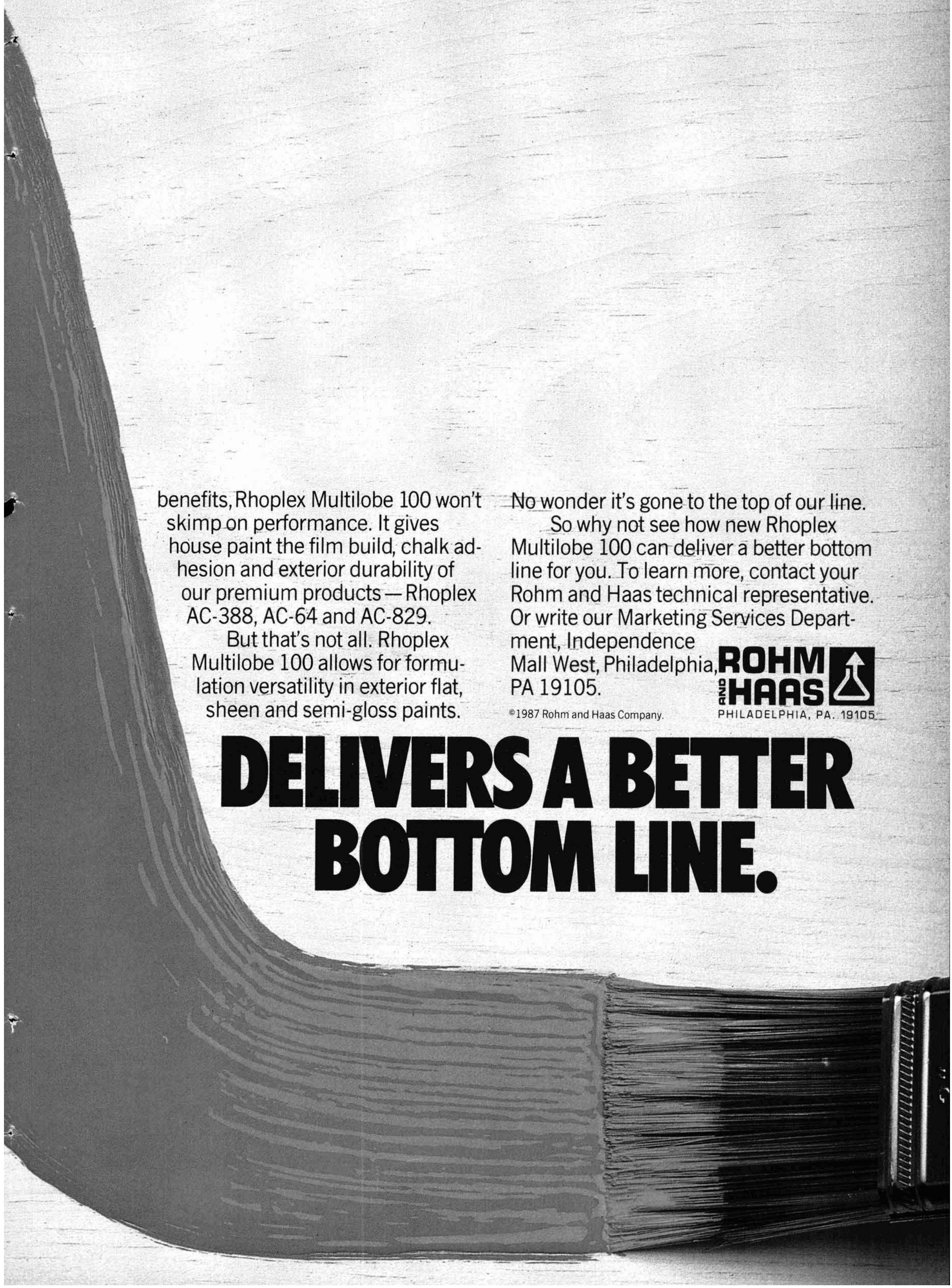

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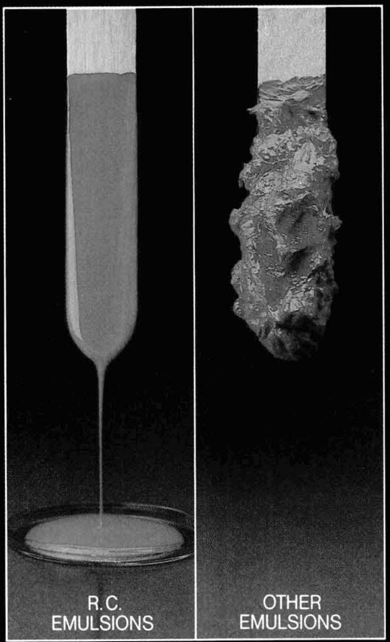

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Assessment of Phosphate/Polymer Protective Coatings on Steel
Part II: Influence of Phosphate Thickness

M.E.R. Shanahan, H. Haidara, and J. Schultz
Centre de Recherches sur la Physico-Chimie des Surfaces Solides*

It is known that, under certain conditions, when zinc-phosphating is applied to steel sheet as a corrosion resistant protection, prior to the application of a subsequent polymer coating, mechanical resistance, particularly to shear, is poor. Reported in Part I of this paper was a torsional test developed to elucidate this phenomenon using criteria of maximum failure stress and the type of failure surface observed. The test has, in this study, been applied to two types of zinc-phosphating on steel substrates, differing only by the amount of accelerator present in the phosphatation bath and after electrodeposition of a polymer coating cured under various conditions of time and temperature. Weaker phosphate layers correspond to those with quicker deposition kinetics and greater final thicknesses. When a polymer coating is in place, the overall mechanical tenacity of the surface layers increases with curing condition severity—an effect probably related to polymer penetration within the porous crystalline layer.

INTRODUCTION

Examination of the literature pertaining to protective conversion coatings on metal, and especially phosphating on steel, shows that phenomena related to the germination and growth of the layers and their physico-chemical characteristics are of fundamental importance.[1-8] The morphology and nature of the phosphate layers constitute a means of anchoring the organic, polymeric coatings applied to the mineral surface, particularly in the automotive industry.

The final systems, constituted of steel/phosphate/polymer(s), must be able to withstand various effects of the environment, such as mechanical and thermal stresses and, to some extent, chemical attack. Differences in coefficients of thermal expansion, compressibility, elastic modulus, and resistance to shock can all lead to stresses of a tangential nature, especially near the interface substrate/coating. It is generally accepted that polymer coated, zinc-phosphated steel is more prone to such shear stresses than its iron-phosphated counterpart. After having developed a torsional test capable of giving reproducible data concerning such coated steel in Part I of this paper,[9] we devoted this second part of the study to the influence of mechanical resistance on the conversion coating thickness.

EXPERIMENTAL

The basis of this part of the study is to compare the intrinsic cohesion/adhesion properties of two layers of phosphate when applied to steel. Both layers of phosphate have the same nature but a different average thickness. A comparison can be made of the results coming from systems both with and without a polymer coating.

The conditions of preparation of the cylinder/plate test pieces and the experimental procedure concerning the torsional test used to assess mechanical performance in shear have been presented in Part I of the paper.[9]

The substrate consists of 0.7 mm thick cold-rolled steel covered with a phosphate layer. The production and the characteristics of the two phosphate coatings considered here are summarized in *Table* 1. In the case of polymer coated steel/phosphate systems, the polymer used was the previously mentioned corona cataphoresis—a cationic resin deposited on the mineral surface in an electro-

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Figure 1—SEM photograph of failure surface (cylinder) of the Z2 system

chemical bath with the substrate as cathode.[9] The average polymer layer thickness was 20 µm. Curing of the polymer was effected under various conditions: at 190°C for 13 min (nomenclature 1); at 200°C for 10 min (nomenclature 2); and at 220°C for 20 min (nomenclature 3).

As before, we shall represent electrodeposition by E and zinc-phosphating by Z. For example, zinc-phosphated steel number 1 (*Table* 1) without polymer is represented by Z1, with polymer coating cured at 200°C for 20 min, this becomes E3/Z1.

Table 1—Production Conditions and Characteristics Of the Two Zn Phosphate Coatings Considered

Pre-phosphatation preparation of steel substrate	Alkaline degreasing: aspersion-immersion; aspersion cycle: 5 min at 60°C Water rinse Affiner rinse by aspersion
Phosphatation:	
Type	Zinc
Method	Immersion in bath
Conditions	3 Min at 55°C
Accelerator	ClO_3^- (conc. higher for Z2 than Z1)
Post-phosphatation	Water rinse Chromic rinse by immersion Heating at 100°C for 2 min
Type of Phosphate: (x-ray, infra-red, x-ray fluorescence):	Phosphophyllite, $Zn_2Fe(PO_4)_2 \cdot 4H_2O$
Weight of deposited layer and estimated thickness[10]	Z1: 2.3 gm/m^2, 1 µm Z2: 4.5 gm/m$_2$, 2 µm

The difference in final thickness is due to different concentrations of accelerator (ClO_3^-) in the phosphate bath.

Table 2—Torsional Test Results for Zinc-Phosphated Steel

System	Test Rate (mm min^{-1})	τ_{max} (MPa)	Failure Surface
Z1	270	46.1 ± 2.6	See text
Z2	270	42.0 ± 1.5	See text

RESULTS AND DISCUSSION

Results for the various systems obtained from the torsional test are presented in *Tables* 2-4 and in the format of the previous paper.[9]

Intrinsic Cohesion of Phosphate Layers Without Polymer Coating

As can be seen from *Table* 2, the maximum shear stress at failure is a rather poor guide to mechanical strength of the steel/phosphate systems, especially given the standard deviation of the results. Due to the limitations of this criterion, it was considered that an analysis of the failure surfaces was more valuable. The analysis was carried out principally using a scanning electron microscope (JEM 100 B) with EDAX option, and an electronic microprobe, Casting. The micrographs and analyses presented here refer to the failure surface on the detached aluminum cylinder after torsional testing. With the essential composition of the phosphate layers known,[10] phosphophyllite, Zn_2 $Fe(PO_4)_2$, $4H_2O$ analysis was carried out using the spectral lines of Zn, Fe, and P.

The analysis is unequivocal since neither adhesive or cylinder contained sufficient amounts of these elements to be detectable. In order to minimize the uncertainty of analysis related to testing very small areas of failure surface, in most cases, three specimens from different parts of a same test piece were studied. A correlation was obtained between the microanalysis and the macroscopic aspect of the failure surfaces. It was noticed that, after failure, virtually the entire substrate surface of the Z2

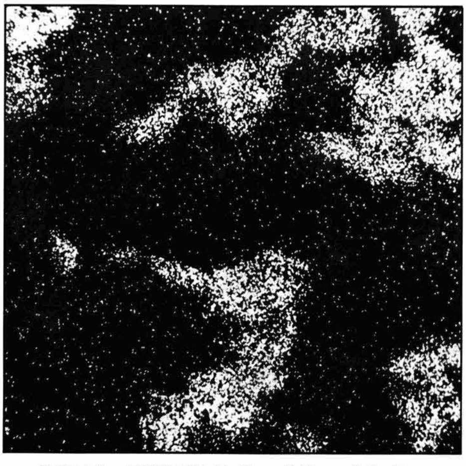

Figure 2—EDAX distribution of *Figure* 1 for Zn

Table 3—Torsional Test Results for Polymer-Coated, Zinc-Phosphated Steel

System	Test Rate (mm min^{-1})	τ_{max} (MPa)	Failure Surface (% interfacial) polymer/phosphate)
E1 / Z1	270	54.5±0.5	15 – 30
E1 / Z2	270	46.1±0.4	Ca. 50

system presented a rather brilliant or shiny aspect compared to the initial phosphated reference steel.

Scanning electron microscopy (SEM) and EDAX observations of the conjugate cylinder surfaces systematically revealed images and spectra of the type exemplified in *Figures* 1 to 4. This was found to be the case irrespective of whether the fracture surface near the outer or near the inner radius of the cylinder was considered. The SEM photo in *Figure* 1 is remarkable for the light-colored areas, seemingly in relief. The EDAX distributions of Zn, Fe, and P, shown in *Figures* 2-4, coincide with these areas. Although the combination SEM/EDAX is incapable of producing a true quantitative analysis, the reproducibility of microanalyses effected for different areas of the fracture surface, the correlations between the light-colored areas in SEM, and the elemental analysis results lead without a shadow of doubt to the conclusion that platelets of phosphate are torn from the steel surface and left embedded in the adhesive layer on the cylinder.

The validity of the correlation between the microanalysis and macroscopic aspect of the failure surface when appraised from its shiny appearance, has also been confirmed for the Z1 system. In general the failure surfaces of the Z1 system present two zones: (1) an exterior annulus near R_e for which the steel substrate is shiny and the width of which is ca. 1 mm; and (2) the rest of the contact zone plate/cylinder for which the plate remains matte gray.

Analysis by EDAX shows that on the cylinder side, the latter zone contains only very small traces of Zn, Fe, and P while the exterior annulus shows, as is the case for Z2, a high proportion of these elements. In fact, SEM leads one to the observation that failure is mixed in this region, taking place partially in cohesion within the phosphate layer and partially interfacially between both the phosphate layer and the steel substrate, and the phosphate and the adhesive.

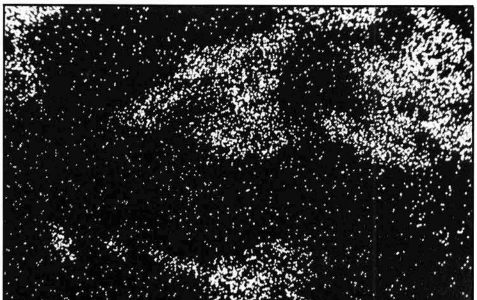
Figure 3—EDAX distribution of *Figure* 1 for Fe

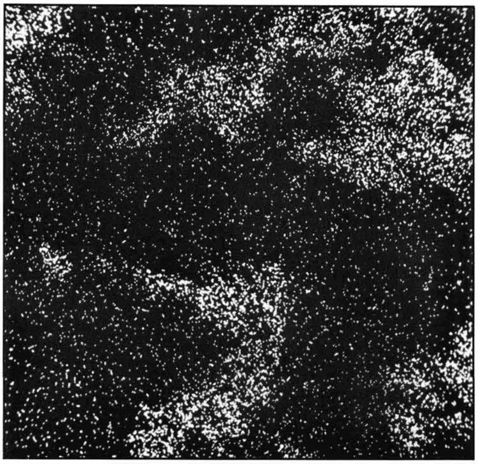
Figure 4—EDAX distribution of *Figure* 1 for P

Complementary analyses carried out with an electronic microprobe coupled to the SEM on the conjugate failure surfaces (substrate) confirmed the previously noted results concerning the locus of failure.

It can thus be said that three criteria exist for characterizing the intrinsic cohesion of the phosphate layers: (1) the maximum stress at failure, although this is unreliable; (2) the visual aspect of the fracture surface, a certain shininess being apparent when much phosphate is torn from the steel; and (3) the results of microanalysis.

Use of the above criteria is sufficient to locate the failure and to evaluate the resistance of various phosphate layers to shear.

It can be seen from *Table* 1 that the Z1 and Z2 systems differ by a single factor in their preparation; the quantity of accelerator in the bath. This in turn leads to a difference in phosphate thickness. The difference in mechanical behavior of the two systems probably is directly relat-

Table 4—Evolution of Apparent Interfacial Polymer/Phosphate Failure With Curing Conditions Of Electrodeposit

System	Test Rate (mm min^{-1})	τ_{max} (MPa)	Failure Surface (% interfacial) polymer/phosphate)
E1/Z1	0.01	39.0±0.5	15 – 30
E1/Z2	0.01	38.0±0.7	Ca. 50
E2/Z1	0.01	39.0±0.5	Ca. 10
E2/Z2	0.01	36.6±0.8	Ca. 50
E3/Z1	0.01	39.5±0.5	0 – 5
E3/Z2	0.01	36.2±0.5	10 – 30

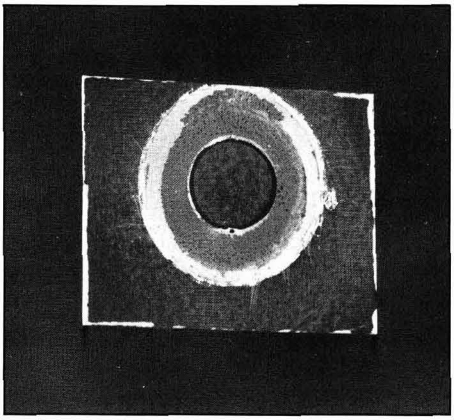

Figure 5—Substrate failure surface of the E1/Z1 system

ed to the kinetics of phosphate layer growth. Various types of analysis (x-ray, infra-red, x-ray fluorescence) conducted on Z2 have shown this phosphate layer to be quite homogeneous and essentially composed of phosphophyllite.[10] The feeble cohesion of this layer would therefore seem to be related to its rapid growth rate. It is suggested that the rapid precipitation of the phosphate layer, provoked by the accelerator in the bath, may lead to insufficient time being available for the deposited crystals to rearrange on the steel surface to produce a compact and stable structure. This unstable, rapidly produced, and less dense crystalline structure will almost inevitably be relatively weak mechanically. Conversely, the much more slowly produced and thinner phosphate layer of Z1 will very likely be more resistant mechanically. Certainly, the failure results would tend to bear out this hypothesis.

Cohesion of Polymer Coatings On Phosphated Steel

The intrinsic behavior of the phosphate layers is of interest, but the overall behavior of a given system could be markedly affected by the presence of a polymer coating. *Tables* 3 and 4 summarize results relative to the two types of phosphate coatings on steel (with polymer coatings cured under three sets of conditions). It should be recalled at this stage that a good criterion of shear resistance for such systems is the paucity of the zone (ii) type failure, i.e., the interfacial separation phosphate/polymer shown in the last column.[9]

Figures 5 and 6 show two examples of failure surfaces (substrate) corresponding respectively to E1/Z1 and E1/Z2 (see *Table* 3). The interfacial polymer/phosphate failure is the lighter-colored area. EDAX microanalyses effected in these regions, but on the conjugate (cylinder) surface showed a higher density of torn away microcrystals for E1/Z2 than for E1/Z1. However, in both cases, the densities are relatively feeble suggesting, essentially, interfacial failure. Outside this region, failure would seem to be cohesive within the polymer coating since in no case was any Zn, Fe, or P found to be present in the EDAX spectra corresponding to the dark regions of *Figures* 5 and 6.

Figure 7 presents the percentage area of the surface corresponding to interfacial polymer/phosphate failure as a function of severity of coating cure conditions. The results corresponding to the nomenclature 0, i.e., cure at 160°C for 10 min (undercure), are not included in *Table* 4 since the test rate for these systems was in fact 270 mm min^{-1} and not 0.01 mm min^{-1}. Nevertheless, the test rate generally had relatively little influence on the failure surfaces, and the results have therefore been included in *Figure* 7 to show the overall trend. An effect shown in this figure (but not, it must be added, from the values of τ_{max} in *Table* 4) is the clear decrease in polymer/phosphate interfacial failure with increasingly severe cure conditions (time, temperature). This is very probably related to the porous crystalline zinc phosphate layer, being to some extent sealed by the interpenetration of polymer into the interstices of the hollow structure. By increasing the temperature and/or the time, this sealing process presumably becomes more efficient and a more rigid final structure is obtained. The final surface layer may be compared to a type of composite material. Another possibility, of course, is that the heat treatment may modify the intrinsic phosphate mechanical properties to some extent. This tendency was observed with both Z1 and Z2 type phosphatations.

However, in common with results obtained for the systems without polymer coatings, Z1 would seem somewhat more resistant to shear than Z2. This could be related to the somewhat greater overall thickness of the latter system, but is more probably related to its less organized and less stable crystal structure, both by leading to a weaker matrix and to some extent hindering the polymer penetration, which in turn, consolidates the structure. One important remark concerning *Figure* 7 is that it depicts how readily and how greatly the mechanical properties of the phosphate/polymer coatings can be influenced by relatively small changes in curing conditions. This, it is suggested, may be one of the reasons for the

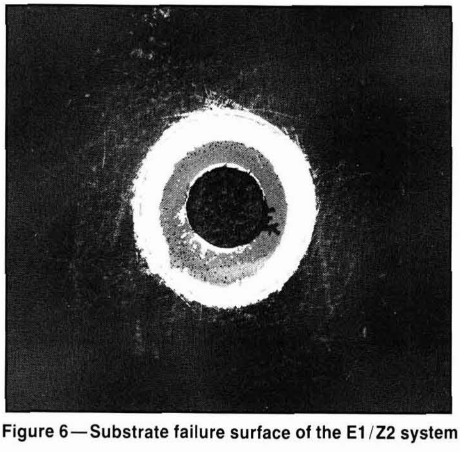

Figure 6—Substrate failure surface of the E1/Z2 system

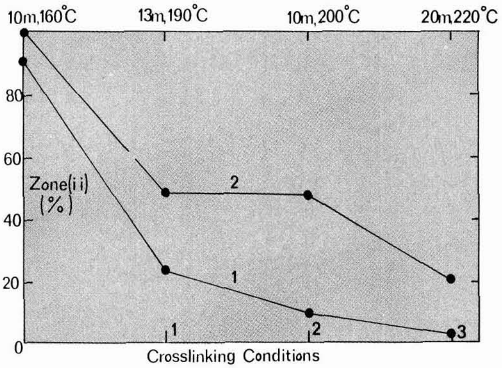

Figure 7—Percentage interfacial polymer/phosphate failure vs curing condition severity for systems 1 and 2

apparent occasional lack of resistance to shear observed with zinc-phosphated systems at an industrial level. Slight changes in production line conditions may vastly influence the quality of the final coating.

CONCLUSION

The results obtained during the second part of this study confirm the validity and simplicity of the torsional test previously developed to assess steel/phosphate/polymer systems. By jointly employing macroscopic criteria, such as the maximum stress at failure and the extent of interfacial polymer/phosphate failure surface, together with microanalysis of surfaces, it has been possible to show, on one hand, the influence of growth kinetics during phosphatation on the intrinsic cohesion and adhesion of the deposited layer, and, on the other hand, the influence of time and temperature conditions during polymer curing on the final mechanical behavior of the system.

ACKNOWLEDGMENTS

The authors are indebted to the Régie Renault for both their financial support and permission to publish this work.

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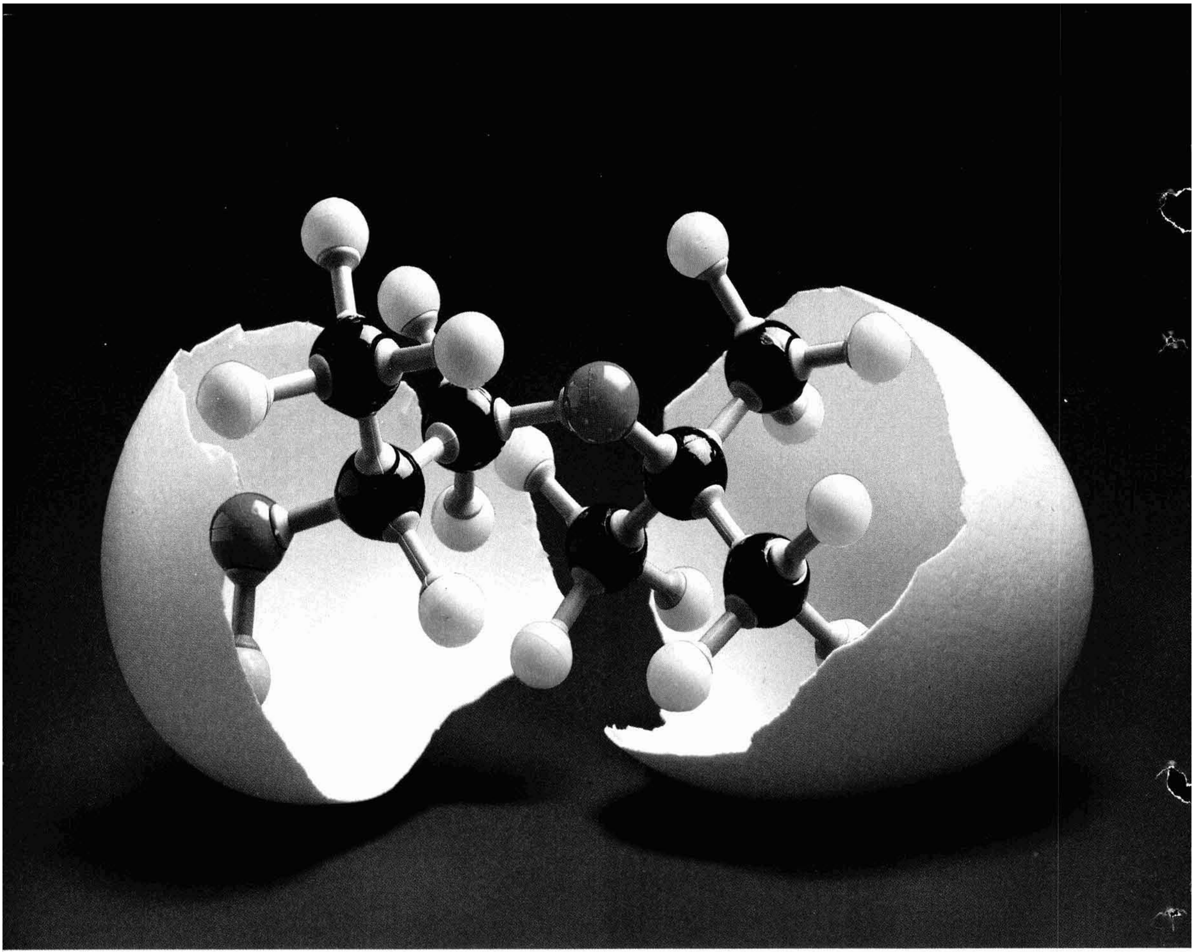

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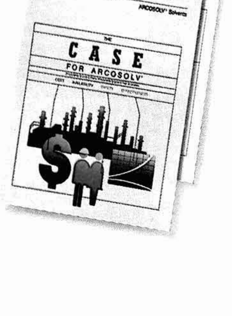

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Assessment of Phosphate/Polymer Protective Coatings on Steel

Part III: Influence of Phosphate Type and Morphology

M.E.R. Shanahan, H. Haidara, and J. Schultz
Centre de Recherches sur la Physico-Chimie des Surfaces Solides*

The final section of this study of the mechanical properties of phosphate and polymer coatings applied to steel is devoted to the nature and morphology of the phosphate layer. Maximum shear strength, failure surface aspects, and surface microanalysis have been used as the essential criteria for judging systems taken to failure using the previously developed torsional test. An iron-phosphate and a zinc-phosphate-based system have been compared, both in the simple state, and with polymer coatings that have undergone various stages of cure. Whereas the polymer-coated, iron-based system is more resistant to failure than its zinc-based counterpart, the roles are inversed when a polymer is not present. The vast improvement in the performance of the iron-based system would seem to be related to the presence of the polymer itself, helping to produce a compact, composite surface coating, and also to the heating cycle involved. This not only solidifies the polymer, but may facilitate a reconstruction of the phosphate layer leading to better steel/phosphate adhesion.

INTRODUCTION

This paper constitutes the third and final part of a study devoted to the shear mechanical resistance of phosphate and polymer protective coatings on steel. The first part of the work involved the establishment of a reproducible mechanical test; torsional shear was adopted with the test criteria of maximum shear stress at failure and the aspect of the failure surfaces.[1] In the second part, the influence of the zinc-phosphate layer thickness and polymer curing conditions on overall performance was broached.[2] This final section is concerned with the comparison of the performance obtained when phosphate coatings of a different nature (zinc or iron) and morphology are employed.

EXPERIMENTAL

Details of sample preparation (cylinder/plate assembly), experimental procedure, methods of analysis, and the assessment of failure surfaces have been previously described.[1,2] In this study, steel sheet treated with both zinc and iron phosphating, with and without subsequent polymer coatings, have been considered. The production and the characteristics of the two types of phosphate layers are summarized in *Table* 1. In our nomenclature, Z refers to zinc phosphating and F to iron phosphating.

A polymer coating was applied to the phosphate treatments. It had the same electrodeposit that was used in the previous work, applied in the same conditions, and had an average thickness of 20 μm.[1,2] Using our previous nomenclature, the presence of a polymer coating is designated by the letter E, and the figures 0, 1, 2, and 3 refer, respectively, to the curing conditions of 160°C for 10 min, 190°C for 13 min, 200°C for 10 min, and 220°C for 20 min.

RESULTS AND DISCUSSION

Torsional testing results obtained on the various systems studied are summarized in *Tables* 2 and 3 and are in the same format as the two earlier papers.[1,2]

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Figure 1—Optical micrograph of failure surface (substrate) of iron-phosphated steel

Resistance of Phosphate Layers Without Polymer Coating

The results of *Table* 2 suggest, from the average values of the maximum failure stress, τ_{max}, that the zinc-phosphated system is mechanically stronger in shear than the iron-phosphated system. However, this effect is not very marked so the mechanical tests were backed-up by failure surface analysis. *Figure* 1 is an optical micrograph of the substrate failure surface of the iron-phosphated system, F. This micrograph is typical, and over the entire failure surface cylinder/plate, these clear, lighter-colored zones can be seen where the phosphate coating had been pulled away, leaving bare steel surface. However, similar appraisal of the zinc-phosphated system, Z, has shown that failure and tearing-off of the phosphate layers is restricted to the localized annulus close to the exterior radius of the aluminum cylinder, where the stresses are highest at failure initiation. Scanning electron microscope (SEM) and EDAX analysis led to the conclusion that the essential failure mode for the Z system was interfacial between the phosphate and the adhesive used to attach the cylinder to the plate. Nevertheless, some traces of cohesive failure within the phosphate layer were evident near the exterior radius of the failure area. Similar analysis of the F system clearly showed that failure was essentially interfacial between the steel and the phosphate layer. Large amounts of phosphate layer remained encrusted in the adhesive attached to the cylinder.

Table 1—Production Conditions and Characteristics Of the Zn and Fe Phosphate Treatments

	Z	F
Pre-phosphatation preparation of steel substrate	Alkaline degreasing: aspersion-immersion; aspersion cycle: 5 min at 60°C Water rinse Affiner rinse by aspersion	
Phosphatation:		
Type	Zinc	Iron
Method	Immersion	Aspersion
Conditions	3 Min at 55°C	3 Min at 55°C
Accelerator	ClO_3^-	—
Post-phosphatation	Water rinse Chromic rinse by immersion Heating at 100°C for 2 min	
Type of Phosphate: (x-ray, infra-red, x-ray fluorescence)[3]	Phosphophyllite $Zn_2Fe(PO_4)_2 \cdot 4H_2O$ (crystalline)	Mixture of iron phosphates and oxides (quasi-amorphous)
Weight of deposited layer and estimated thickness[3]	2.3 gm/m^2 1 μm	<1.8 gm/m^2 <0.8 μm

Table 2—Torsional Test Results for Zinc And Iron-Phosphated Steel

System	Test Rate (mm min^{-1})	τ_{max} (MPa)	Failure Surface
Z	270	46.1 ± 2.6	See text
F	270	40.5 ± 2.8	See text

The three criteria employed (maximum failure stress, optical microscopy, and SEM/EDAX analysis) all show that under the production conditions summarized in *Table* 1, the zinc-phosphated system undeniably gives better mechanical resistance in shear than does the iron-phosphated system. *Figure* 1 and the SEM/EDAX results suggest that the poor resistance of the F system is mainly due to feeble adhesion of the phosphate layer to the steel substrate rather than an intrinsic cohesive weakness of the phosphate layer itself.

Resistance of Phosphate Layers With Polymer Coating

The torsional test results for the polymer-coated Z and F systems, having undergone variously severe curing conditions, as specified above, are summarized in *Table* 3. *Figures* 2 and 3 represent failure surfaces of the polymer coated Z and F systems, respectively. In both cases, *Figures* 2a and 3a represent curing conditions of 190°C for 13 min (nomenclature 1) and *Figures* 2b and 3b correspond to conditions of 220°C for 20 min (nomenclature 3).

Table 3—Torsional Test Results for Polymer-Coated, Zinc and Iron-Phosphated Steel

System	Test Rate (mm min^{-1})	τ_{max} (MPa)	Failure Surface (% interfacial) polymer/phosphate)
E0/Z	270	52.3 ± 1.6	80-90
E1/Z	270	54.5 ± 0.5	15-30
E1/F	270	51.1 ± 0.7	≲15
E2/Z	270	55.7	5-10
E2/F	270	54.3	5-10
E3/Z	270	55.6 ± 1.1	Ca. 5
E3/F	270	54.5 ± 0.6	Ca. 5

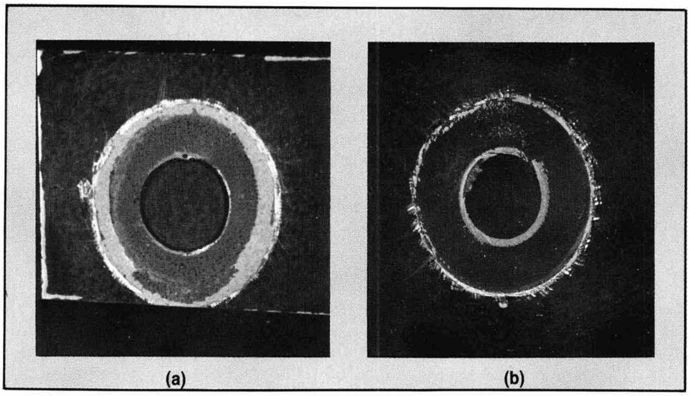

Figure 2—Failure surface (substrate) of polymer-coated, zinc-phosphated system: (a) curing cycle 190°C for 13 min; (b) curing cycle 220°C for 20 min

Perhaps the most surprising observation stemming from the results of *Table* 3 and the corresponding figures is the evolution in behavior of the iron-phosphated system after electrodeposition of the polymer coating. While we showed previously that the F system was weaker than the Z system when no polymer coating was in place, it can be seen that after electrodeposition and polymer cure the roles would seem to have been reversed. The difference may be relatively small. Nevertheless, it can be seen that the polymer-coated, iron-phosphated system tends to present a higher tenacity when subjected to shear stress. It is of equal interest to consider the maximum failure stresses of *Tables* 2 and 3. While there is little difference in failure stress between the various polymer-coated systems, the overall value is significantly higher than that observed for the systems without polymer, for the same test rate. There is clearly some consolidation of the system associated with the presence of polymer coating.

EDAX microanalysis effected on the conjugate (cylinder) failure surfaces, corresponding to the lighter regions of *Figures* 2a and 3a (apparent interfacial polymer/phosphate failure) led us to two conclusions concerning the locus of failure. As far as the iron-phosphated system is concerned, failure is essentially interfacial between the polymer and the phosphate layer. However, with respect to the zinc-based system, failure is mixed. The failure is essentially near the interface in question, but a large number of phosphate microcrystals are torn out and remain encrusted in the polymer.

To try and elucidate the evolution of the iron-based system, due to polymer deposition, the following experiment was conducted. Samples of the F system, already prepared, were heated to 200°C for 10 min but *without* any polymer coating. This was to emulate the curing cycle without the influence of the polymer. Such samples were tested in torsion as described previously. The results were perhaps a little surprising in that this heat treatment alone considerably improved the cohesive/adhesive properties of the F system. Regions of steel/phosphate adhesive separation practically disappeared.

In addition, the average failure stress of ca. 40 MPa became a respectable value of ca. 48 MPa. Presumably a structural or morphological rearrangement of the phosphate layer takes place during the heating cycle. This rearrangement may explain the improved steel/phosphate adhesion, but will be insufficient to explain the capacity of this layer to establish good adhesion with the subsequent polymer coating.

Figure 4 demonstrates the percentage polymer/phosphate failure as a function of curing conditions of the polymer-coated F and Z systems. Unfortunately, no undercured system based on iron-phosphate was available

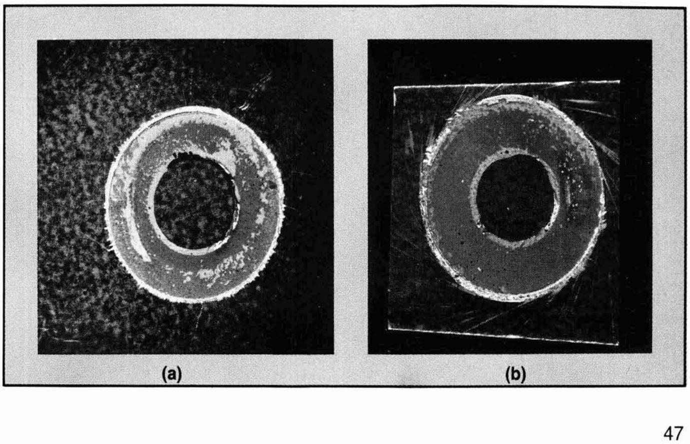

Figure 3—Failure surface (substrate) of polymer-coated, iron-phosphated system: (a) curing cycle 190°C for 13 min; (b) curing cycle 220°C for 20 min

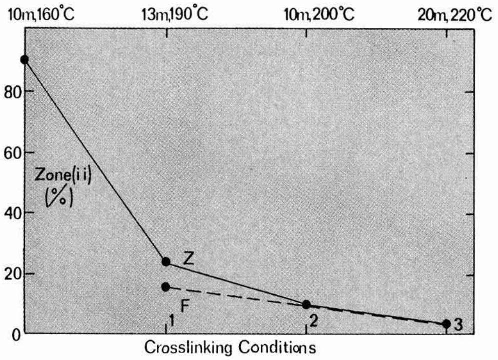

Figure 4—Percentage interfacial polymer/phosphate failure vs curing condition severity for systems Z and F

for testing. Nevertheless, the trend can be seen. The zinc-based system is largely influenced by the cure conditions, as indeed remarked in the earlier paper,[2] whereas the polymer-coated, iron-phosphate system would seem to show less dependence.

We suggest that the good iron-phosphate/polymer interfacial resistance and the relatively slight variability of failure mode, as exemplified by Figure 4, are related principally to two factors. The iron phosphate layer is compact and quasi-amorphous in nature. This will presumably augment the number of active sites on the surface capable of interacting with the polymer.[4] This same quasi-amorphous, yet to some extent porous structure, will tend to present a fine structure to the liquid polymer. As such, capillary effects will be increased and the polymer will be able to flow into the phosphate structure. On solidification, a phosphate/polymer composite phase will result. Both of these effects, site-density and capillary flow, will clearly be less significant for the zinc-based system which tends to have relatively large and widely spaced crystals. Due to this structure, the number of functional groups of the polymer (hydroxyles, carboxyles, etc...) capable, in principle, of forming chemical bonds with the phosphate layer, will be reduced. To the previous information, we may also add the fact that iron-rich compounds tend to be stable at high pH values ($\simeq$ 12), conditions found during electrodeposition.[5]

The inversion of behavior found in this study, i.e., the relative shear strength of the Z and F systems, depending on the absence or presence of a polymer coating, merits comment. Clearly the study of purely phosphated systems without electrodeposit is of fundamental value as far as the understanding of the physical chemistry is concerned. Nevertheless, this work shows that it is the mechanical behavior of the final coated system which must be examined in order to understand the overall protective properties.

CONCLUSION

The final part of this study of the mechanical, adhesive, and cohesive properties of steel/phosphate/polymer systems in shear has been devoted to the role played by the morphology and nature of the phosphate layer. In particular, iron and zinc-based phosphatation have been considered. While the iron-based system would seem to present better overall mechanical properties when covered with a subsequent polymer coating, the pure phosphatation alone is weak. The vast improvement obtained would seem to be related to the presence of polymer and its related cure cycle. The heating cycle alone probably allows some restructuration of the quasi-amorphous phosphate layer. In addition, this quality of being quasi-amorphous probably induces good polymer adhesion for two reasons. First, a large number of chemically active sites will be available on the surface, and second, polymer flow into the interstices will be facilitated by capillary action thus producing a final composite phosphate/polymer phase of high compactness.

ACKNOWLEDGMENTS

The authors are indebted to the Régie Renault for both their financial support and permission to publish this work.

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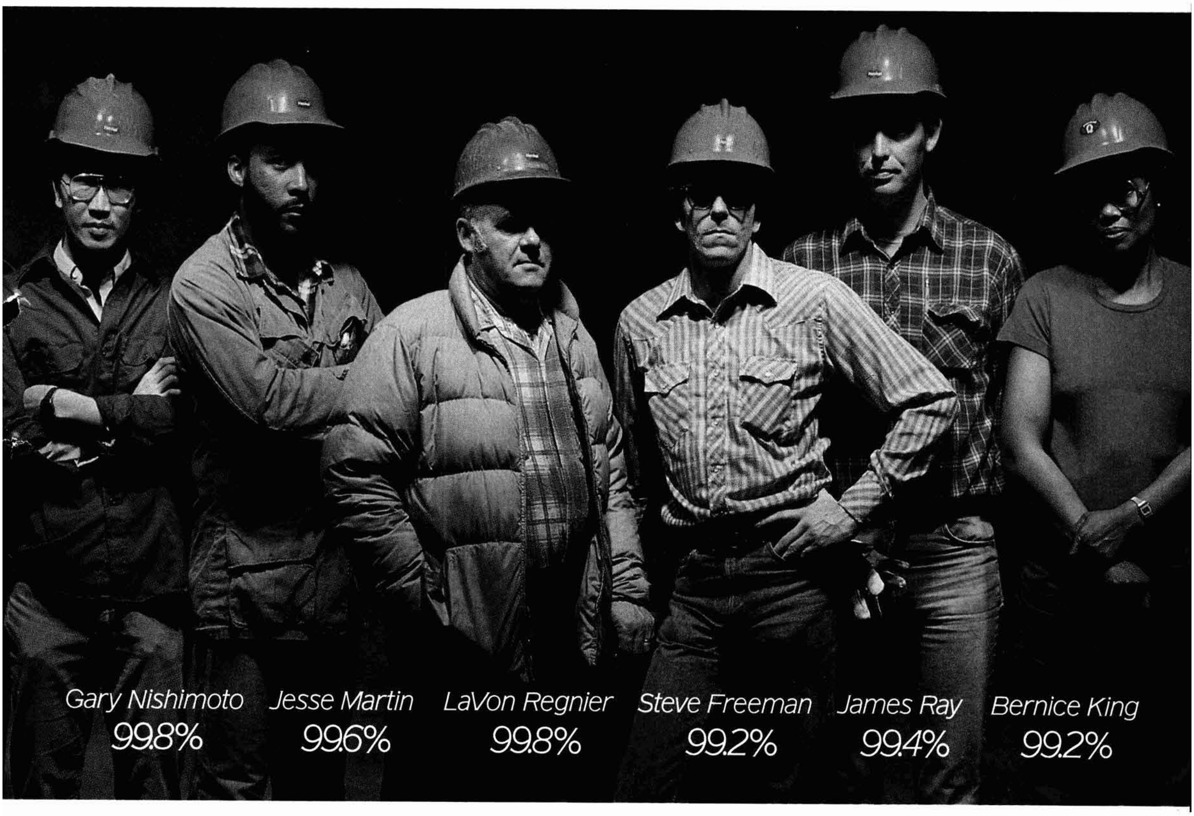

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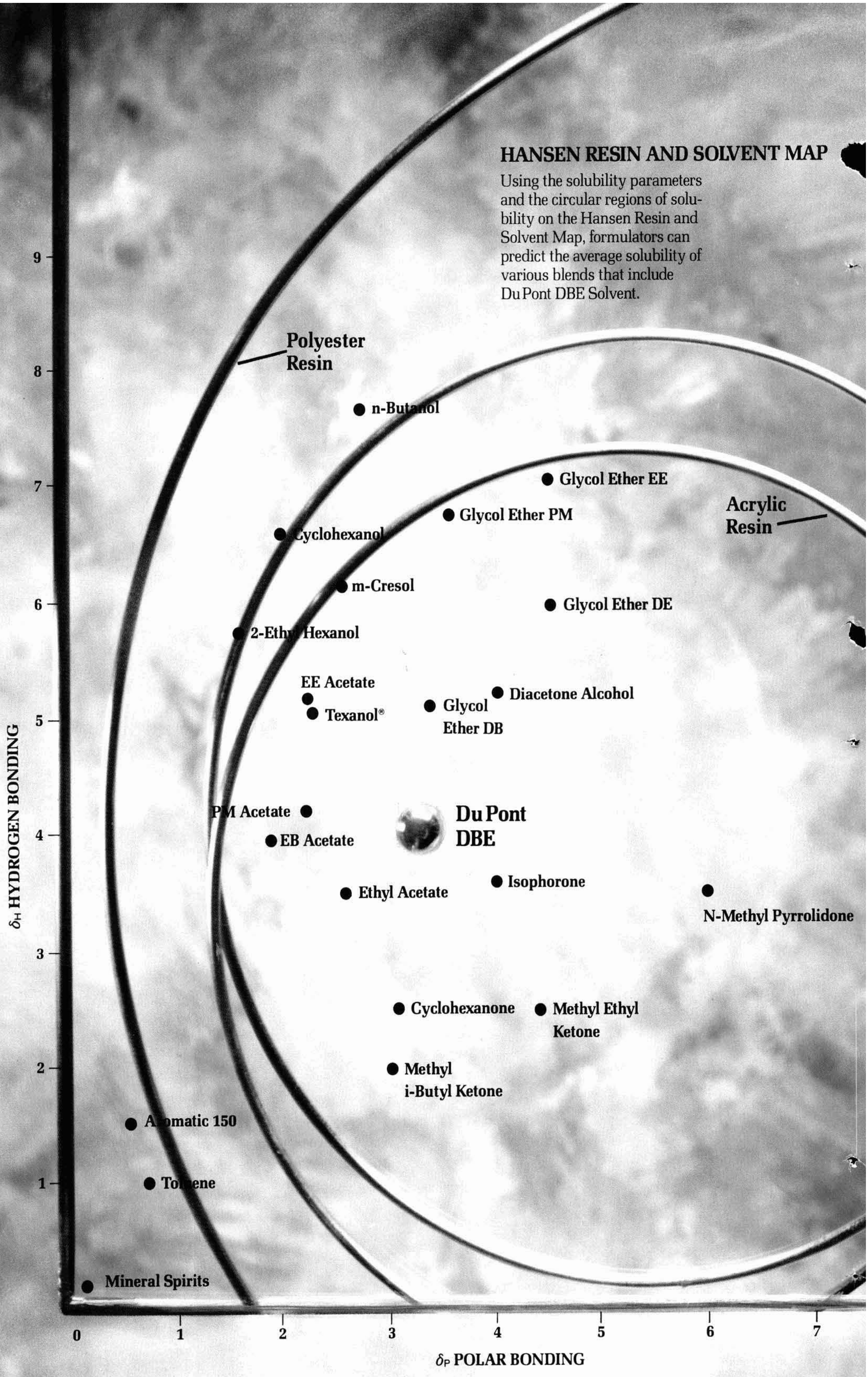

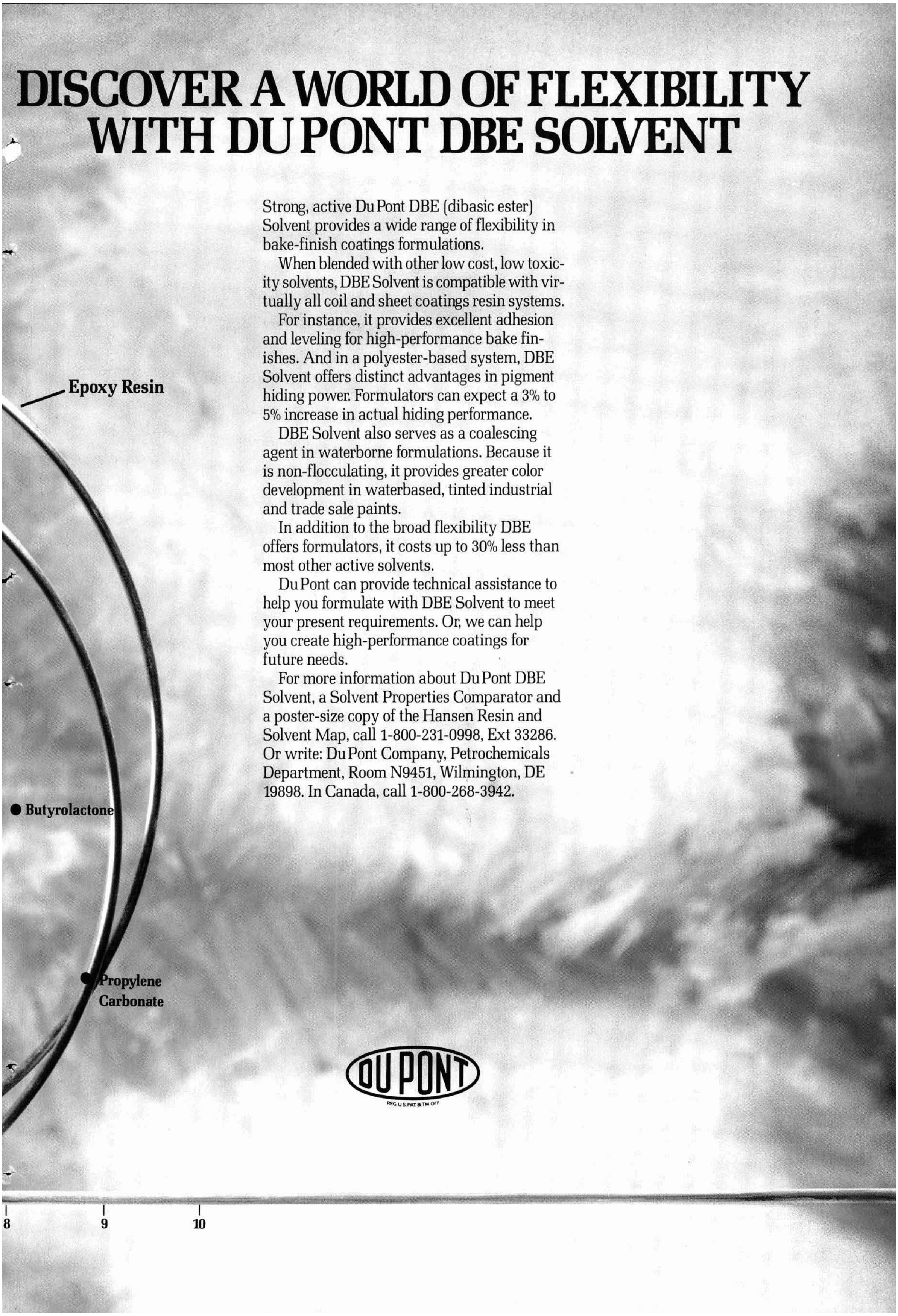
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Solvent Self-Diffusion In Polystyrene-Solvent Systems

Frank D. Blum
University of Missouri-Rolla*

Stephen Pickup
Drexel University†

Solvent self-diffusion coefficients have been measured for a variety of solvents in polystyrene solutions and gels using the pulsed-gradient, spin-echo NMR method. It is shown that the NMR method can be used to measure diffusion coefficients in a very wide range of polymer systems and the general trends found can be used to estimate diffusion coefficients in other polymer-solvent systems. Diffusion data as a function of composition may then be used to predict the diffusion controlled drying of coatings. In semi-dilute to concentrated (<80 wt% polymer) solutions it is found that normalized (D/D_o) solvent self-diffusion coefficients follow a "universal" curve as a function of concentration which is independent of the temperature and type of solvent. This allows the prediction of solvent self-diffusion coefficients over a wide range of concentration provided the self-diffusion coefficients for the pure solvents (D_o) at a given temperature are known. In high solids solutions, the solvent self-diffusion coefficients were found to follow Arrhenius behavior with energies of activation increasing with increasing concentration. In polystyrene beads, the self-diffusion coefficient measurements reflected the diffusion of solvent within the bead. It was also shown that the solvent diffusion in the crosslinked system was similar to that found in noncrosslinked systems, suggesting that the crosslinks do not provide an additional impediment to diffusion.

INTRODUCTION

The diffusion of solvents in polymer systems is of fundamental importance to the coatings industry for a variety of reasons. For example, the latter stages of the drying of coatings are usually controlled by the diffusion of the solvents to the surface.[1–4] The diffusion of solvents and other small molecules can play a significant role with respect to processing of polymer systems.[5] The coloration and decoloration of a surface coating may be intimately related to solvent diffusion.[6] Since the diffusion coefficients of solvents above the glass transition temperature (T_g) of the system are usually non-Fickian, in most cases a temperature-concentration study must be performed in order to obtain the diffusion profile as a function of environmental conditions. Concentration dependent measurements are not usually possible using sorption-desorption techniques.[3,4] In fact, independent measurements of the concentration dependence of solvent diffusion coefficients may be necessary to understand the "average" diffusion coefficients measured by traditional techniques.[4] Once the diffusion vs concentration profile is known, it is then possible to model the drying of films.[7] Presently, the major impediment to this type of modeling is the lack of good solvent diffusion coefficient data.

Transport of small molecules in crosslinked polymeric media is also of great interest in the polymer field. Polymer coatings are often crosslinked for increased stability. Crosslinked polymer beads are also used as supports for catalysts, ion exchange resins, solid-phase peptide synthesis, and packing for chromatographic columns. In many of these applications, the effectiveness of the system is determined by the transport of small molecules in the polymer matrix. NMR spin-lattice relaxation times have been used to measure the differences in mobility of the solvent inside and outside of the bead from which

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Presented by Robert J. Gambogi at the 65th Annual Meeting of the Federation of Societies for Coatings Technology, in Dallas, TX, on October 6, 1987.

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exchange rates can be approximated.[8] However, very little work has been done directly on the translational mobility inside the beads.

In the last few years, major developments in the measurement of diffusion coefficients have occurred. Perhaps the most significant developments have been in the areas of quasi-elastic light scattering and pulsed-gradient, spin-echo (PGSE)-NMR. These two techniques measure mutual and self-diffusion coefficients, respectively. Their use is a welcome supplement to the classical techniques, such as permeation, adsorption/desorption, or radio-isotope labeling.[4,9] The PGSE-NMR technique for measuring self-diffusion coefficients was first developed by Stejskal and Tanner for a home-built apparatus.[10] Subsequent refinement by three groups[11–14] has allowed this technique to be used on commercial spectrometers. In many cases, multi-component diffusion measurements are made in less than an hour.[12–14] In favorable cases, diffusion coefficients in heterogeneous systems can also be accurately determined with the PGSE technique.[14,15] The application of the technique in polymer systems[14,16] and colloids[14] has also been reviewed.

In this paper, we present the results of self-diffusion measurements of several solvents in different polystyrene systems. First, the dilute to concentrated solution region (0-80 wt%) at and above room temperature for a variety of solvents has been studied. We found that the normalized self-diffusion behavior is independent of the polymer-solvent pair and the molecular weight of the polymer.[17] Second, we have probed the self-diffusion of cyclohexane, toluene, and n-butyl acetate in polystyrene at higher temperatures (60-105°C) and high solids content (75-95 wt% solids). We found that the self-diffusion coefficients show Arrhenius behavior with temperature. Finally, for completeness, we also describe our previous work on the diffusion coefficients of toluene in polystyrene beads of different degrees of crosslinking.[18] These results are compared to similar measurements in solutions of linear polystyrene at similar concentrations (based on the solvent content of the swollen beads) in order to determine the effect of crosslinks on solvent transport. It is found that the crosslinks have little direct effect on the solvent diffusion aside from limiting the swellability and, therefore, the concentration of solvent in the bead.[18] The results of all three of these studies provide predictive ability which can be extended to other polymer-solvent systems.

EXPERIMENTAL

Materials

Linear polystyrenes (PS) from Dow Chemical Co. (M_v = 280,000 g/mol) and Polysciences (M_v = 18,000 g/mol) were used as received. The samples were prepared by adding weighed amounts of PS and solvent to 5 mm NMR tubes. The tubes were sealed to prevent solvent evaporation and placed in a heating block at 80-100°C until homogeneous. The volume fractions were calculated using the densities at 25°C and assuming ideal volumes of mixing. The solvents were all of high purity and used as received.

The crosslinked beads (supplied by W.T. Ford of Oklahoma State University) were synthesized with varying amounts of divinyl benzene by suspension polymeriza-

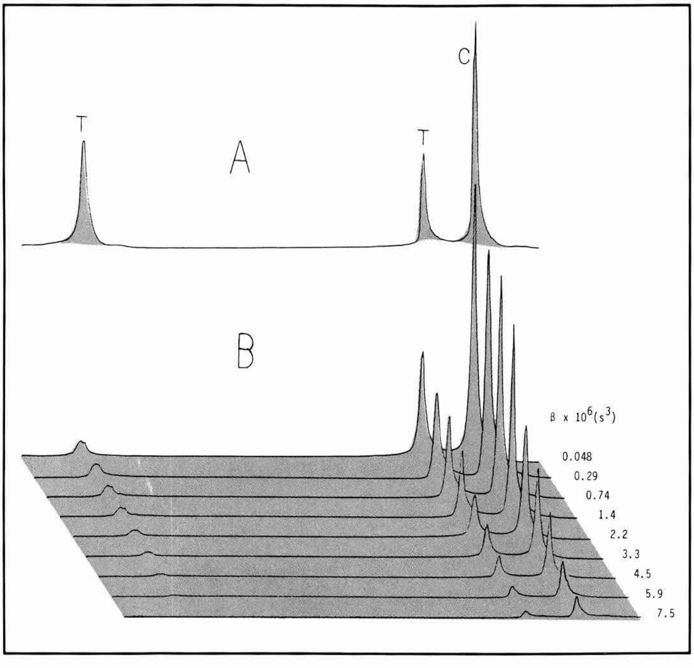

Figure 1—(A) Normal and (B) PGSE (as a function of β) NMR spectra of a toluene/cyclohexane/polystyrene solution. The toluene and cyclohexane resonances are denoted T and C, respectively

tion.[19] The degree of crosslinking was assumed to be the percent of divinyl benzene in the feed. The samples with the beads were prepared by adding excess toluene to the beads in 10 mm NMR tubes. A vortex plug was used to push out the excess interstitial solvent. A small amount of solvent remained in the tube above the plug in order to keep the beads saturated. Solvent concentrations in the beads were calculated based on the increased volume of the beads after saturation with toluene.

PGSE Technique

All self-diffusion measurements were made using the PGSE-NMR technique on a JEOL-FX90Q NMR spectrometer operating at 90 MHz for protons. The JEOL temperature controller was used to control the temperature to within ±2°C. Typical spectra were taken with a 2000 Hz window, 4096 points, and a pulse delay of 8 sec. The number of scans ranged from 8-300 depending on the concentration and gradient pulse length.

The PGSE-NMR method measures self-diffusion coefficients by tagging the nuclear spins with precessional frequencies which are dependent on the position of the nuclei within the sample tube. This is done with the use of a pulsed linear magnetic-field gradient. Since the separation of NMR energy levels is much less than thermal energy, the NMR experiment does not perturb the thermal motion (i.e., Brownian diffusion) of the molecules. The method and its use on a commercial spectrometer have been reviewed.[14] The basic NMR pulse sequence consists of a 90°-180° spin echo sequence with two field gradient pulses added. With this technique, the attenuation of the NMR spin-echo signal intensity is given by:

$$A = A_o \exp [-\gamma^2 G^2 D \beta] \qquad (1)$$

where A is the signal intensity, A_o is maximum signal intensity when $\beta = 0$, γ is the magnetogyric ratio of the proton, G is the field-gradient strength (in this case, 0.05 T/m), D is the self-diffusion coefficient, and $\beta = \delta^2 (\Delta - \delta/3)$ where δ is the length of the field-gradient pulse, and Δ is the fixed time between the preparation (90°) and refocusing (180°) pulses.[14] The field gradient, G, can be calculated using cyclohexane as a sample of known self-diffusion coefficient. Plots of ln A vs β yield a straight line for molecules in isotropic solutions, with a slope of $-\gamma^2 G^2 D$, but marked curvature may be found in heterogeneous systems.

Non-exponential behavior of the solvent decay curves is found in the presence of the swollen-polystyrene beads. The time scale over which the PGSE experiment operates, on our instrument, is such that the solvent in the beads diffuses ca. 12 μm during the measurement. Since the beads in this study have diameters of more than 200 μm, much of the solvent in the beads remains there over the experimental timescale. Therefore, it is possible to determine the diffusion coefficient of the solvent in the bead with little or no contribution from exchange processes. The NMR experiment can therefore be a probe of diffusion on a microscopic scale.

In the heterogeneous systems, such as the polymer beads, the signal intensity of the PGSE experiment is attenuated by an apparent diffusion coefficient under the limiting conditions of $p_a >> p_b$ and $D_a << D_b$, where the subscripts a and b refer to the solvent inside and outside of the bead, respectively, p is the weight fraction, and D is the self-diffusion coefficient. The polymer beads meet these requirements and the apparent diffusion coefficient of the solvent is given by:[15]

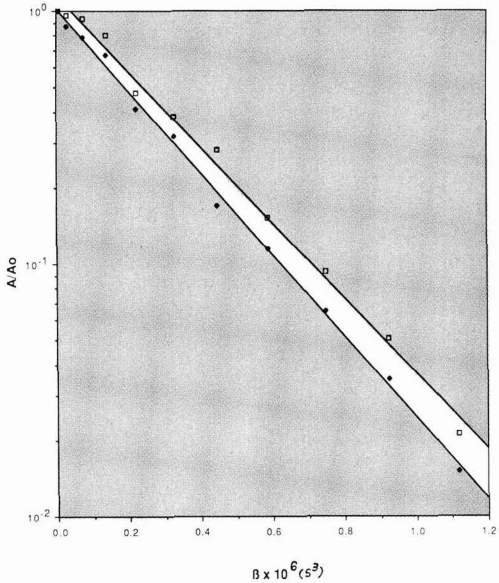

Figure 2—The PGSE plots for the toluene (□) and cyclohexane (◆) resonances as a function of β. The slopes of the lines are proportional to the self-diffusion coefficients

$$D_{app} = D_a + p_b D_b/(1 + \gamma^2 \delta^2 G^2 \tau_a p_b D_b) \qquad (2)$$

where τ_a is the mean lifetime of the free solvent. This relation suggests that in the limit of large δ, $D_{app} = D_a$. Thus, a semilog plot of intensity vs β should asymptotically approach a line of slope $-\gamma^2 G^2 D_a$ in the limit of large β.[20] In the next section, examples of solvent diffusion for both isotropic and restricted diffusion are shown.

RESULTS AND DISCUSSION

As an example of a multicomponent diffusion coefficient measurement, the normal and PGSE spectra of a toluene/cyclohexane/polystyrene solution (33/44/23 wt% of each) at ambient temperature are shown in *Figure* 1. The polystyrene resonances are broad enough that the spectra are dominated by the narrow solvent peaks. The total measurement time was less than a half-hour. The effects of diffusion are clearly shown in the decay of the intensities of the resonances with increasing β values. In this case, it is quite easy to measure both diffusion coefficients from a single set of spectra. In fact, as many as 12 diffusion coefficients have previously been determined from a single run.[13] Plotted in *Figure* 2 are the ln (intensity) vs β data for both solvents. The data in *Figure* 2 show the exponential dependence of the intensity on β [equation (1)] and yield diffusion coefficients of 2.13×10^{-9} and 1.95×10^{-9} m^2/s for toluene and cyclohexane in polystyrene solution, respectively.

In *Figure* 3, the normalized self-diffusion coefficients of the solvents in binary polystyrene solutions are plotted as a function of the volume fraction of polymer. The normalized self-diffusion coefficient is defined as $D(1-p)/D_o$, where D_o is the self-diffusion coefficient in the neat solvent and p is the volume fraction of polymer. The $(1-p)$ term corrects for the proper reference volume required by Fick's laws.[21] As can be seen from the figure, the normalized self-diffusion coefficients for the solvents in the polystyrene systems all fall along a similar curve as a function of polymer volume (or weight) fraction. This is consistent with results for other polymer-solvent systems.[17,21] In general, it appears that the normalized self-diffusion coefficient of the solvent is not significantly affected by solvent type, temperature, or polymer molecular weight[17] (not shown). The thermodynamic solvent qualities range from thermodynamically good (toluene) to poor (cyclohexane) solvents for polystyrene.

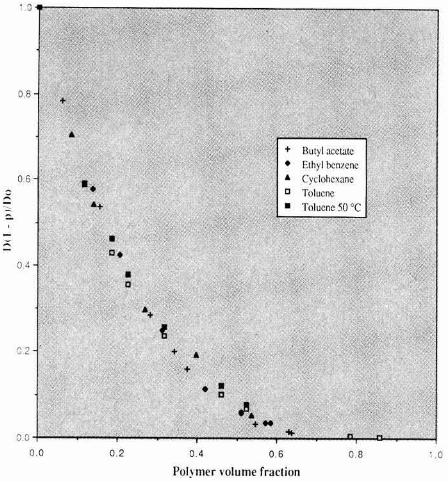

Figure 3—Normalized solvent self-diffusion coefficients for several solvents as a function of the volume fraction of polystyrene (18,000 g/mol) for the lower concentration region. The measurements were made at 25°C except for one toluene containing sample which was at 50°C

The universal nature of the solvent self-diffusion curves has previously suggested that specific chemical interactions do not play a determining role in solvent diffusion. For example, in aqueous systems, water capable of hydrogen bonding shows the same behavior, when normalized, as toluene or cyclohexane in polystyrene solutions.[17,21] It has previously been postulated that solvent self-diffusion in this concentration range is dominated by a mechanism similar to a surface drag effect.[22] The self-diffusion results are then consistent with a view that a solvent molecule next to a macromolecular segment, which moves very slowly compared to neat solvent, has a reduced self-diffusion coefficient. The number of motionally slowed molecules is roughly determined by the volume fraction of solvent. Since the solvent next to the polymer rapidly exchanges with the bulk-like solvent, relative to the timescale of this NMR experiment, similar reductions are found for all solvent molecules regardless of the system. The strength of the interaction may determine how long a solvent molecule interacts with the polymer, but even in strongly hydrogen bonded systems, this time is short compared to the timescale of the experiment. Regardless of the reasons for this type of universal behavior, its predictive ability is remarkable because all that is required for prediction of the diffusion coefficient of the solvent is knowledge of the diffusion coefficient of the pure solvent at a given temperature and the volume fraction of polymer.

Since the sensitivity of this type of analysis must decrease as the polymer concentration is increased, we have chosen to examine the high concentration region in a different way. In addition, the solvent diffusion in highly concentrated systems becomes very slow and, in some cases, impossible to measure at room temperature with our apparatus. We have therefore chosen to measure the diffusion coefficients of toluene, cyclohexane, and n-butyl acetate at higher temperatures. At the temperature chosen, the systems are above their T_g. In some cases, these temperatures are similar to those which might be experienced during a baking process.

Shown in *Figures* 4-6 are the solvent self-diffusion results (log scales in units of m^2/s) for the three systems as a function of both concentration and temperature. As expected, the self-diffusion coefficient of the solvent decreases with increasing polymer concentration. A comparison of the solvent self-diffusion data reveals that there is very similar behavior in all three systems as a function of both temperature and concentration. The results are even quantitatively similar, which is perhaps due to the similarity in molecular size of all three solvents. The temperature dependence of the self-diffusion coefficients can be approximated as Arrhenius-like, with the energies of activation given in *Table* 1. For concentrations less than 90 wt%, the energies of activation are not greatly concentration dependent, although there seems to

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STEPHEN PICKUP is a visiting Assistant Professor of Chemistry at the University of Missouri-Columbia (UMC). Dr. Pickup earned his Ph.D. Degree in Polymer Chemistry from Drexel University. His thesis topic was NMR measurements of diffusion coefficients in surfactant and polymer systems. Dr. Pickup was chosen as a finalist in the Sherwin-Williams Award Symposium in 1985 for his work on solvent diffusion in swollen-polystyrene beads.

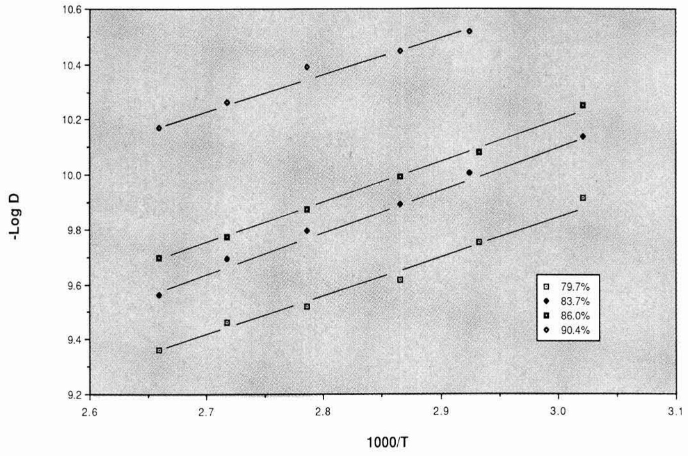

Figure 4—Temperature dependence of the self-diffusion coefficients of n-butyl acetate in polystyrene as a function of temperature. Concentration is listed in wt% polystyrene

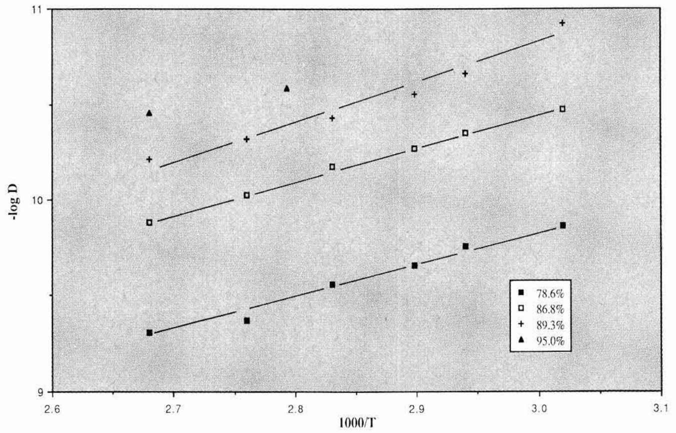

Figure 5—Temperature dependence of the self-diffusion coefficients of cyclohexane in polystyrene as a function of temperature. Concentration is listed in wt% polystyrene

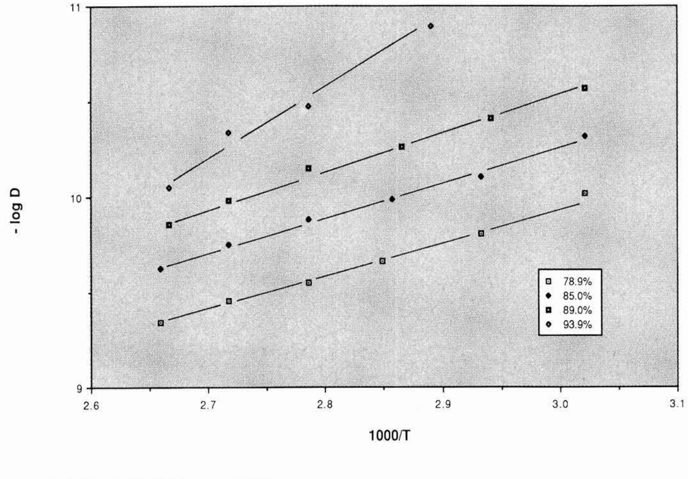

Figure 6—Temperature dependence of the self-diffusion coefficients of toluene in polystyrene as a function of temperature. Concentration is listed in wt% polystyrene

Table 1—Calculated Energies of Activation For the PS-Solvent Systems

Wt% PS	ΔE_a(kcal/mol)^a n-Butyl Acetate	Cyclohexane	Toluene
0	—	3.7^b	2.5^b
79-80	6.8 (0.4)	8.0 (0.5)	8.3 (0.4)
84-85	7.1 (0.3)	—	8.4 (0.4)
86-87	6.9 (0.2)	8.0 (0.2)	—
89-90	5.7 (0.6)	9.3 (0.8)	9.0 (0.3)
94	—	—	16.5 (1.4)

(a) Uncertainties given as ± 1 s. d. in parentheses.
(b) From references 23 and 24.

be a slight increase with increasing concentration. It is also noted that the energies of activation are higher than those for the pure solvents by a considerable amount.[23] This is consistent with lower concentration data for cyclohexane-PS data.[24] At higher concentrations, there is considerable uncertainty in the E_a values and, in some cases, not enough data to calculate them accurately because the solvent diffusion is too slow. It should be noted that it is possible to obtain diffusion coefficients for solvents as high as 95 wt% polymer.

Finally, we have attempted to probe the effect of crosslinking on the solvent diffusion coefficients. Shown in *Figure* 7 is a semi-log plot of signal intensity vs β for toluene in 20% crosslinked polystyrene beads.[8] The initial, rapidly decaying portion of the curve is attributed to free and exchanging solvent. The linear portion in the limit of large β represents the diffusion of solvent within the beads as predicted by equation (2). The slope of this line is used to predict the diffusion coefficient of toluene inside the beads.

In *Figure* 8, the diffusion coefficients of solvents within the beads (normalized for convenience) vs concentration (as determined from the swell ratios of the saturated beads) are plotted for several different crosslink densities. Also shown in *Figure* 8 are the diffusion coefficients of toluene in solutions of linear polystyrene at several different concentrations. The data essentially fall on the same curve. In fact, it has the same "universal" behavior shown in *Figure* 3. This suggests that the crosslinks themselves have very little effect on transport of solvent in the beads. The determining factor for the diffusion of solvent is the concentration of solvent within the bead and not the crosslinks. At present, we cannot say with certainty if there is any upper concentration limit above which the crosslinks effect solvent transport.

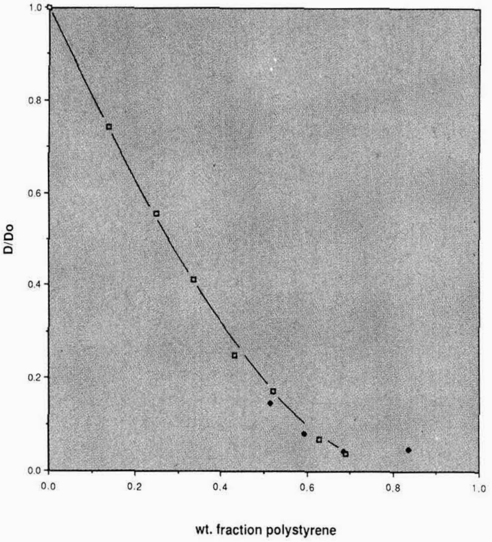

Figure 8—The ratio of diffusion of toluene to that of pure toluene as a function of concentration for polystyrene solutions (□) and in swollen crosslinked beads (♦) with crosslink densities of 5.7, 10, 20, and 40% having polymer concentrations of 51, 59, 68, and 83%, respectively, as determined from the swell ratios

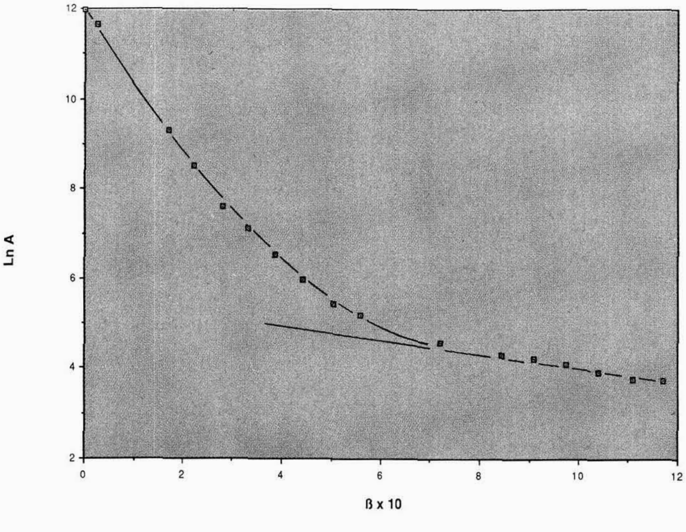

Figure 7—Signal intensity as a function of β for toluene in 20% crosslinked polystyrene beads. The straight line represents the best fit in the region of large β which was used to determine the diffusion coefficient of the solvent inside the bead

CONCLUSIONS

Using the PGSE-NMR method, it is possible to determine the self-diffusion coefficients of solvents over a very broad range of temperatures and concentrations, and in heterogeneous systems. In the 0-80 wt% polymer range, the solvent self-diffusion coefficients show "universal" behavior. This can be used as a tool to accurately predict the solvent diffusion coefficients with only the knowledge of the self-diffusion coefficient of the pure solvent and the volume fraction of the pure solvent. At higher solids content, the diffusion coefficients decrease with concentration as expected. These can be determined in solutions as high as 95 wt% polymer. In this range, the diffusion behavior is Arrhenius-like. The energies of activation are lower for n-butyl acetate than cyclohexane or toluene and have values of 7-9 kcal/mol. The effects of chemical crosslinks on solvent transport in polystyrene beads are not significant for the systems studied. In the swollen polystyrene beads, the solvent self-diffusion coefficients are similar to those in polystyrene solutions of the same solvent concentrations.

ACKNOWLEDGMENTS

The authors wish to thank the E.I. du Pont de Nemours & Co., Marshall R&D Laboratory for their financial support of this project. The authors also thank Drs. R.D. Redfearn and D.T. Wu of Marshall R&D Laboratory for their helpful discussions. Warren T. Ford and M. Periyasamy of Oklahoma State University were coworkers on solvent diffusion in beads.

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Effects of Different Polyol-terminated Urethane Prepolymers on the Properties Of Their Corresponding Crosslinked Films

Chin-Ping Yang and Lung-Ta Lee
Tatung Institute of Technology*

Polyglycols (PG) and toluene diisocyanate (TDI) were reacted at various molar ratios to produce isocyanate-terminated polyurethanes and were then reacted with glycerine, pentaerythritol (PE), or trimethylol propane (TMP) to produce hydroxyl-terminated urethane prepolymers of different molecular weights. These prepolymers were mixed with an equivalent amount of blocked isocyanate in a mixture of m-cresol and naphtha to give the varnishes. Pretreated copper wires were coated and baked to give polyurethane coated magnet wires. The mechanical properties of the polyurethane crosslinked films, the change of functional groups during the crosslinking reaction, and the properties of magnet wires coated with polyurethane varnishes are discussed.

INTRODUCTION

The advantages of polyurethane coatings were recognized by people after Otto Bayer and his co-worker discovered the addition reaction of diisocyanate groups.[1] During World War II, the manufacturing of chemicals for urethane coatings as well as for fiber elastomers developed into a strategic industry. In recent years, polyurethane resins, with their outstanding characteristics, have been widely used in many industrial fields, such as coatings, elastomers, foams, leathers, and fiber-treating agents. The main purpose of this research is to study the mechanical properties of thermally crosslinked polyurethane insulating coating for magnet wires.

There are two procedures for producing crosslinked polyurethane. The first begins by synthesizing prepolymers which contain isocyanate groups in the terminal and/or middle of polymer chains. The prepolymers are then reacted with a chain extender or crosslinking agent to give crosslinked polyurethane. The second method is to first synthesize prepolymers, which contain hydroxyl groups in the terminal and/or middle of polymer chains, followed by a reaction with a crosslinking agent, such as polyisocyanate or an isocyanate regenerator to give crosslinked polyurethane.

In the first procedure, polyol or polyamine, which can easily react with isocyanate groups, is used as the crosslinking agent. In the second procedure, polyisocyanate or blocked isocyanate, which can easily react with hydroxyl groups, is used as a crosslinking agent. Polyurethane varnishes are generally synthesized as described below:[2-7] isocyanates or blocked isocyanates with or without catalyst are reacted with various polyols to give hydroxyl-terminated prepolymers and then mixed with crosslinking agents to form coating varnishes which are used for the manufacturing of magnet wires.

In 1974, Takizawa and Ito[8] reported on a polyurethane insulating varnish. They synthesized a polyurethane varnish with trimethylol propane, toluene diisocyanate (TDI), and Desmodur® AP-stable to form brittle crosslinked films. The brittleness of the crosslinked films limited their practical application. Some modified polyurethane varnishes[9-15] for improvement in certain properties are also reported. Okada, et al.[16] reported on a polyester urethane insulating varnish. They synthesized a

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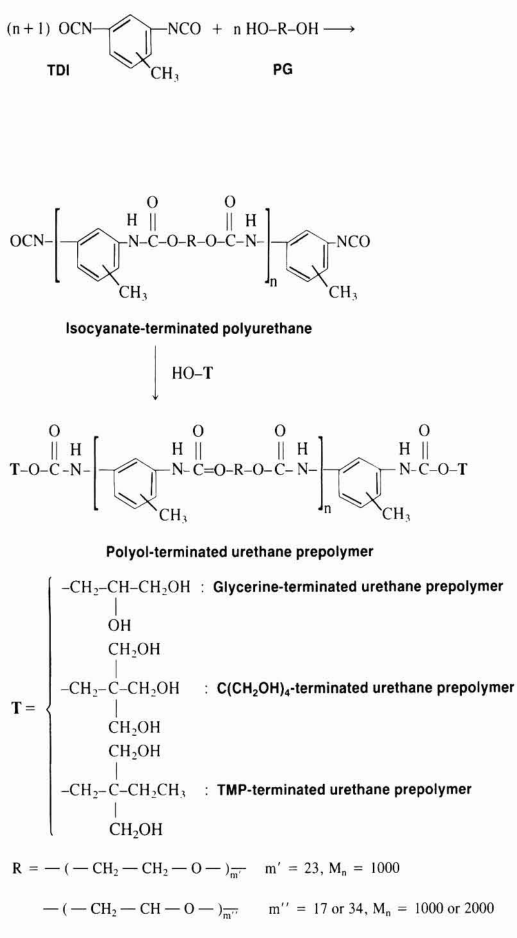
Desmodur is a registered tradename of Bayer AG/Mobay.

hydroxyl-terminated polyester with dimethyl terephthalate, ethylene glycol, and glycerol. Then, the polyester was reacted with TDI mixed with Coronate AP-stable for producing coating varnish, which was used to produce high-quality magnet wire.

Glycerine, pentaerythritol (PE), and trimethylol propane (TMP) are commonly used to synthesize polyester coatings, polyurethane foams,[17-19] and elastomers.[20-23] Few polyurethane coatings prepared from glycerine or PE have been reported. We successfully used polyglycols (PG)[9] and polyester-ether[10] to modify the brittleness of the crosslinked films of the TMP/TDI series. At that time, the urethane prepolymer synthesized was a mixed molecule type [i.e., a mixture composed of TMP-(TDI-TMP)$_n$ and TMP-(TDI-PG)$_n$-TDI-TMP]. In this study, PG were still chosen as modifiers. Changing molar ratios of PG/TDI and terminating with polyols such as TMP, glycerine, and PE can produce different chain lengths of a single molecule type of hydroxyl-terminated urethane prepolymer, [polyol-(TDI-PG)$_n$-TDI-polyol], which were then mixed with an equivalent amount of blocked isocyanate and baked to form crosslinked films. The practicability of these three series of crosslinked films were investigated.



Scheme 1

EXPERIMENTAL

Materials

Monomers used in synthesizing urethane prepolymers and blocked isocyanate were reagent-pure TMP (Hayashi Pure Chemicals, Ltd., Japan), PE (T.C.I., Japan), TDI (San Ten Chemical), and glycerine and phenol (WAKO Pure Chemicals, Ltd., Japan).

Polyglycols used in synthesizing urethane prepolymers were polypropylene glycol with $M_n = 1000$ [PPG(#1000)] (Hayashi Pure Chemicals, Ltd.), polypropylene glycol with $M_n = 2000$ [PPG(#2000)] (WAKO Chemicals, Ltd.), and polyethylene glycol with $M_n = 1000$ [PEG(#100)] (T.C.I.).

Copper wires used in processing were annealed and pretreated with a diameter of 0.6 mm and supplied by Tatung Cable Co.

Synthesis

Urethane Prepolymer: Given amounts of TDI were added to a 100 mL three-necked flask equipped with an electric stirrer, a reflux condenser, a dropping funnel, and an inert nitrogen blanket. A calculated quantity of PG was added dropwise into the flask. The mixture was kept at room temperature for 30 min, gradually heated to 50-60°C for 30 min, and then cooled to room temperature. A quantity of glycerine, TMP, or PE was then added before the temperature was raised to 40-50°C for 1 hr, to 80°C for 30 min, to 100°C for 30 min, to 120°C for 2 hr, and to 140°C for completion. The resultant product was a glycerine-, TMP-, or PE- terminated urethane prepolymer.

Crosslinking Agent (Blocked Isocyanate): A given quantity of TDI was added to a 100 mL three-necked flask equipped as previously described. The mixture of quantitative TMP and phenol in beaker was heated to 50°C for dissolution and then cooled to room temperature. The mixture was then added dropwise into the flask and kept at room temperature for 30 min, gradually heated to 40-50°C for 1.5 hr, to 80°C for 1 hr, to 110°C for 3.5 hr, and to 120°C for 6.5 hr for completion. When the viscosity of the reaction medium increased, 10 mL of anisol were added to the flask to reduce the viscosity of the reaction medium to enable smooth stirring. After the reaction was completed, 50 mL of m-cresol were added to the flask. The product, a blocked isocyanate, was a golden and viscous liquid at room temperature.

Film Casting

A 40% solid content solution was prepared by dissolving and mixing glycerine-, TMP-, or PE-terminated

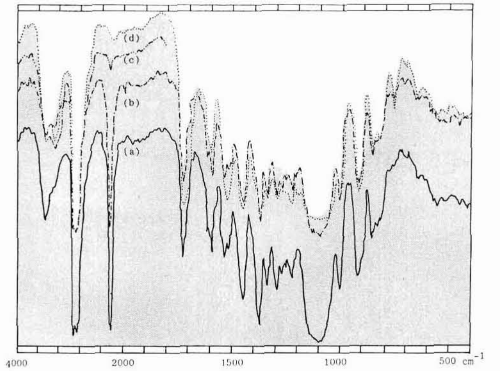


Figure 1—Infrared spectra of (a) PPG(#2000)/TDI = 3/4- and (b, c, d) PPG(#2000)/TDI/glycerine = 3/4/2-prepolymers at different reaction temperatures. (a): room temperature, 30 min; (b): 100°C, 30 min; (c): 120°C, 90 min; (d): 120°C 120 min

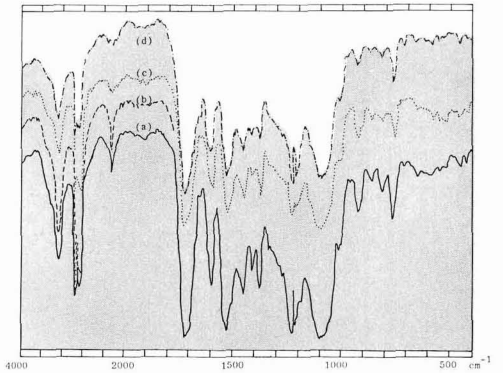


Figure 2—Infrared spectra of the crosslinked films prepared from PPG(#1000)/TDI/glycerine = 2/3/2 prepolymers with equivalent phenol-masked urethane triisocyanate at 220°C for different curing times. (a): 10 min; (b): 20 min; (c): 100 min; (d): 120 min

urethane prepolymer and an equivalent amount of blocked isocyanate in m-cresol. Thin 10 cm diameter circles of the above solution were spread on aluminum foil. Upon baking at 220°C for 40 min in an air oven, a crosslinked film was formed which adhered to the aluminum foil. The foil was dissolved in an HCl (6N) at room temperature for 4-5 min to release the crosslinked polyurethane film.

Wire Coating

A 40% solid content solution was prepared by dissolving and mixing urethane prepolymer and an equivalent blocked isocyanate in m-cresol and naphtha. (Naphtha was used to adjust the viscosity of the solution.) The solution was charged into a 15 cm long test tube. The tube was covered with a steel plate with a small round hole in the center as a die. A segment of clear copper wire of 0.6 mm in diameter and 20 cm long was inserted into the tube through the die and drawn back to be baked at 220°C for 20 min in an air oven. The progressively larger dies were replaced and the same procedures were repeated four times to give polyurethane coated magnet wires with overall diameter within 0.672 mm.

Magnet Wire Test

The manufactured magnet wires were all tested according to Testing Method JIS-C-3211:

(1) Appearance: The surface of the specimen must be smooth and glossy and the specimen cannot adhere at room temperature.

(2) Pinhole test: Testing solution: 0.2% NaCl + HCl (4 drops) + phenolphthalein (2 drops); Positive pole: metallic plate; Negative pole: coated magnetic wire.

Half reaction: Cathode: $2\,H_2O + 2\,e^- \longrightarrow H_2 + 2\,OH^-$
Anode: $2\,H_2O \longrightarrow 4\,H^+ + O_2 + 4\,e^-$
$2\,Cl^- \longrightarrow Cl_2 + 2\,e^-$

Power: DC 12 volts; Time: 1 min. If a red bubble appears, the specimen is rejected.

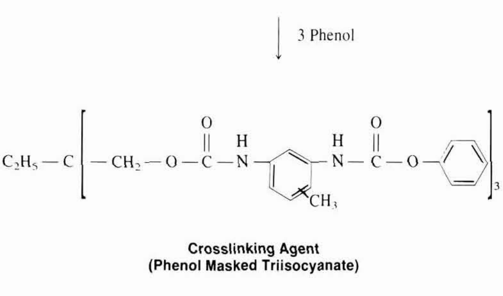
(3) Flexibility test: The specimen is continuously coiled 10 turns. The film of the specimen cannot crack.

(4) Aging test: The specimen is heated at 170°C for 6 hr in an oven and then coiled two turns. The film of the specimen is then coiled two turns. The film of the specimen cannot crack.

(5) Heat shock: The specimen is coiled three turns and then heated at 130°C for 1 hr. The film of the specimen cannot crack.

(6) Acid resistance: The specimen is immersed in sp. gr. 1.2 H_2SO_4 for 24 hr. The film of the specimen cannot bubble, swell, and break away.

(7) Alkali resistance: The specimen is immersed in NaOH (1%) for 24 hr. The film of the specimen cannot bubble, swell, and break away.



Crosslinking Agent (Phenol Masked Triisocyanate)

Scheme 2

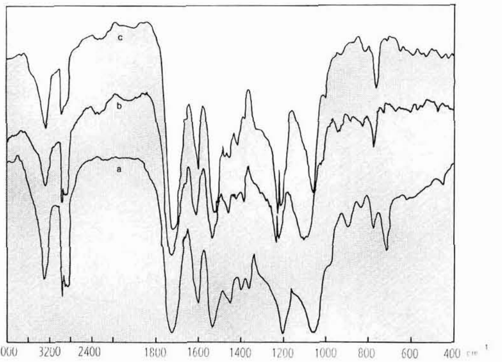


Figure 3—Infrared spectra of the crosslinked films prepared from a: TMP-; b: glycerine-; c: PE-terminated prepolymers

(8) Oil resistance: The specimen is immersed in insulated oil for 24 hr. The film of the specimen cannot bubble, swell, and break away.

(9) Solvent resistance: The specimen is immersed in benzene for 24 hr. The film of the specimen cannot bubble, swell, and break away.

Mechanical Properties Test

The crosslinked polyurethane film was cut into a dumbbell shape with a length of 7 cm, a breadth of one cm at the neck, and a thickness less than 0.5 mm. An Instron Universal Tester with a load cell of 50 kg was used to study the stress-strain behavior of the sample at room temperature at a drawing speed of 5 cm/min.

RESULTS AND DISCUSSION

Syntheses of Urethane Prepolymers

TDI was reacted with PG to give isocyanate-terminated polyurethane, and then glycerine, PE, or TMP was added to produce hydroxyl-terminated urethane prepolymers, as shown in *Scheme* 1, where R and T represent PG and polyol segments. Changing the molar ratio of PG/TDI and using different PG gives single molecule type urethane prepolymers of different molecular weights.

IR spectrum was used to observe the formation of urethane prepolymer. In *Figure* 1, (a) is the IR spectrum of PPG(#2000)/TDI = 3/4-isocyanate-terminated polyurethane, (b) is the IR spectrum of the product of the above prepolymer terminated with a calculated quantity of glycerine after being heated from room temperature to 100°C within one hour and kept at 100°C for 30 min, and (c) and (d) represent the IR spectra of the mixture which was heated at 120°C for 1.5 hr and 2 hr, respectively. IR spectra show that the intensity of the isocyanate absorption peak at 2250 cm^{-1} decreased on heating. The disappearance of the isocyanate peak at 2250 cm^{-1} is due to the completion of reaction.

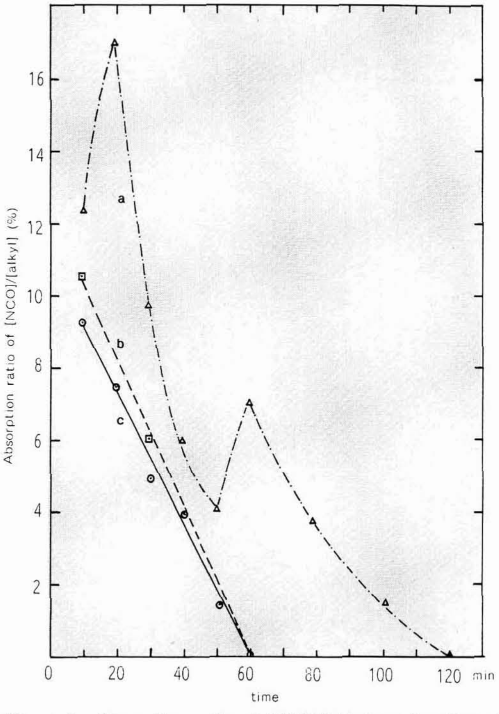


Figure 4—Absorption ratio of (NCO)/(alkyl) as function of curing time. a: glycerine-; b: TMP-; c: PE-terminated prepolymers at the molar ratio of 2/3/2

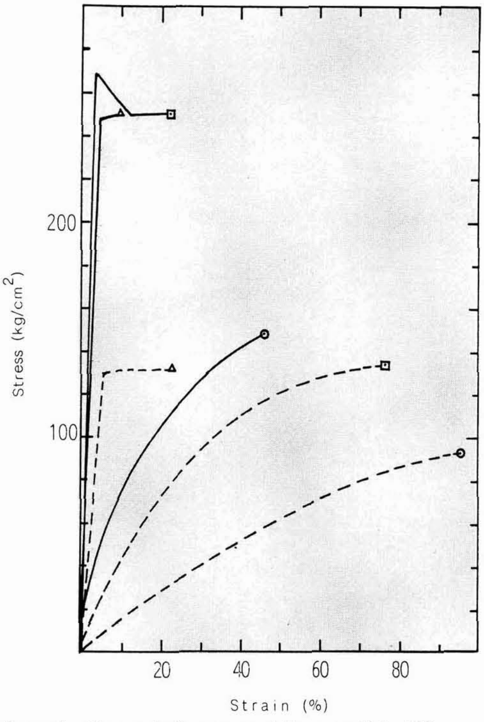


Figure 5—Stress-strain curves of the crosslinked films prepared from PPG(#1000)-TDI-T-urethane prepolymers at the molar ratio of PPG/TDI/T =(—): 1/2/2; (---): 2/3/2. Where T was (Δ): PE-; (⊙): glycerine-; (⊡): TMP-prepolymers

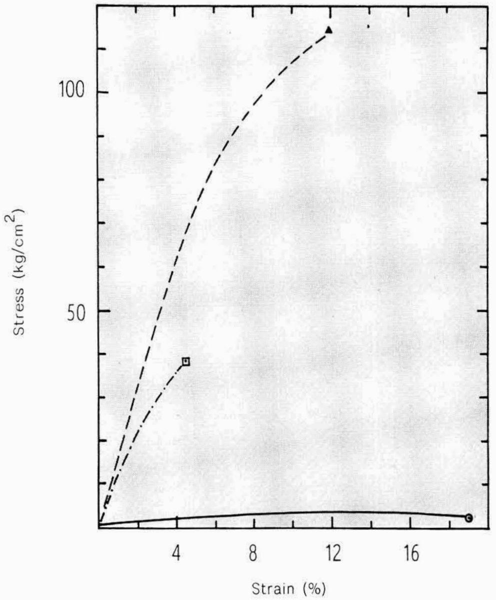


Figure 6—Stress-strain curves of the crosslinked films prepared from PPG(#2000)-TDI-T-prepolymers at the molar ratio of PPG/TDI/T = 1/2/2. Where T was (▲): PE-; (⊙): glycerine-; (⊡): TMP-prepolymers

Synthesis of Crosslinking Agent

There are two procedures for the synthesis of crosslinking agent of polyurethane from phenol, TMP, and TDI. One is that 1 mole of TMP was initially reacted with 3 moles of TDI at a lower temperature, about 10-30°C, and then treated with 3 moles of phenol at a higher temperature to obtain the crosslinking agent. Another is that the mixture of TMP and phenol was added dropwise into the TDI at low temperature followed by elevating the reaction temperature. Comparatively, the latter is easier for giving a uniform low molecular weight crosslinking agent than the first procedure does in absent solvent. In the low temperature, phenol acts as a diluting agent. The formation of crosslinking agent is shown in *Scheme* 2.

IR Spectra of the Crosslinking Reaction

IR spectra of the crosslinked films prepared from PPG(#1000)/TDI/glycerine = 2/3/2-containing urethane prepolymer with an equivalent blocked isocyanate at 220°C for different curing times are shown in *Figure* 2. From the change of isocyanate absorption peak at 2250 cm^{-1}, it can be observed that when the crosslinking reaction of blocked isocyanate and urethane prepolymer took place, the blocked isocyanate deblocked phenol first and then reacted with hydroxyl groups of urethane pre-

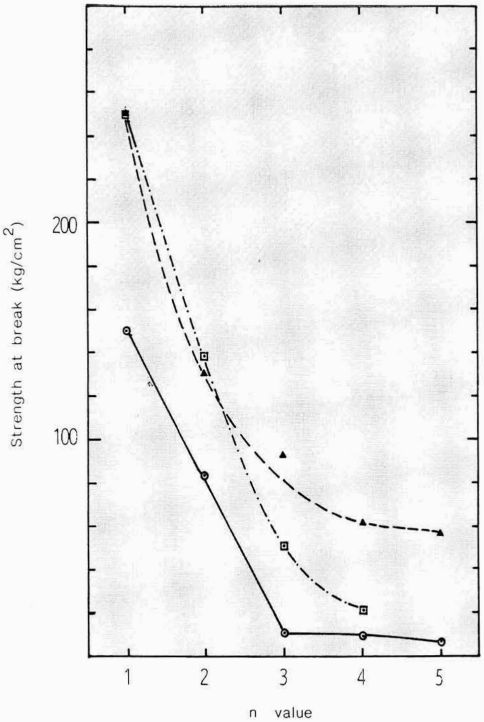


Figure 7—The chain length (average n value) of prepolymer dependence of strength at break for the crosslinked films prepared from PPG(#1000)-TDI-(▲): PE-; (⊙): glycerine-; (⊡): TMP-prepolymers

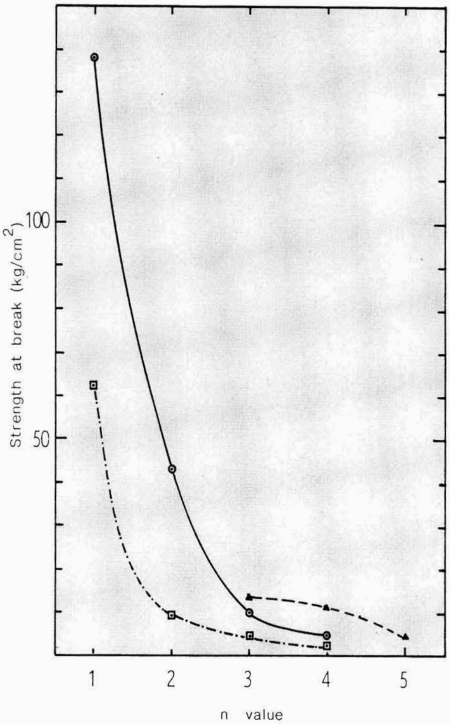


Figure 8—The chain length (n value) of prepolymer dependence of strength at break for the crosslinked films prepared from PEG(#1000)-TDI-(▲): PE-; (⊙): glycerine-; (⊡): TMP-prepolymers

polymer. However, the rate of deblocking phenol to regenerate isocyanate groups is faster than that of the reaction of urethane prepolymer with isocyanate groups. The isocyanate absorption peaks are observable in IR spectra.

Figure 3 represents IR spectra of the crosslinked films prepared from PPG(#1000)/TDI/glycerine = 2/3/2-, PPG(#1000)/TDI/PE = 2/3/2-, and PPG(#1000)/TDI/TMP = 2/3/2-containing urethane prepolymers. It can be observed that all the crosslinked films show similar absorption peaks.

The ratio of the intensity of the absorption peaks of isocyanate groups at 2250 cm^{-1} to that of alkyl groups at 2900 cm^{-1} is varied with the curing time, as shown in *Figure* 4. *Figure* 4a shows that the isocyanate group is regenerated first and then reacted with the hydroxyl groups of the prepolymer to form urethane linkages. The absorption ratio of (NCO)/(alkyl) increased initially, and then gradually decreased. In about 50-60 min, the ratio of (NCO)/(alkyl) increased again. This feature is caused by the fact that the reactivity of the secondary hydroxyl group of glycerine is slower than that of the primary hydroxyl groups. After 60 min, the ratio of (NCO)/(alkyl) decreased rapidly because no new isocyanate groups were regenerated. The absorption ratios of (NCO)/(alkyl) of the crosslinked films prepared from PE- or TMP-terminated urethane prepolymer decreased linearly with the curing time and approached zero after baking at 60 min. This phenomenon is due to the reactivity of hydroxyl groups of PE- and TMP-terminated urethane prepolymers which are the same.

Mechanical Properties

The stress-strain curves of the crosslinked films prepared from different terminated urethane prepolymers are shown in *Figures* 5 and 6. With the use of PPG(#1000) as diol, the tensile strengths of the crosslinked films prepared from PE-terminated prepolymers are about equal to those of prepared from TMP-terminated prepolymers. The crosslinked films prepared from glycerine-terminated prepolymers are weaker and softer than those prepared from PE- or TMP-terminated prepolymers. On the other hand, the elongations at break of the crosslinked films prepared from glycerine-terminated prepolymers are larger than those of PE- or TMT-containing crosslinked films. In the case of using PPG(#2000) as diol, the tensile strengths of PE-containing crosslinked films are stronger than those of TMP-containing crosslinked films. The elongation at break of the crosslinked films prepared from glycerine-terminated prepolymers are also larger than those of PE- or TMP-containing crosslinked films.

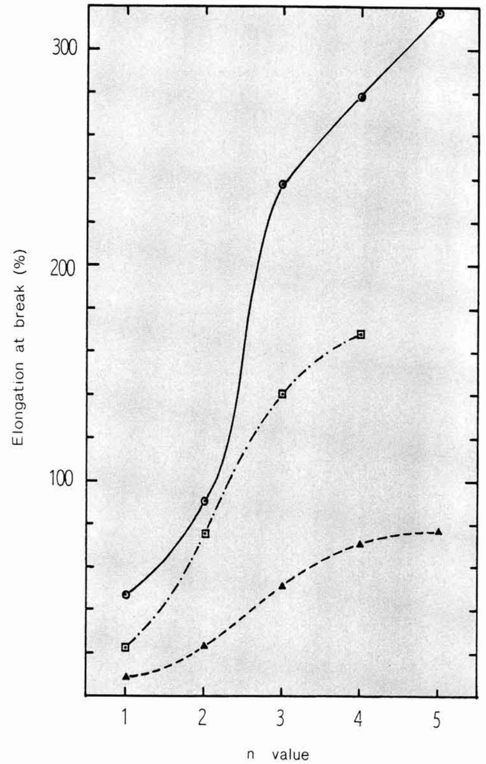


Figure 9—Chain length (n value) of urethane prepolymer dependence of elongation at break for the crosslinked films prepared from PPG(#1000)-TDI-(▲): PE-; (⊙): glycerine-; (⊡): TMP-prepolymers

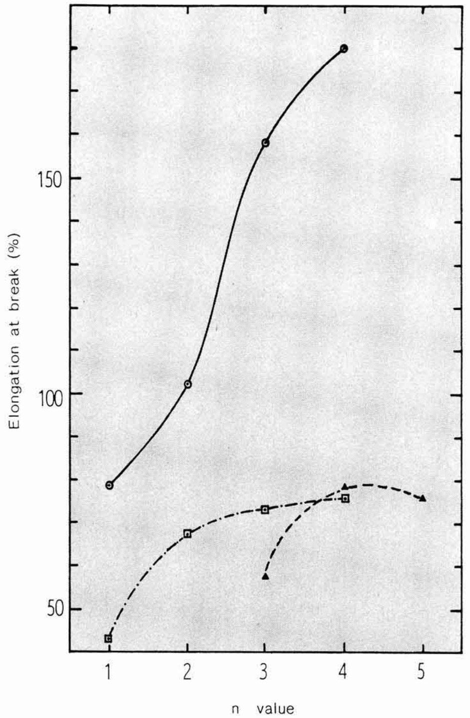


Figure 10—Chain length (n value) of urethane prepolymer dependence of elongation at break for the crosslinked films prepared from PEG(#1000)-TDI-(Δ): PE-; (⊙): glycerine-; (⊡): TMP-prepolymers

The chain length (average n value) of urethane prepolymer dependence of the strengths at break for the crosslinked films prepared from different prepolymers are shown in *Figures* 7 and 8. It can be observed that the longer the chain length is, the weaker the strength. When the n values are between the range of 1 and 3, strength at break decreases rapidly, but there is no marked change in strength after the n value is larger than 3. This phenomenon is because the increase of n value leads to a decrease of the crosslink density, resulting in a decrease of tensile strength with the increase of n value.

The chain length of prepolymer dependence of elongation at break for the crosslinked films prepared from different prepolymers is shown in *Figures* 9 and 10. It can be seen that the longer the chain is, the greater the elongation at break.

The stress-strain curves of the crosslinked films prepared from prepolymers made with different polyols are shown in *Figures* 11-13. These curves show that the crosslinked films prepared from PPG(#1000)-containing prepolymers are stronger than those prepared from PEG(#1000) or PPG(#2000)-containing crosslinked films. The elongations of PEG(#1000)-containing crosslinked films are greater than those of PPG(#1000) or PPG(#2000)-containing films. The strength and elonga-

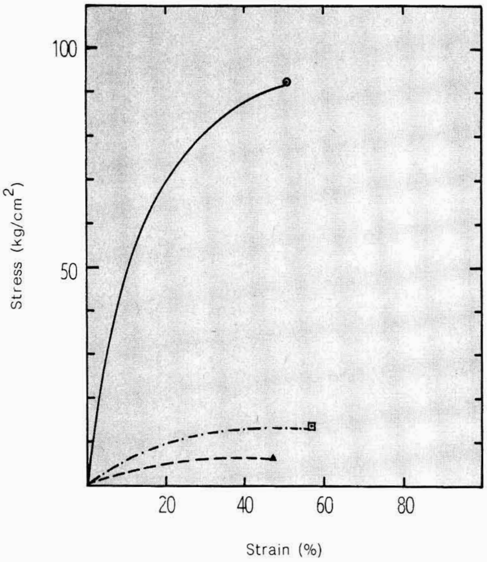


Figure 12—Stress-strain curves of the crosslinked films prepared from PG/TDI/PE=3/4/2-prepolymers with different PG-content. (⊙): PPG(#1000); (⊡): PEG(#1000); (▲): PPG(#2000)

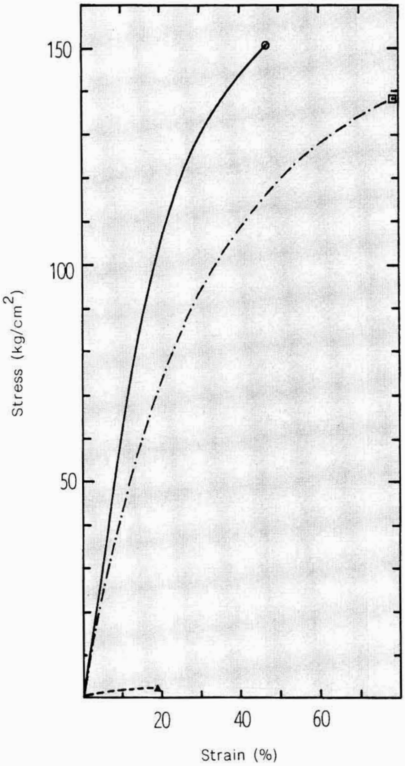


Figure 11—Stress-strain curves of the crosslinked films prepared from PG/TDI/glycerine=1/2/2 prepolymers with different PG-content. (⊙): PPG(#1000); (⊡): PEG(#1000); (▲): PPG(#2000)

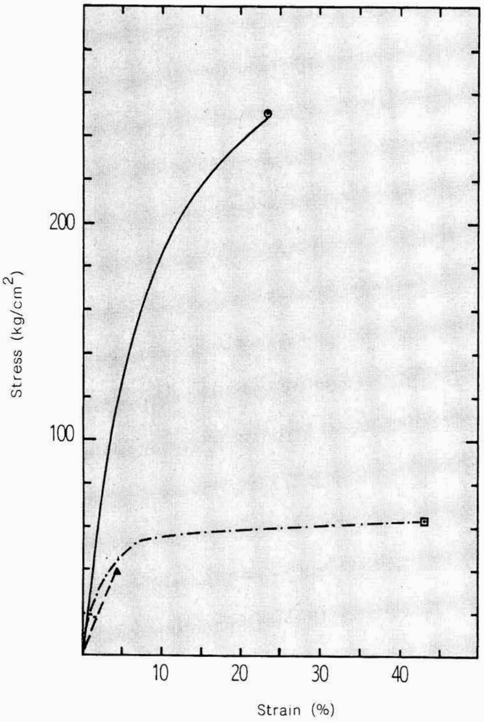


Figure 13—Stress-strain curves of the crosslinked films prepared from PG/TDI/TMP=1/2/2-prepolymers with different PG-content. (⊙): PPG(#1000); (□): PEG(#1000); (▲): PPG(#2000)

Table 1—Test Results of PG/TDI/$C_3H_5(OH)_3$-containing Polyurethane Magnet Wires

Polyglycol	Molar Ratio PG/TDI/$C_3H_5(OH)_3$	Appearance	Pin hole	Flexibility	Aging Test	Heat Shock	Acid Resist.	Alkali Resist.	Oil Resist.	Solvent	Remarks
PPG(#1000)	1 : 2 : 2	O[a]	O	O	O	O	O	O	O	O	a
	2 : 3 : 2	O	O	O	O	O	O	O	O	O	a
	3 : 4 : 2	O	O	O	O	O	O	O	O	O	a
	4 : 5 : 2	X	X	O	O	O	O	O	O	O	a
	5 : 6 : 2	X	X	X	O	O	O	O	O	O	a
PPG(#2000)	1 : 2 : 2	X	X	X	X	X	O	O	O	O	r
	2 : 3 : 2	X	X	X	X	X	O	O	O	O	r
	3 : 4 : 2	X	—	—	—	—	—	—	—	—	r[b]
	4 : 5 : 2	X	—	—	—	—	—	—	—	—	r[b]
PEG(#1000)	1 : 2 : 2	O	O	O	O	O	X	O	O	O	r
	2 : 3 : 2	O	X	O	O	O	X	O	O	O	r
	3 : 4 : 2	O	X	O	O	O	X	O	O	O	r
	4 : 5 : 2	X	X	O	O	O	X	O	O	O	r[b]
	5 : 6 : 2	X	—	—	—	—	—	—	—	—	r[b]

(a) O: accepted; X: rejected; a: accepted; r: rejected; —: unmeasured.

(b) The manufactured magnet wires adhered at ambient temperature.

tion of PPG(#2000)-containing crosslinked films are less and lower than those of the other crosslinked films.

Test Results of Magnet Wires

Test results of the magnet wires are listed in *Tables* 1-3. The magnet wires prepared from PPG(#1000)/TDI/glycerine = 3/4/2, and 2/3/2, and 1/2/2-prepolymers have tested satisfactory to the Testing Method JIS-C-3211. With prepolymers with the molar ratios of PPG(#1000)/TDI/glycerine = 4/5/2 and 5/6/2, the length of PG/TDI segment increases and the crosslink density decreases in crosslinked films, so that magnetic wires manufactured in these molar ratios are rejected because they adhere at room temperature and their pinhole tests are unsatisfactory. All PPG(#2000)-modified polyurethane magnet wires made from PPG(#2000)-TDI-glycerine-coating varnishes tested unsatisfactory.

The magnet wires prepared from PPG(#1000)/TDI/PE = 5/6/2, 4/5/2, 3/4/2, 2/3/2, and 1/2/2 tested satisfactory to the Testing Method. With prepolymers with the molar ratios of PPG(#1000)/TDI/PE = 6/7/2, 7/8/2, and 8/9/2, the molecular weights are too large to dissolve uniformly in m-cresol and naphtha, so these three are not suitable for producing magnet wires. The magnet wires manufactured from PPG(#2000)/TDI/PE-containing prepolymers are acceptable only at a molar ratio of PPG(#2000)/TDI/PE = 1/2/2.

The PPG(#1000)/TDI/TMP = 4/5/2, 3/4/2, 3/4/2, 2/3/2, and 1/2/2-containing prepolymers can give excellent

Table 2—Test Results of PG/TDI/$C_3H_5(OH)_3$-containing Polyurethane Magnet Wires

Polyglycol	Molar Ratio PG/TDI/$C(CH_2OH)_4$	Appearance	Pin hole	Flexibility	Aging Test	Heat Shock	Acid Resist.	Alkali Resist.	Oil Resist.	Solvent	Remarks
PPG(#1000)	1 : 2 : 2	O[a]	O	O	O	O	O	O	O	O	a
	2 : 3 : 2	O	O	O	O	O	O	O	O	O	a
	3 : 4 : 2	O	O	O	O	O	O	O	O	O	a
	4 : 5 : 2	O	O	O	O	O	O	O	O	O	a
	5 : 6 : 2	O	O	O	O	O	O	O	O	O	a
	6 : 7 : 2	—	—	—	—	—	—	—	—	—	i[b]
	7 : 8 : 2	—	—	—	—	—	—	—	—	—	i
	8 : 9 : 2	—	—	—	—	—	—	—	—	—	i
PPG(#2000)	1 : 2 : 2	O	O	O	O	O	O	O	O	O	a
	2 : 3 : 2	O	X	X	X	X	O	O	O	O	r
	3 : 4 : 2	O	X	X	X	X	O	O	O	O	r
	4 : 5 : 2	X	X	X	X	X	O	O	O	O	r
	5 : 6 : 2	X	—	—	—	—	—	—	—	—	r[c]
PEG(#1000)	3 : 4 : 2	O	X	O	O	O	X	O	O	O	r
	4 : 5 : 2	O	X	O	O	O	X	O	O	O	r
	5 : 6 : 2	O	X	O	O	O	X	O	O	O	r
	6 : 7 : 2	X	—	—	—	—	—	—	—	—	r[c]
	7 : 8 : 2	X	—	—	—	—	—	—	—	—	r[c]

(a) O: accepted; X: rejected; a: accepted; r: rejected; —: unmeasured.

(b) The urethane prepolymer was insoluble in solvents.

(c) The manufactured magnet wires adhered at ambient temperature.

Table 3—Test Results of PG/TDI/TMP-containing Polyurethane Magnet Wires

Polyglycol	Molar Ratio PG/TDI/TMP	Appearance	Pin hole	Flexibility	Aging Test	Heat Shock	Acid Resist.	Alkali Resist.	Oil Resist.	Solvent	Remarks
PPG(#1000)	1 : 2 : 2	O[a]	O	O	O	O	O	O	O	O	a
	2 : 3 : 2	O	O	O	O	O	O	O	O	O	a
	3 : 4 : 2	O	O	O	O	O	O	O	O	O	a
	4 : 5 : 2	O	O	O	O	O	O	O	O	O	a
	5 : 6 : 2	X	—	—	—	—	—	—	—	—	r[b]
PPG(#2000)	1 : 2 : 2	O	X	O	O	O	O	O	O	O	r
	2 : 3 : 2	X	X	X	X	X	X	O	O	O	r[b]
	3 : 4 : 2	X	X	O	X	X	X	O	O	O	r[b]
PEG(#1000)	1 : 2 : 2	O	X	O	O	O	X	X	O	O	r
	2 : 3 : 2	O	X	O	O	O	X	X	O	O	r
	3 : 4 : 2	O	X	O	O	O	X	X	O	O	r
	4 : 5 : 2	X	X	O	O	O	X	X	O	O	r[b]
	5 : 6 : 2	X	X	O	O	O	X	X	O	O	r[b]

(a) O: accepted; X: rejected; a: accepted; r: rejected; —: unmeasured.
(b) The manufactured magnet wires adhered at ambient temperature.

quality magnet wires and pass the Testing Method. The magnet wires manufactured from PPG(#1000)/TDI/TMP = 5/6/2-prepolymers tested unsatisfactory because they adhered at room temperature. All PPG(#2000)-modified polyurethane magnet wires were rejected as a result of poor aging performance. All PEG(#1000)-modified polyurethane magnet wires were also rejected because of pinholing, poor acid resistance, and alkali resistance.

From the test results of magnet wires previously described, it can be inferred that excellent quality magnet wires could be prepared by using PPG(#1000) as diol and the chain length of prepolymers be shorter than those of PPG(#1000)/TDI/glycerine = 3/4/2, PPG(#1000)/TDI/PE = 5/6/2, or PPG(#1000)/TDI/TMP = 4/5/2-containing prepolymers. The purpose of adding naphtha to varnishes is to reduce the viscosity of varnishes so that the wire coating will be smooth.

ACKNOWLEDGMENT

The authors are grateful to Dr. T. S. Lin, President of Tatung Institute of Technology, for his encouragement and support.

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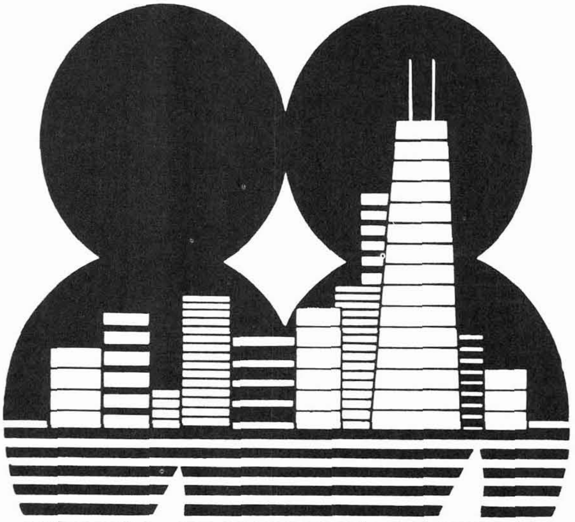

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Study of the Parameters Affecting the Emulsion Polymerization Of Vinyl Acetate

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The emulsion polymerization of vinyl acetate initiated by potassium persulfate in presence of Ag(I) and other metallic ions was studied. During this study, an ionic surfactant, sodium lauryl sulfate, and Igepal® CO-970, a nonionic surfactant, were used. The samples obtained were characterized gravimetrically to obtain the reaction yield, and by GPC for the molecular weight.

It was found that silver ions in conjunction with potassium persulfate, in the presence of either surfactant, accelerates the polymerization of the vinyl acetate.

The use of any metallic salt in the presence of Igepal CO-970 improves the overall conversion.

INTRODUCTION

Emulsion polymerization, due to certain particular aspects, such as good temperature control and viscosity, has become one of the important commercial polymerization processes for vinyl acetate, acrylates, and the copolymerization of butadiene and styrene, etc. A further advantage is that the products may be used directly and the required additives may be blended in.

With a good selection of monomers, initiators, surfactants, and reaction conditions, a great variety of products may be manufactured, each meeting the specifications for its applications. The fields covered by these applications run from paints, coatings, adhesives, and floor polishes to paper coatings.

Early work[1,2] on the polymerization of vinyl acetate suggested that it followed case II of the Smith-Ewart theory.[3,4] Further work by Priest,[5] however, suggested that the initiation occurred in the aqueous phase; this was confirmed by the work of Napper and Parts[6] and of Dunn and Taylor.[7] The kinetic aspect of the reaction was studied by Litt, Patsiga, and Stannett.[8] They showed that the reaction rate was of the order one, when compared to the concentration of the initiator; whereas it is of the order zero, when compared to the surfactant concentration.

The results obtained by the various workers seem contradictory, and none of the previously suggested models for emulsion polymerization kinetics applies to the experimental data.

According to certain researchers,[9] these apparent contradictions are not due to faulty experimental procedures, but are the result of a change in the ionic force in aqueous medium due to the presence of potassium persulfate.

Other workers have already shown the influence of metallic ions in the reaction medium. In the polymerization of acrylamide, Singh and coworkers[10] have shown that silver ions improve the reaction rate, whereas the presence of cuprous ions have a negative effect on the polymerization.

Bataille and coworkers,[11] working with styrene and potassium persulfate, have again shown that the silver ions have a positive effect on the reaction rate, whereas the presence of ferrous ions reduces both the reaction rate and the molecular weight. Dash and coworkers,[12] working with methyl methacrylate, showed that the polymerization rate is accelerated in the presence of silver ions, whereas it is slightly increased in the presence of Mn(II) ions, and depressed in the presence of Cu(II) and Zn(II) ions. The overall conversion is furthermore decreased in the presence of Cu(II) or Zn(II) ions.

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Table 1—General Formulation

Materials	Function	Quantity (grams in the reactor)
Vinyl acetate	Monomer	167.707
Potassium persulfate ...	Initiator	0.11
Sodium lauryl sulfate...	Surfactant[a]	3.30
Igepal CO-970	Surfactant[a]	1.9
Demineralized water ...	Dispersing medium	500.0

(a) The surfactants were not used simultaneously.

Table 2—Percent of Conversion After 40 Minutes of Reaction Time

Metallic Salts 1.35 × 10^{-5} mol/L	% Conversion
Reference	43
$AgNO_3$	66
Fe $(NO_3)_3$	57
$MnSo_4$.................................	53
Co $(NO_3)_2$..............................	20
Cu $(NO_3)_2$..............................	4

Samal and coworkers,[13] working with methyl methacrylate and $KHSO_3$ in the presence of cobalt (II) ions, noticed that the reaction rate increased as the level of the cobalt ions increased, up to a concentration of 5×10^{-4} M of Co(II) ions. Both the conversion and the conversion rate decreased as the cobalt ion concentration was further increased. All this illustrates that the quality of the water used in emulsion polymerization is of prime importance.

This is of particular interest in a research program on continuous polymerization;[14] one must take into consideration the material which the reactor is made of and develop, if possible, an improved initiator system.

As the effect of the ion varies with the polymerization system, we studied the effect of the metallic salts $AgNO_3$, $CoCl_2$, $Co(NO_3)_2$, $CoSO_4$, $Cu(NO_3)_2$, $MnSo_4$, and $Fe(NO_3)_3$ on the conversion, reaction rate, and molecular weight of the vinyl acetate polymerization in a batch system.

MATERIALS AND METHODS

The monomer used (vinyl acetate) was obtained from Aldrich Chemical at 99% purity and was redistilled to eliminate the inhibitors. After distillation, the monomer was kept in a brown bottle and placed in a freezer at a temperature of −9.5°C.

The initiator used was potassium persulfate, $K_2S_2O_8$, supplied by Anachemia Chemical. A reagent grade with 99.5% purity, it was recrystallized in demineralized water.

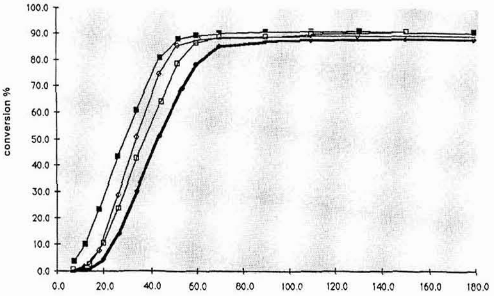

Figure 1—Conversion vs reaction time—surfactant SLS: ♦: reference; □: $AgNO_3$, 1.31 × 10^{-6} mol/L; ◇: $AgNO_3$, 1.47 × 10^{-4} mol/L; ■: $AgNO_3$, 1.31 × 10^{-5} mol/L

The surfactant, sodium lauryl sulfate (SLS), an ionic surfactant ($C_{12}H_{25}SO_4Na$), was supplied by Fisher Chemical as laboratory grade. It was used as such and its critical micelle concentration (CMC) was 7.8-8.2 μM.[15]

The other surfactant was Igepal CO-970, a nonionic surfactant, supplied by G.A.F. Corporation—it has the chemical formula $C_9H_{19}C_6H_4(OCH_2\text{-}CH_2)_mOH$ where m = 50. It has a CMC of 2.8 μM.[15]

The metallic salts were obtained from various companies: Anachemia, Analar, Fisher, and Baker, and were of reagent, ACS grade, analytical certified ACS, or analytical reagent grades, respectively.

Demineralized water was used throughout as a dispersing medium and nitrogen gas blanket was used as well. To stop the polymerization, an aqueous solution of 0.0423 g/L of hydroquinone was used. Using 2 mL of the aqueous solution allowed for 15-20 ppm of hydroquinone in each sample taken during the reaction.

Polymerization was carried out essentially the same way as described elsewhere.[16] However, great care was taken to eliminate as much oxygen as possible from the reaction medium, and during the reaction by bubbling the nitrogen through sulfuric acid before using it as a blanket over the reacting emulsion. The reactor consisted of a one liter glass vessel having a flange closing system. The cover itself had four openings, permitting easy access to the mixture for the various control, agitation, addition, or sampling systems.

The reaction temperature was kept at 60°C, with an agitation of 165-170 rpm. The general formulation used is give in *Table* 1. Samples (≈ 12) were taken throughout the reaction and analyzed for conversion, molecular weight distribution polydispersity, and particle size.

The conversion was obtained by gravimetric analysis (i.e., after exposure to an infrared lamp, the drying procedure was completed under vacuum, and after correction, the conversion was calculated). The molecular weight and molecular weight distribution were determined by a Waters Associates GPC-200 model using four columns, having pore size of 10^4 Å to 10^7 Å. The sample concentrations were kept at 0.4% w/v and the solvent for the GPC work was tetrahydrofuran.

The micrographs were obtained by using a scanning electronic microscope model JSM-840 of Japan Electron Optics Laboratory.

Viscosity measurements were also conducted on the latex samples at different reaction times and for different reaction conditions (i.e., with and without metallic salts).

For all intents and purposes, no marked changes were noticed.

ANALYSIS OF THE REACTION

Silver Ion Catalyzed Polymerization

In *Figure* 1, we have plotted the conversion vs the reaction time. Three different concentrations of $AgNO_3$ were used: 1.31×10^{-6} mol/L, 1.31×10^{-5} mol/L, and 1.47×10^{-4} mol/L. One can notice three peculiar phenomena. In all the cases where the silver ion is present, we have both a decrease of the induction time combined with an improvement in the total conversion. Furthermore, there is an optimum conversion concentration, i.e., 1.31×10^{-5} mol/L of $AgNO_3$.

The increased polymerization or decreased induction time might be explained by the ionic condition of the emulsion. The rate of decomposition of potassium persulfate in a saturated aqueous vinyl acetate solution (0.3 mol/L) is about ten times higher[17] than in pure water and the presence of extra ions Ag^+ or NO_3^- might alter this balance either way.

One might explain the reaction kinetics by the following equations:

$$S_2O_8^{=} + M \xrightarrow{k_1} R_i^{\cdot} + SO_4^{\dot{-}} \qquad (1)$$

$$SO_4^{\dot{-}} + M \xrightarrow{k_2} R^{\cdot} \qquad (2)$$

where M is the monomer.

In the presence of a metallic ion such as silver we would have the following equations:

$$Ag^+ \text{ (aqueous)} + S_2O_8^{=} \xrightarrow{k} AgS_2O_8^{-} \text{ (aqueous)} \qquad (3)$$

$$AgS_2O_8^{-} \text{ (aqueous)} + M \xrightarrow{k_p} M^{\cdot} + Ag^+ \text{ (aqueous)}\ HSO_4^{-} + SO_4^{\dot{-}} \qquad (4)$$

$$M^{\cdot} + M \longrightarrow R^{\cdot} \qquad (5)$$

The silver ion, by the reactions indicated in equations (3) and (4), enhances the reaction rate of polymerization by decreasing the overall activation energy.

Some investigators[18] have found that there was relatively low end-group content in polymers initiated by $Ag^+S_2O_8^{=}$ and they attributed this to the scavenging activity of silver ions for the sulfate ion radicals.

$$Ag^+ + SO_4^{\dot{-}} \longrightarrow Ag^{++} + SO_4^{=} \qquad (6)$$

$$Ag^{++} + SO_4^{\dot{-}} \longrightarrow Ag^{+++} + SO_4^{=} \qquad (7)$$

This also suggests that the silver ions can act as a transfer agent, which would normally decrease the molecular weight as observed by certain workers.[11] In our case, however, as can be seen in *Figure* 2, after a very gradual increase in the molecular weight up to a conversion of 80%, the molecular weight increases abruptly. This rapid rise is due to the increased amount of long chain branches per molecule of vinyl acetate polymer which occurs as the polymerization progresses. This phenomena confirms results obtained by other workers.[19]

Polymerization Catalyzed by Cobalt, Copper, and Iron Ions

In *Figure* 3, the conversion of vinyl acetate vs time in the presence of various metallic salts is shown.

A standard concentration of 1.35×10^{-5} mol/L of metallic salt was used throughout this series of experiments, as this concentration gave the best results for the silver nitrate.

The induction period in the presence of the Ag (I), Fe (III), and Mn (II) ions is much shorter. The only difference between the curves with these salts and the reference run without them is the induction period. Globally, the reaction rate between these four cases is the same, and the curves between 15-80% conversion run parallel. The Ag (I) ion, however, has a slight edge.

The addition of a cobalt salt does not change the reaction rate but causes a much longer induction time.

Another way of looking at this is the conversion after reacting for 40 min (see *Table* 2). These figures were actually taken from the reaction results of *Figure* 3.

If we compare the reaction rate of Co (II) and Cu (II), we notice that they both have identical induction periods,

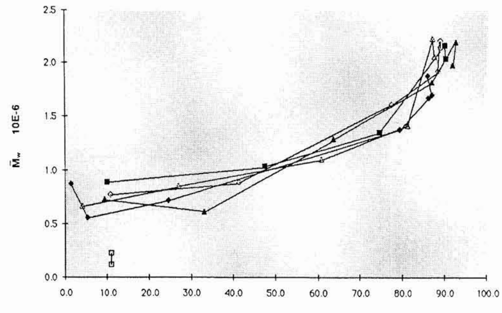

Figure 2—Variation of weight average molecular weight ($\bar{M}_w$) vs conversion—surfactant SLS: ♦: reference; ◇: $AgNO_3$, 1.35×10^{-5} mol/L; ■: Co $(NO_3)_2$, 1.35×10^{-5} mol/L; □: Cu $(NO_3)_2$, 1.35×10^{-5} mol/L; ▲: Fe $(NO_3)_3$, 1.35×10^{-5} mol/L; Δ: $MnSO_4$, 1.37×10^{-5} mol/L

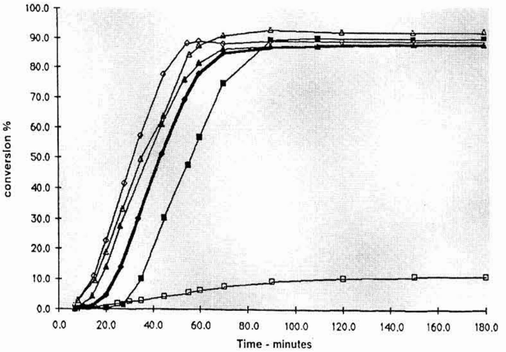

Figure 3—Conversion vs reaction time—surfactant SLS: ♦: reference; ◇: $AgNO_3$, 1.35×10^{-5} mol/L; ■: Co $(NO_3)_2$, 1.35×10^{-5} mol/L; □: Cu $(NO_3)_2$, 1.35×10^{-5} mol/L; ▲: $MnSO_4$, 1.35×10^{-5} mol/L; Δ: Fe $(NO_3)_3$, 1.35×10^{-5} mol/L

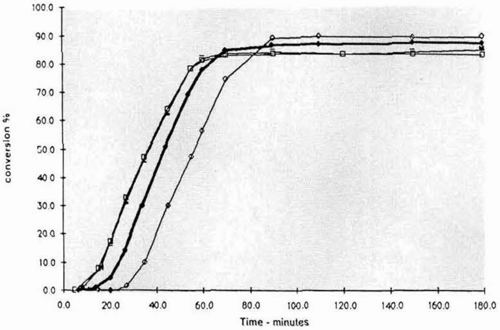

Figure 4—Conversion vs reaction time—effect of anion—surfactant SLS: ♦: reference; ◇: Co $(NO_3)_2$, 1.35 × 10^{-5} mol/L; ■: $CoCl_2$, 1.35 × 10^{-5} mol/L; □: $CoSO_4$, 1.35 × 10^{-5} mol/L

about 25 min. However, the global conversion with Co (II) reaches 90% as compared to 10% with Cu (II). This effect of the Cu (II) is impressive, since it not only affects the yield but also the molecular weight, as it reached about only one third of the value obtained in the other polymerization. These results illustrate that the copper ions act as a transfer agent and deactivate the reaction, thus confirming the results reported by other workers.[20,21] One might explain this behavior by the formation of a stable pair of radicals, Cu (II)-Cu(II), which decompose slowly by formation of a nonreactive complex, Cu (II)-Cu (I),[21] or by the formation of a complex monomer, Cu (II).[22] These effects eliminate the metallic ions which would normally favor the reaction-producing radicals and at the same time make monomers less available.

The presence of the Fe (III) ions did not negatively affect the reaction as compared to the effect of Fe (II) ions in the case of styrene. This discrepancy might be due to the fact that the Fe (II) ions scavenge SO_4- radicals with the formation of Fe (III) ions, while in our case we have no Fe (II) ions but only Fe (III) ions.

$$Fe^{++} + SO_4^{\dot{-}} \longrightarrow Fe^{+++} + SO_4^{=} \qquad (8)$$

As already indicated, the Co (II) ions had a slight negative effect, mainly due to the induction delay phenomena observed by other workers.[13,24]

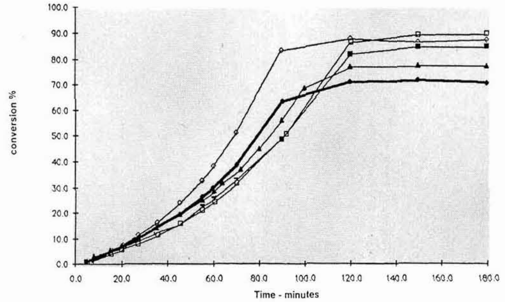

Figure 5—Conversion vs reaction time—surfactant Igepal CO-970: ♦: reference; ◇: $AgNO_3$, 1.35 × 10^{-5} mol/L; ■: Cu $(NO_3)_2$, 1.35 × 10^{-5} mol/L; □: Cu $(Cl)_2$, 1.35 × 10^{-5} mol/L; ▲: $CuSO_4$, 1.35 × 10^{-5} mol/L

Table 3—Effect of the Anion on the Induction Time

Metallic Salt	Induction Time (min)	Maximum Conversion (%)
Ag NO_3 (reference)	14.3	90
Co $(NO_3)_2$	28.5	92
Co Cl_2	8.5	87
Co SO_4	8.5	85

Effect Due to the Anion

In *Figure* 4, we noticed that the reaction containing $CoCl_2$ and $CoSO_4$ salts gave roughly the same results as the $AgNO_3$, Fe $(NO_3)_3$, and M_nSO_4 (shown in *Figure* 3). The anions in these cases do not affect the overall polymerization. For the Co $(NO_3)_2$ case, however, we have a delayed reaction, although the reaction rate is about the same.

It is interesting to note here the effect of the anion on the induction time (see *Table* 3). The $(SO_4^{=})$ and the (Cl −) ions gave a shorter induction time.

Effect Due to the Surfactant

In the previous set of experiments we were using SLS, which is an anionic surfactant. In this set of experiments, whose results are graphically shown in *Figure* 5, we selected two metallic salts, i.e., silver nitrate [Ag(I)] and copper nitrate [Cu (II)] because these salts gave the most diverging results with the ionic surfactant.

Ag (I) gave the best overall results and the Cu (I) the worst overall results (see *Figure* 3). The surfactant used was Igepal CO-970, whose formula was given earlier.

If we examine *Figure* 5, three effects can be noted. First, there is no induction time and none of the salts have a retarding effect; all the reactions start at the same moment. Second, the Ag (I) ion gives a reaction rate much higher than the reference run and the runs with the other salts. Third, in all cases the presence of the metallic salt gives a higher global conversion as compared to the reference run which does not contain any metallic salt. In *Table* 4 we have the percentage of conversion after 90 min of reaction. We notice that the Cu (II) ion still has an inhibiting effect when the conversion is around 50% as compared to 63% for the reference run. This inhibiting effect is, however, much smaller than when SLS is used. The conversion has improved considerably with the Cu (II) as it went from 10% with the anionic surfactant to 85% in this case with the nonionic surfactant.

One must remember that due to the relatively high solubility of vinyl acetate in water, the polymerization occurs both in the organic phase (micelle) and in the

Table 4—Conversion After 90 Minutes of Reaction

Metallic Salt	Conversion (%)	Maximum Conversion
Nil (reference)	63	70
Ag NO_3	83.5	87
Cu $(NO_3)_2$	49	85
Cu SO_4	56	78
Cu Cl_2	51	90

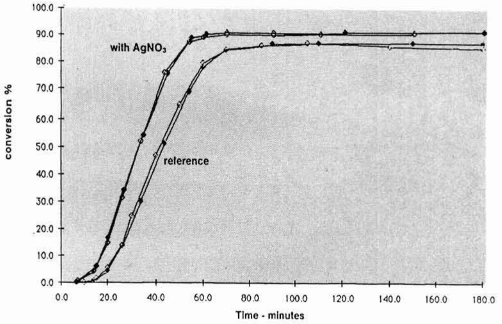

Figure 6—Conversion vs reaction time—reproducibility studies: top two curves: $AgNO_3$, 6.56×10^{-5} mol/L; lower two curves: reference

aqueous phase.[25] We think that in this case, by just changing the surfactant, the polymerization in the organic phase would be the dominant factor. With the surfactant being nonionic, there is no negative charge making an electrostatic barrier at the micelle interface, preventing the radicals of $SO_4^{\dot{-}}$ or of $MSO_4^{\dot{-}}$ from moving in the micelle.

Reproducibility of the Experiments

To have a certain degree of confidence in the results obtained and to evaluate if the results have any degree of significance, the experiments were repeated. In *Figure* 6, we have given two sets of some of the repeated data. From this, we can see that the experiments are reproducible and the differences discussed in the paper are significant.

CONCLUSION

There are no general theories which might satisfactorily explain all the cases studied here. The addition of the Ag (I) ion improved both the reaction rate and the total conversion of the vinyl acetate polymerization using either an anionic or a nonionic surfactant.

When using SLS as a surfactant, each case must be studied separately. We had an improvement in the reaction condition with Ag (I) ions, Fe (III) ions, and Mn (II) ions, whereas we had poorer reaction response (i.e., lower reaction rate and/or total conversion) with Co (II) ions and Cu (II) ions.

The nature of the anion is also important and can go either way. For example, with ($SO_4^{=}$) and (Cl −) ions we had a shorter induction time, but with (NO_3 −) ions we had a longer induction time.

In all the cases where a nonionic surfactant was used with a metallic salt, we always had an improvement in the total conversion.

ACKNOWLEDGMENT

We would like to thank the "Fonds FCAR-ACSAIR" of the province of Quebec for its financial support for this project.

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______	**Infrared Spectroscopy Atlas**	(Mbr.–$75; Non-Mbr.–$100)	______
______	**Color-matching Aptitude Test Set**	($400)	______

Pictorial Standards of Coatings Defects

______ Complete Manual ($90) ______

Individual Standards ($3 ea., plus $3 per photo as noted) ______

______ Adhesion *(1)* ______ Blistering *(4)* ______ Chalking *(1)*

______ Checking *(1)* ______ Cracking *(1)* ______ Erosion *(1)*

______ Fillform Corrosion *(3)* ______ Flaking *(2)*

______ Mildew *(3)* ______ Print *(1)* ______ Rust *(4)*

______ Traffic Paint Abrasion *(2)* ______ Traffic Paint Chipping *(2)*

______ Record Sheets (pad of 100 sheets) ($3.50) ______

______ **Glossary of Color Terms** ($6.00) ______

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Check the <u>one block</u> which applies most specifically to the company or organization with which *you* are affiliated.

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

YOUR POSITION:

Check the <u>one block</u> which best describes *your* position in your company or organization.

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

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June 1987 Subcommittee Reports of ASTM Committee D-1

The June meeting of ASTM Committee D-1 on Paint and Related Coatings and Materials was held on June 14-17, 1987 at the Hyatt Regency Hotel in Dearborn, MI. In the three and one-half days preceding the final session and general meeting of Committee D-1, 135 members and guests met in 135 scheduled meetings of D-1 subcommittees and working task groups. The present membership of Committee D-1 is 523.

Special Highlights of the meeting included talks by Mackenzie K. Endo, of Ford Motor Co., on "Automotive Paint Testing," Leonard Dick, of PPG Industries, Inc., on "The Genesis of Automotive Colors," David Allman, of DuPont, on "Directional Color Measurement of Metallic Finishes," and Ron Steinmayer, of General Motors, on "Vehicle Corrosion Management."

Awards presented included: Society Award of Merit to H. Fujimoto; W.T. Pearce Award to C.K. Schoff (1987); Henry A. Gardner Award to S. LeSota and Certificate of Appreciation to C.J. Sherman.

D-1 Chairman J.C. Weaver has been notified of his selection to receive the SES-ASTM Robert J. Painter Memorial Award, which is given to "an individual for special services in the field of standardization through company program, managerial support, or educational research."

Appointments were made as follows: Sub. D01.23—W.C. Golton as Vice-Chairman; Sub. D01.26—R.T. Marcus as Vice-Chairman; D01.35—T.H. Golson as Vice-Chairman.

The Nominating Committee, S.B. Levinson (Chairman), reported the following slate of nominees for D-1 offices for 1988-89: Chairman—John C. Weaver; 1st Vice-Chairman—Joseph M. Behrle; 2nd Vice-Chairman—Laurance R. Thurman; 3rd Vice-Chairman—Hiro Fujimoto; Recording Secretary—Harold M. Werner; Membership Secretary and Treasurer—Robert A. Orr; Executive Subcommittee Members-At-Large—Eugene A. Praschen (two-year term); Bobby L. Williamson (four-year term); William C. Golton (six-year term); Mary E. McKnight (six-year term); and Mary E. Sites (six-year term).

Highlights

The following items of major interest to the coatings industry warrant special emphasis:

Transfer Efficiency—A Provisional Subcommittee on Transfer Efficiency established two working groups and is starting documents on this subject. See Sub. D01.08 and Sub. D01.55.

VOC Standards Technology Training Course—Sub. D01.21.14 reports on this course and a technical document developed.

Calculation of VOC and Volume Solids—Sub. D01.24.30 is a new task group developing a standard practice on this subject.

Latex Flat Wall Paint Specification—Sub. D01.41 reports on the status of this newly-approved specification, D 4717.

Water Repellency Treatment of Masonry Surfaces—Newly-organized Sub. D01.47 reports on several new projects, including resistance to *acid rain*.

Gouache Paints—Sub. D01.57 reports on a new international task group to develop a quality standard for these artists' paints.

Brush Filaments and Bristles—Sub. D01.61.05 is a new task group to develop a test method for bulk density of these materials.

Future Meetings

January 24-27, 1988—Clearwater Beach, FL (Holiday Inn).
June 26-29, 1988—Baltimore, MD (Committee Week).
January 22-25, 1989—Orlando, FL (Committee Week).

New Standards

New D-1 standards since the January 1987 meetings of D-1 approved by the ASTM Committee on Standards in the months shown: (subcommittee jurisdiction).

APPROVED MAY 1987

D 4706-87, Test Method for Determining Qualitatively Methylol Group in Phenolic Resins (Sub. D01.33)

D4707-87, Test Method for Measurement of Paint Spatter Resistance to Roller Application (Sub. D01.42)

D 4708-87, Practice for Preparation of Free Films of Organic Coatings (Sub. D01.23)

D 4709-87, Specification for Methyl Acrylate (Sub. D01.35)

D 4710-87, Specification for Acetaldehyde (Sub. D01.35)

APPROVED JUNE 1987

D 4712-87, Guide for Testing Industrial Water-Reducible Coatings (Sub. D01.55)

D 4713-87, Test Method for Nonvolatile Content of Printing Inks, Resin Solutions, and Vehicles (Sub. D01.56)

DIVISION 1 ADMINISTRATIVE

SUBCOMMITTEE D01.06 INTERNATIONAL COORDINATION

J.C. Weaver, Chairman

The International Standards Organization, ISO, with general headquarters in Geneva, includes Technical Committee 35 (TC 35) with its Secretariat in Delft, Netherlands. TC 35 on Paints and Varnishes and its various constituent subcommittees, SC-2 Pigments, SC-9 Paints, SC-10 Media, and perhaps others, will hold its plenary meetings September 21-25, 1987 in the Hotel BudaPenta, Budapest, Hungary. (Committee D-1 hosted a similar meeting in April, 1977 at ASTM Headquarters.)

Committee D-1 is only an Observer (O) member of TC 35 because it has not found a cost effective way to be a Participating (P) member via the protocol strictures of ANSI in New York, NY. Nevertheless, certain task groups (TG's) within D-1 subcommittees do, or may, find useful coordinations and even round-robins in selected working groups (WG's) of subcommittees of TC 35.

Internationalizations of industrial corporations accelerate, especially between North America and Western Europe, and make even more desirable some commonalities of membership and selective round-robins between D-1's TG's and TC 35's WG's.

TC 35's Annual Report of 1986, received in March, 1987, lists (1) about 60 (20 P and 40 O) member countries, (2) six subcommittees and over 30 WG's (Germany 12, UK 9), and (3) an 11-page comprehensive status report of actions on nearly 90 items of work. In addition, there are some proposals of work.

Pigment spectrometry by TC 35/SC-2 may be of interest in Sub. D01.26, D01.31, D01.56 and D01.57, of D 4302 and D 4303, toward spectral curves akin to those in the NPIRI (Printing Inks) Volume 4 on Pigments, which were mainly produced at Lehigh University by D01.56 Printing Inks chair, Ms. Jacqueline M. Fetsko.

Toxic metals in general and selenium in particular need analytical methods for "soluble" metal content and will be addressed by TC 35/SC-9/WG-14 in Budapest on September 21. (USA methodology is on total, not just "soluble" metal.)

Nonvolatile determination for environmental regulation (called VOC in USA EPA) is in TC 35/SC-10's Draft Proposal DP 3251.2, and therefore relates to Sub. D01.21.13.

Isocyanate monomer content in "binders" by a gas chromatographic method is proposed in a seven-page draft in TC 35/SC-10 and can be of interest to Sub. D01.21 and D01.33.

D-1 seeks a member willing to take over from J.C. Weaver, from about 1965, the big burden of coordination of D-1 with TC 35 either officially through ANSI or on informal specifics at task group levels, and to bring up to date the Index of ISO/TC 35 Standards in the back of the ASTM Standards Volumes 06.01, 06.02, and 06.03.

SUBCOMMITTEE D01.08 ENVIRONMENTAL CONCERNS

J.J. Brezinski, Chairman

Plans were reviewed for a special semiannual report from the ASTM Coordinating Committee on Environmental Activities (CCEA) that would highlight new or early stage standards programs in ASTM Society that relate to environmental concerns. A Status Report on the past CCEA programs was circulated.

Publication on VOC Determination—It was reported that the preparation of this special document is on schedule in Sub. D01.21.14. The chairman will prepare a revised draft of the document (minus the Appendix Sections) and circulate for comment to interested persons, coauthors, and to representatives of EPA.

The objective of the program is to have the document available for use in the ASTM VOC Training Seminars scheduled to begin late October this year. Publication as a polished ASTM document will be considered at a later date.

Transfer Efficiency Measurements—The Provisional Subcommittee on Transfer Efficiency has approved the Scope and Limitations Section of two developing documents on the subject. Separate task groups were organized to develop draft ASTM practices for the determination of transfer efficiency under laboratory conditions and under production conditions.

The plan is to have the two practices circulated this Fall in a joint Sub. D01.08/D01.55/D-1 letter ballot. The jurisdiction for the two new task groups has been transferred to Sub. D01.55 on Factory-Applied Coatings on Preformed Products. With this action, the Provisional Subcommittee on Transfer Efficiency has been dissolved.

Oven Incineration Credit (for VOC)—Sub. D01.21 has agreed to consider development of an ASTM guide for the determination of oven incineration credit (for VOC). A general procedure developed for use with automotive coatings processes will be used as the basis: this procedure requires the determination of the loss of VOC at various stages of a multi-step coatings process.

Calculation of Formulation Physical Constants of Coatings—Sub. D01.24 on Physical Properties of Liquid Paints and Paint Materials has initiated consideration of the development of an ASTM Standard Practice for *calculating formula density (weight and volume basis), weight and volume solids, average volatiles density, and VOC for coatings as supplied (unreduced) and as applied (reduced for application).*

A draft document prepared earlier by a group of coatings suppliers and automotive coatings appliers will be modified to incorporate suggested changes then it will be circulated in a Sub. D01.24 letter ballot.

The chairman was asked to explore the feasibility of the transfer from Committee E-34 to D-1 of the responsibility for the ASTM Standard E 848 on the Safety and Health Requirements Relating to Occupational Exposure to Water Insoluble Chromates.

SUBCOMMITTEE D01.18 EDITORIAL

S. LeSota, Chairman

D01.18.02, Indexing, S. LeSota, Chairman, reviewed the proof sheets of the revised galley proofs of the latest D-1 index. Although progress has been good, there are still serious flaws that must be corrected. It was disappointing to find broad categories (organic coatings, protective coatings), typographical errors, and inconsistencies still remaining. To prevent future problems, greater care must be taken to choose a proper title and index terms for any new standard, or reapproved standard. It is obvious that there is still a long way to go.

SUBCOMMITTEE D01.94 AWARDS & MEMORIALS

S.B. Levinson, Chairman

The following were recommended to receive the awards listed: ASTM Honorary Membership—H.A. Wray; D-1 Honorary Membership—H.E. Ashton, S. LeSota, P.C. Stievater; Henry A. Gardner Award—K.A. Trimber; Certificates of Appreciation—A.L. Cunningham, L. DiCarlo, W.J. Hart, C.L. Perez, A.N. Scarlatti, S.B. Schroeder, J.H. Smith, and W.B. Van DerLinde.

DIVISION 20 RESEARCH AND GENERAL METHODS

SUBCOMMITTEE D01.20 SAMPLING, STATISTICS, ETC.

H.E. Ashton, Chairman

The chairman advised that the editors of *Standardization News* had rejected the notice on the availability of the software program for use with D 3980, "Practice for Interlaboratory Testing of Paint and Related Materials," because it was "too commercial." It was pointed out that the only reason for mentioning the price was to avoid anyone assuming the program could be obtained without cost. The chairman agreed to see if the announcement could be modified to make it acceptable.

He also read the reply sent to the Society voter who had requested changes at the next revision of D 3980. The letter explained the rationale for two items and asked for clarification on two of the points raised. No reply had been received in the three months prior to the meeting nor to the subcommittee ballot sent because of the voter's early interest in the work of Subcommittee XIV (now 20).

The D 3980 revision, consisting mostly of definitions, received comments from only two voters as the result of the concurrent ballots. The comments were discussed and, where not considered appropriate, the voters will receive an explanation. On the subcommittee ballot, one member voted negatively on one revised definition and selected more than one option for referring to single and multilaboratory parameters. He will be asked to consider withdrawing the negative and to clarify his choice of terms. The preferred "intralaboratory" and "interlaboratory" will be submitted to committee ballot with the other terms.

The chairman stated he would resume work on the Practice for Reporting or Scoring Qualitative Test Results, when D 3980 is ready for submission.

SUBCOMMITTEE D01.21 CHEMICAL ANALYSIS OF PAINTS AND PAINT MATERIALS

H. Fujimoto, Chairman

D01.21.13, Coordination of VOC Standards and Information, H. Fujimoto, Chairman, covered the following topics:

(1) D 2369, "Volatiles in Paints," published in *1987 Book of Standards,* Vol. 06.01, has an error in procedure in Paragraph 7.3. After work done to get this method published using 60 min @ 110°C, Paragraph 7.3 reads, "Heat aluminum foil containing specimen for 20 min @ 110° ± 5° in forced draft oven," instead of "60 min @ 110° ± 5°. All books sent out after the error was discovered will have an "errata" included. Books still to be published will have a correction printed in the method. It has not been decided what to do about books already sold.

(2) Dennis Crumpler of the EPA is interested in the gas pycnometer method for determining volume solids. The present method takes three hours. D01.21 has reactivated TG 26, W. Culhane, Chairman. They will conduct a round-robin using a helium gas pycnometer to determine Volume Non-Volatile.

(3) Henry Smith, New Jersey Dept. of Environmental Protection, tried to find a method for determining total solvent recovery using D 2369. This illustrates one of many regulatory agencies and people who are looking for guidance in this field.

(4) R.J. Connor, NPCA, spoke concerning non-flat architectural coating regulations in California. In San Diego and the Bay Area districts, six-month extensions have been granted to extend 380 g/L limits for VOC instead of requiring 250 g/L. The 9th EPA district has challenged these extensions and has begun contacting paint manufacturers for information about production and solvent content of products. There is some question about whether 250 g/L will be enforced. The prime concern of the paint industry is that this stringent architectural paint VOC regulation might "creep across the country."

(5) According to the U.S. EPA, 1987 is the year of compliance for air quality standards. Sixty-six counties and 19 states can't meet new Federal Air Quality standards at present. These counties have nine months to develop clean-up plans or face penalties which would include fines and construction bans. The estimated cost of clean air is $1.9 billion.

(6) E.A. Praschan, General Motors, distributed an in-house method for determining amount of oven incineration for VOC credit. He proposed that Sub. D01.21 set up a task group to address this problem. Sub. D01.21 will do this and ascertain if it falls within the scope of Sub. D01.21.

(7) The Bay Area Air Quality Management District is addressing the use of D 2369 for printing inks. They feel it is too severe a test method. They are proposing a distillation test procedure in which any high-boiling volatile material (below 0.1 mm Hg vapor pressure) should not be classified as VOC since they are absorbed into the paper. Their initial study shows that whereas D 2369 gives 19.43% VOC for a black ink, their method of analysis shows only 2.3%.

(8) California is controlling VOC in salvage going to landfills. Total allowable VOC waste products in landfill is 1%. Their means of measurement is a distillation procedure. Obtaining a representative sample of the salvage material is a problem.

(9) D01.08 has a "Provisional Subcommittee on Transfer Efficiency" (TE) to evaluate and expedite the publication of two methods on TE, a laboratory method developed by CENTEC, "Practice for the Determination of TE of Spray-Applied Coatings under Laboratory Conditions," and a method designed to determine TE under manufacturing conditions (parts are weighed before and after spraying and baking).

The laboratory method is useful only for comparing efficiencies of different spray equipment and for measuring relative performance of different coatings. The second method (determination made under manufacturing conditions) will initially be used only for automotive products. Draft #1 of both methods will be submitted for concurrent Sub. D01.21/D01.55 balloting prior to the January, 1988 meeting for comments.

(10) Sub. D01.21.80 on Exploratory Analytical Research is going to look at new direct ways to approach determining VOC's. They are soliciting any new ideas.

D01.21.14, VOC New Publication and Workshop, H. Fujimoto, Chairman, reported that it has been demonstrated a number of times that laboratories which are experienced in determining VOC using Method 24 (D 2369, D 4017, and D 1475) obtain significantly more reliable results than many other laboratories which are unfamiliar with the test methods. As a result, it was decided to conduct one or more "Paint VOC Workshops" to educate the inexperienced analysts in how to properly use the test procedures. It will be directed at laboratories from industry and Government Regulatory Agencies, commercial testing laboratories, and others.

H. Fujimoto and R.W. Scott will be the instructors with W.C. Golton as a back-up. They will demonstrate and discuss all methods used for the determination of VOC, except for D 4017 (Karl Fischer determination of water), which will be shown on a video tape (cannot use pyridine in a classroom environment). The first two-day workshop will be held at ASTM Headquarters in Philadelphia on October 27th and 28th, 1987. Tuition cost will be $395. If it is successful, additional workshops will be held at other locations around the country (the second one would probably be held in California). Publicity for the first workshop has been started.

An ASTM document entitled "VOC Determination in Paints, Inks and Related Coatings" is being written to be used as source and reference material by attendees at the workshop. It will have a loose-leaf format and will include a description of the evolution of VOC, regulations, definitions, calculations, VOC data sheets with discussion, sections of test procedures with discussion of application, limitations and precision, EPA Method 24 with discussion, transfer efficiency method with discussion, related VOC methods currently under development or study, a bibliography and an appendix which will include copies of all the test methods, data sheets, instructions and a table of current VOC limits. A number of volunteers are contributing to this publication which is being compiled and edited by J.J. Brezinski.

Upper management of the EPA has acquired copies of the outline of the Paint VOC Workshop and have contacted J.J. Brezinski as to its purpose and goals. They stated they think it will have an effect on VOC implementation. Therefore, they are interested in participating in these workshops in various areas so that government inspectors could attend.

Flyers on "Paint VOC Workshop" are being distributed to pertinent paint manufacturers, state regulatory bodies, private commercial laboratories and all those who might be interested in paint VOC measurements.

Documents by Glidden and Dexter-Midland show the wide variation possible in VOC determinations, especially when % water is involved in the calculation. These documents will be discussed.

D01.21.22, Analysis of Electrocoat Bath Samples, W.B. Van DerLinde, Chairman, reported that samples have been shipped to the participants of the round-robin on the test method to determine solvent concentrations in electrocoat baths and permeates. The results are expected to be available by the end of this June. The statistical evaluation and the draft of the test method should be presented at the January meeting.

L. Pattison has offered to organize the round-robin on the test method to measure ion contamination in electrocoat samples. She has written the test method based on suppression ion chromatography and will expand that to include ion exchange technology.

The task group agreed with the outline of the "practice" for the analysis of electrocoat bath samples. A full draft will be submitted at the next meeting. The draft will cover the electrocoat test methods developed to this date. New test methods will be incorporated as revisions of the "practice." To focus on the completion of the work on the test methods and "practice" in progress; no new test methods for the analysis of electrocoat baths will be considered at this time.

A new TG, Sub. D01.21.24 will be formed on "Ion Chromatography of Electrocoat Bath Samples" with L. Pattison as chairman. W.B. Van DerLinde will prepare the samples for use in Sub. D01.21.24's round-robin. The meeting will be back-to-back with Sub. D01.21.22. This will enable Sub. D01.21.22 to concentrate on the "Practice for Analysis of Electrocoat Baths."

D01.21.23, Thermoplastic Traffic Marking, J.M. O'Brien, Sr., Chairman. H. Fujimoto conducted the meeting in the absence of the chairman. The results of the Sub. D01.21 ballot on "New Standard Methods for Chemical and Gravimetric Analysis of White and Yellow Thermoplastic Traffic Markings Containing Lead Chromate and Titanium Dioxide" received one negative from E. Lewis of the Kansas State Dept. of Transportation. It has not been resolved as yet. Numerous comments were received from Lewis and also from A.M. Snider, Jr. The comments involved a lot of rewriting which must either be incorporated or rejected.

D01.21.26, Revision of D 2697, Volume, Non-Volatile, W. Culhane, Chairman, was brought up-to-date on the subject by H. Fujimoto. D 2697 was approved and published in the *1986 Book of Standards*. Attention of the group will now turn to developing a non-volatile volume method using a helium pycnometer. This method has the potential of being faster and more precise than D 2697. W. Culhane was introduced as the new chairman.

An internal Sherwin-Williams method for use of the helium pycnometer was distributed. Interested parties were asked to comment on the method by the end of July. If no serious objections are received, interlaboratory testing of the method could then begin. Both Hariba and Micrometric have volunteered to lend instruments to those labs who participate in the testing. Six labs indicated an interest. Copies of the method will be sent to both instrument manufacturers to insure that it is suitable for their products.

H. Fujimoto suggested that the sample preparation method be changed to 1 mil dry (sprayed or drawdown) applied to glass or Teflon and baked for 60 min at 110°C. The thinner film and higher baking temperature are needed to completely remove solvent from the coatings. There was no objection to this suggestion. The method will be rewritten to reflect this.

Four types of coatings were mentioned as candidates for the inter-laboratory test: an automotive OEM topcoat coating, a baking alkyd, an exterior architectural latex, and a high P/B coating (either primer or exterior latex). PPG will supply all coatings except for the automotive OEM, which will be received from Inmont. Samples will be sent out with a procedure by mid-August.

D01.21.46, X-Ray Analysis of Pigments, A.M. Snider, Jr., Chairman, discussed the proposed "Standard Test Method for Determination of Titanium Dioxide Content in Paint by X-Ray Fluorescence Spectroscopy," which had received two negatives in a recent Sub. D01.21 ballot.

H.W. Ling had requested revision of Paragraph 3.1 and H.E. Ashton had provided several technical and editorial comments. R.W. Scott had given a comment similar to Ling's. The balloted form of the proposed method with the suggested revisions handwritten on the draft was presented to the group and discussed. A motion was passed to submit the revised draft to Sub. D01.21 ballot.

Also discussed was an earlier request from R.A. Licciardello that ASTM adopt the Cosmetic, Toiletry and Fragrance Association, Inc.'s method J4-1 that deals with the detection of amphibole asbestos in talc. The potential presence of asbestos in talc is of interest to both the cosmetic and paint industries. The suggested method uses first x-ray diffraction and then light microscopy to determine whether asbestiform amphibole is present.

Two unresolved questions must be explored: (1) Is the proposed method the best, or even an acceptable, method for the intended task? and (2) Which task group should deal with a method that is part x-ray and part microscopy, especially when the technical merits of the method are not yet resolved?

A.M. Snider will work with an independent consulting laboratory that routinely analyzes asbestos to evaluate the proposed method and will communicate with C.W. Fuller, Chairman of Sub. D01.31 on Pigments, about involvement of Sub. D01.31, if microscopy is the preferred method.

Anyone who knows of unmet needs for x-ray analysis of pigments, or who knows of existing x-ray methods that should be included in the ASTM method collection, is urged to contact Snider at 412-492-5239. H.D. Swafford suggested reactivation of work on a method of qualitative analysis of pigments by x-ray diffraction.

D01.21.53, Trace Levels of Monomers in Paints, H.D. Swafford, Chairman, reported that a round-robin was conducted last November to evaluate a capillary column version of "Standard Test Method for Determining the Unreacted Monomer Content of Latexes Using Gas Chromatography," which was balloted by D-1 in July, 1986. Precision statements were generated from the round-robin data by PPG using their computer program based on E 180. Although the precision appeared to be a little worse than what was obtained for the packed column method, it was the consensus of the group to incorporate the precision information, listing results for each monomer present, and then submit the method to Sub. D01.21 ballot.

After considerable discussion, the task group members agreed that it should evaluate a liquid chromographic method for the determination of unreacted isocyanate monomers in polyurethanes. It was decided not to pursue the evaluation of a gas chromatographic procedure based on D 3432, "Standard Test Method for Free Toluene Diisocyanates in Urethane Prepolymers and Coating Solutions by Gas Chromatography." PPG and Mobay have submitted LC methods and R.A. Orr has agreed to submit Dow's LC method. The chairman will send copies of all three methods to interested parties, requesting that a choice be made as to which method should be pursued.

A round-robin to evaluate the chosen method will be conducted prior to the January, 1988 D-1 meeting. Monomers to be included in the round-robin are TDI at concentrations of 0.1-2.0%, HDI at concentrations of 0.05-2.0% and IPDI at concentrations of 0.2-4.0%. R. Eritano of Mobay has agreed to supply samples needed for the round-robin. A total of seven labs have agreed to participate.

D01.21.54, Revision of D 4017, Water in Paints and Paint Materials by Karl Fischer Method, J.L. Abbamondi, Chairman. H. Fujimoto chaired the meeting in the absence of the chairman. The wide variation in precision data on water determination gives problems when calculating VOC and leads to the poor precision for VOC of water-reducible paints. The group and other Sub. D01.21 members felt, after reviewing GM's round-robin data, that if the method precision could be tightened up it would help in VOC determinations. The EPA is interested also.

There will be a round-robin conducted shortly so that results will be available at the next meeting. Two water samples and a control will be sent to collaborators. Directions for the round-robin will include the following points: (1) Run the control until the correct result for % water is obtained on it. Once the operator has confidence in his ability, then go on to the two other water samples; (2) Direct that the sample be dispersed using pyridine as the solvent (Crescent Chemical recommends using chloroform; however, some labs are not able to use this chemical). It is important that the sample be dispersed well and that it stirs rapidly; and (3) Sacrifice the first sample. It has been noted that better agreement is obtained between the second and third samples.

If precision obtained from this round-robin is better, the method and precision statement will be revised. Samples were to be sent out in July.

D01.21.56, Revision of D 3960, VOC of Paints and Related Coatings, M.E. Sites, Chairman, reported that a section will be added to this Practice to enable the user to calculate VOC on the basis of applied solids. This calculation was reviewed by the group. An appendix also will be added which will include a precision statement generated from data gathered in a round-robin conducted by General Motors and its paint suppliers. Four labs were involved. There were six samples, four solvent-reducible and two water-reducible automotive topcoats. The operators in all labs were persons experienced in running all the tests involved. Data generated from this round-robin had much better precision than that obtained in round-robins involving labs not familiar with running tests for VOC.

These additions will be balloted concurrently by Sub. D01.21 and D-1.

D01.21.71, Lead in Paint-Rapid Test, H.D. Swafford, Chairman, discussed a negative received from D. Fairley of Calcoast Analytical Labs of Berkeley, CA on the 4/15/87 Sub. D01.21 ballot on "Standard Test Method for Detection of Lead in Paints by Direct Aspiration Atomic Absorption Spectroscopy." The negative was ruled "not persuasive."

Comments made by J.J. Brezinski and A.M. Snider, Jr. were accepted and incorporated into a new draft of the method which will be submitted for inclusion in the next D-1 ballot.

Sub. D01.21.71 will revert to inactive status. Results of the new D-1 ballot will be dealt with at the January, 1988 Sub. D01.21 TG Chairman and wrap-up meetings.

D01.21.80, Exploratory and Analytical Research, W.C. Golton, Chairman, reported the response to an appeal to D-1 members to submit Standards produced two letters:

(1) F.W. Billmeyer, Jr. wrote that modern methods for molecular weight measurement have already been placed in the *Book of Standards* by Committee D-20 and they should be applicable to finishes polymers. The group agreed to request publication of these Standards in the appropriate D-1 volume.

(2) A. Abe proposed publication of a review article covering the application of instrumental techniques to paint materials, emphasizing scope and limitations. The chairman counterproposed the organizing of a symposium on the subject, which could be the basis for an ASTM publication. Six members volunteered to work on the symposium. It was recommended that a new task group be created to organize a symposium on analysis and characterization of paints and paint materials.

The group then turned its attention to needs for new analytical methods.

VOC: There is a need for a simple, fast, and reliable method to replace the current methods for volatile organic content. H.D. Swafford and H. Fujimoto will explore a gas chromatographic approach to the problem and report back at the next meeting.

Chromium: While not added, Cr (VI) and CR (III) can theoretically migrate into electrocoat baths from metal treatment chemicals used up the assembly line. Conventional approaches to determining their ions will not work due to interference in the formulas. L. Pattison will explore ion chromatography as a means to handle this problem and report back at the next meeting.

Cyanide and Sulfide: Like the chromium example, these ions are not added to paint, but there have been some reports of their presence by regulatory agencies due to interferences with the official tests, and it is difficult to prove their absence. L. Pattison also agreed to investigate the application of ion chromatography to this problem.

SUBCOMMITTEE D01.22 HEALTH AND SAFETY

J.J. Brezinski, Chairman

Revision of Method D 3278, "Flash Point of Liquids by Setaflash Closed Cup Apparatus" — The subcommittee letter ballot yielded one negative by J.M. Nielsen which suggested changes in the definition of Flash Point.

The negative was ruled "not persuasive" (6-0) as: (1) The definition had been reviewed by the ASTM Coordinating Committee on Flash Point, and (2) The phrase at the end of the definition for Flash Point adequately addresses the suggested changes. The method is now ready for D-1 ballot.

D-1 Letter Ballots:

Reapproval of Method D 3630, "Practice for Determining Constituents Classified as Hazardous Contained in Protective Coatings" — The long negative and comments by H.E. Ashton were ruled persuasive. A joint review with Sub. D01.21 is planned to consider his suggestions and to identify other necessary changes.

Reapproval of Method D 4206, "Sustained Burning of Liquid Mixtures by the Setaflash Tester (Open Cup)" — The negative vote cast by H.E. Ashton relates to the test equipment and cleaning of same. These questions will be referred to the ASTM Coordinating Committee on Flash Point for resolution of the negative.

Reapproval of Method D 4207, "Sustained Burning of Low Viscosity Mixtures by the Wick Test" — Comments received by Ashton were accepted and integrated into the method, which is now ready for Society ballot.

New Method, "Determination of the Upper Layer Separated from a Viscous Liquid" — Five negatives were received.

A negative by W.D. Hines was ruled "not persuasive" (4-0-2) as the significance and use sections are considered satisfactory to describe the intended use of the method.

A negative by D.I. Dietz advocated the placement of metric units first with English units in parentheses (contrary to established ASTM D-1 practice). The negative was ruled "not persuasive" (5-0-1).

A negative by D.L. Jacobs erroneously assumed that the test graduated cylinder used a rubber stopper; the designated glass cylinder uses a glass stopper. The negative was ruled "not persuasive" (5-0-1).

A negative by W.S. Zimmt was, as stated by the author, editorial in nature. Several of the comments were accepted and incorporated in the method. These were also the subject of comments by Ashton. The negative was ruled "not persuasive" (6-0).

A negative by J.M. Klaskin was withdrawn. He had been informed that his suggestions were adopted prior to the receipt of the negative vote.

Comments by D. MacGregor, L. Garza, and Ashton were reviewed and several were included in the revision of the method.

With these changes, the method is ready for Society ballot.

The chairman will continue efforts to develop liaison with Committee E-27 (E-27.04 on Flammability and Ignitability of Chemicals) and to identify stewards for the various standards under Sub. D01.22, as well as representation from Sub. D01.22 on the ASTM Coordinating Committee on Flash Point.

SUBCOMMITTEE D01.23 PHYSICAL PROPERTIES OF APPLIED PAINT FILMS

M.P. Morse, Chairman

D01.23.10 on Adhesion, H.E. Ashton, Chairman, reported that the recent revision of D 3359, "Measuring Adhesion by Tape Test," has been approved by Society ballot.

A representative of the 3M Company has objected to several aspects of D 3359 and has requested immediate revision or withdrawal of the method. After considerable discussion, the group decided that the method would not be withdrawn at this time since it had just been revised to change the adhesion strength specifications of the tape in accordance with information received from the suggested source of supply. It was agreed that the 3M representative would be informed of the extensive use of this method in the industry, the reasons for standardizing it, and the need for retaining the revised specified tape characteristics until its manufacturer had verified that these tape characteristics should be revised further.

The group will continue to search for alternate tapes that meet the specifications of the method. Also, efforts are being made to develop techniques that will provide improved precision for measurements on thick films.

D01.23.14 on Hardness, Abrasion, and Mar Resistance, D.J. Wilverding, Chairman, is attempting to develop an instrumental method for measuring mar resistance of coating surfaces. A round-robin test has been initiated to determine the suitability of the Balanced Beam Tester, equipped with a rounded stylus, for measuring this property. In addition to performing Balanced Beam Tester measurements on the coated panels of the round-robin test, the collaborators will perform test with pencil, coin, eraser, and an Ericsson mar tester.

D01.23.15 on Slip Resistance, H.A. Ball, Chairman, is conducting a round-robin test to determine the precision of static friction measurements of coating surfaces using an inclined plane procedure (Method A of D 4518). Sets of coated panels differing in perceived slip resistance are being forwarded to the collaborators who will be asked to perform tests on both dry and wet surfaces using sled surfaces of neoprene rubber and polished and mildly sand-blasted steel.

Those collaborators possessing a horizontal pull tester have been urged to use it to perform slip tests on the round-robin panels to determine what type of instrument should be used in the precision evaluation of Method B of D 4518.

D01.23.16 on Water Vapor Permeability, M.P. Morse, Chairman, has a round-robin test underway to establish the precision of extensively revised Method D 1653, "Water Vapor Permeability of Organic Coating Films." Two flat latex-based paints and one semi-gloss alkyd-based paint are being tested by the collaborators using both dry and wet cup methods at 23°C and 50% RH.

It was agreed that the method should be revised to provide permeability comparisons of films at a selected common thickness and to provide suggestions as to materials to use for supporting films over the test cups.

D01.23.18 on Flexibility, M.P. Morse, Chairman, reported that a new method, "Mandrel Bend Test of Attached Organic Coatings," recently submitted to D-1 ballot, and which will be designated as a revision of D 522, combines present methods D 522 and D 1737 that measure coating elongation by conical and cylindrical mandrel, respectively. This revised method provides procedures for obtaining crack resistance values and elongation values for applied coatings.

Suggested editorial changes received in the D-1 ballot have been incorporated into the revised method. It is being submitted to Society ballot.

SUBCOMMITTEE D01.24 ON PHYSICAL PROPERTIES OF LIQUID PAINTS

C.K. Schoff, Chairman

D01.24.19, Viscosity by Efflux Cups, J. Peters, Chairman, made final changes in the revision of D 4212, "Test Method for Viscosity by Dip-Type Viscosity Cups," which will be balloted at the Sub. D01.24 level and reviewed by Sub. D01.56. The interlaboratory testing to gather data for a more realistic Zahn cup precision statement had not been carried out, but it is hoped that it will be done before the next D-1 meeting. Changes were made in the program for the interlaboratory testing after which the group felt ready to go ahead.

D01.24.19b, Viscosity by Ford Cup, discussed D 1200, "Test Method for Viscosity of Paints, Varnishes, and Lacquers by Ford Viscosity Cup." A new revision of the method was presented and discussed in detail. One question was whether the test temperature should be changed to 23°C (73°F) instead of 25°C (77°F). The lower temperature is recommended for paint testing methods and is a more realistic room temperature. The higher temperature is the standard viscosity test temperature and calibrating oil viscosities are given at this temperature. It was pointed out that most Ford (and other) cup viscosity measurements were carried out at whatever temperature the room was at, regardless of what the test method specified.

It was suggested that greater emphasis be given to recommending that the next lower cup be used if the flow time is below 20 sec and that the recommended time intervals for the No. 3 and 4 cups should be 30-100 sec rather than 20-100 sec. Various changes and corrections will be made and then the revision will be submitted for Sub. D01.24 ballot.

D01.24.20, Rotational Viscometers, C.K. Schoff, Chairman, discussed the revised version of D 4287, "Test Method for Viscosity of Paints and Varnishes at a High Rate of Shear by the ICI Cone/Plate Viscometer." On the whole, the group was satisfied with the revision which will be submitted for Sub. D01.24 ballot after a few changes and corrections.

D01.24.26, Electrical Properties of Liquid Paints and Related Materials (Including Solvents), discussed Draft #5 of the proposed method for "Analysis of the Electrical Resistivity of Liquid Paint and Related Materials." The group decided that the method was ready for testing via an interlaboratory test program, the details of which were discussed and decided. Four solvents (n-butyl acetate, PM acetate, MAK, and butyl alcohol) will be tested with Ransburg and/or Byk/Mallinkrodt DC resistivity testers.

D01.24.27, Flocculation, met to discuss techniques for determining degree of pigment flocculation to see whether any of them would make good ASTM methods. Two in-house methods were presented, one using light microscopy (of wet paints), the other using viscosity and settling. Several others were mentioned. The microscopy technique will be put into ASTM guide form for the next D-1 meeting in January, 1988. A method based on electron microscopy of dried films also will be presented. Other flocculation identification methods would be appreciated at this time.

D01.24.30, Calculation of VOC & Volume Solids, E.A. Praschan, Chairman Pro-Tem. The first project of this new group is the development of a "Standard Practice for Calculating Formulation Physical Constants of Coatings." Praschan submitted a draft of the practice that had been developed by a group of automotive industry and coatings people. The document was aimed at the automotive industry, but really should have quite general application. It arose from the need to define the terms and the methods of calculation of VOC and other parameters in order to satisfy EPA and other regulatory agencies as to the validity of such numbers. The draft was discussed and a number of changes suggested. The revised document will be submitted for Sub. D01.24 ballot.

D01.24.31, Viscosity by ISO Cup, H.A. Wray, Chairman, discussed the rationale for proposing an ISO cup method. The cup is not widely used in the U.S., but is the viscosity method specified in international shipping regulations and is one of several criteria used to permit shippers of paint and other viscous materials to transport products as less hazardous materials than would be indicated by flash point alone. It is anticipated that various countries, including the U.S., will incorporate such criteria in their regulations in the future. Thus, it would be to the advantage of the U.S. firms to have an ASTM standard available at that time. Furthermore, it would be to the advantage of the U.S. multinational firms to have an ASTM standard to determine viscosity by ISO flow cup in their worldwide operations.

The rationale was discussed and accepted. It was suggested that a news release be sent to *Standardization News* outlining the need for the method and requesting help in the task group. A proposed method based on ISO 2431 will be submitted for Sub. D01.24 ballot.

SUBCOMMITTEE D01.26 OPTICAL PROPERTIES

C.J. Sherman, Chairman

D01.26.02, Color Measurement, C.S. McCamy, Chairman, met at the National Bureau of Standards on May 26, 1987.

A proposed revision of ASTM D 1535, "Standard Method of Specification of Color by the Munsell System," was presented and discussed. It was thought that the description of the method of interpolating between the chips of the Munsell Book of Color might be improved by reference to the description of this process by Billmeyer and Kelley. Then the standard will be forwarded to the Sub. D01.26 chairman for submission for concurrent Sub. D01.26/D-1 ballot.

W.N. Hale, Jr. reported no progress on the revision of D 3134, "Practice for Selecting and Defining Color and Gloss Tolerances of Opaque Materials and for Evaluating Conformance," but retains the responsibility for its preparation.

A proposed revision of D 1729, "Visual Evaluation of Color Difference of Opaque Materials," which had been completed by Chairman McCamy, was discussed. It is being forwarded for concurrent Sub. D01.26/D-1 ballot.

D01.26.06, Hiding Power, L. Schaeffer, Chairman, met in Dearborn, MI, on June 16, 1987. The main discussion was of a newly proposed version of D 2805, "Test Method for Hiding Power of Paint," with a simplified worksheet and calculation procedure. Evidence was presented indicating that D 2805 is generally not well understood and, for that reason, much neglected, despite its status as the sole ASTM instrumental hiding power method.

It was agreed that the proposed revision would be submitted for Sub. D01.26 ballot. In addition, further modifications were discussed to make the method more applicable for industrial finishes and powder coatings. All of these variations could be incorporated into a single omnibus D 2805 standard.

A survey was proposed regarding hiding power methods now being used in the paint industry, for other possible improvements over the present D 2805. Information in this regard, mailed to the Sub. D01.26 chairman at ASTM Headquarters will be greatly appreciated.

D01.26.11, Gloss and Goniophotometry, M.P. Morse, Chairman, met in Dearborn, MI, on June 16, 1987. The chairman briefly reviewed the present objectives of the task group which are: (1) to determine the extent to which the gloss scales produced by D 523, "Standard Test Method for Specular Gloss," are non-linear with respect to visual gloss rating scales and (2) to determine what actions can be taken to correct any nonlinearity.

The visual gloss ratings and instrumental gloss values reported for several series of painted panels by Francis O'Donnell in his Ph.D. thesis at RPI are being analyzed to determine the extent of the nonlinearity of their relationships.

As a result of obtaining more detailed information as to the viewing conditions used in the visual ratings of these sets of panels, the chairman has revised some of the previously presented graphical plots of visual gloss ratings vs D 523 60° instrumental gloss readings. For a set of 20 gray panels, when only the visual ratings made at 60° are plotted against the instrumental readings, the relationship becomes approximately linear and, when the experimental errors of the visual ratings and instrumental gloss values are considered, it is concluded that the 60° gloss scale is sufficiently linear for all practical purposes.

The chairman presented some new graphical plots of visual gloss ratings vs D 523 60° gloss values obtained by O'Donnell for a set of five black, five gray, and five white panels, where reflected images of screen mesh, a bare filament, and a diffuse source were used to obtain the visual ratings. These plots indicate that the relationships between visual gloss ratings and D 523 60° gloss values are approximately linear within each of the three colors. However, the slopes of the lines drawn through the plotted points differ somewhat for the different reflected images and the lines are separated according to color. Thus, there is a need to insert a statement in D 523 that cautions users of the method not to inter-compare gloss values obtained for coated panels differing significantly in their brightness level.

Some graphical plots of the visual ratings by individual observers vs D 523 60° gloss values were presented. It is evident that the observers differed in their sensitivity in distinguishing visual gloss differences and it is evident that they produced different relationships between visual gloss ratings and the instrumental gloss values.

The chairman proposes to prepare a report covering these latest evaluations of O'Donnell's reported results.

D01.26.23, Retroreflectance of Horizontal Coatings, N.L. Johnson, Chairman, met on May 27, 1987 at the National Bureau of Standards. The first item of business was a further review of the revision of D 4061. Additions of suggested specimen size, description of instrument field of review and the additional terms co-viewing and co-entrance angle were agreed to. Based on this and the January 1987 review, a third proposed revision is now ready to be sent to simultaneous Sub D-1/D01.26 ballot.

The second item was the discussion of a proposed new round-robin to update the precision and bias statement in D 4061. It was agreed that the measurement angles of the current portable instruments would be included as well as those in current specifications.

The final item of business was the discussion of the proposed measurement practice for portable retroreflectance instruments used to test road markings. Items to be included are procedures for use on the road, calibration practices, reference to manufacturers operating procedures, optical geometry description, performance characteristics, significance and use relative to visual observation.

D01.26.24, Tinting Strength, C.J. Sherman, Chairman, met in Dearborn, MI. The latest ballot on D 332, "Tinting Strength of White Pigments" with a change in title to "Relative Tinting Strength of White Pigments by Visual Observation" received one negative vote and no comments. The negative was a minor editorial comment and was included. The method is now ready for Society ballot.

D 2745, "Instrumental Tinting Strength of White Pigments" is being revised. The K/S tables, that were originally included to facilitate the calculation of tinting strength, will be left out because the calculation can be done readily with a calculator. The revised method will be submitted for concurrent Sub. D01.26/D-1 ballot.

SUBCOMMITTEE D01.27 ACCELERATED TESTS FOR PROTECTIVE COATINGS

B.L. Williamson, Chairman

D01.27.02, Water Tests, D.M. Grossman, Chairman, discussed ballot results on four water tests: D 870 (Immersion), D 1735 (Water Fog), D 2247 (100% RH), and D 4585 (Controlled Condensation). There was one negative from H.E. Ashton on each of these. These negatives were ruled "not related" to the ballot, since the ballot was limited to adding a procedure for a second post-test evaluation after the samples dry out. The issues in the negative had been considered at previous meetings, but apparently a notification letter was never sent to Ashton. The letter will be sent with the chairman's apologies.

D01.27.04, Light and Water Exposure Apparatus, L.E. Thieben, Chairman, discussed a negative ballot from Ashton on D 3361 (Unfiltered Carbon Arc). A change in scope to "covers panel preparation and evaluation" was voted "not persuasive." All other changes suggested were deemed editorial changes, most of which were accepted.

A revised draft of D 822 (Carbon Arc Weathering Apparatus) was discussed. This revision will include other cycles that are in use. To include as many cycles as possible, a survey of Sub. D01.27 membership will be made.

A task force involved with writing a new standard practice for Xenon accelerated weathering has agreed to survey the Sub. D01.27 membership to determine where the Xenon is being used; if Xenon is used for coatings, what model and conditions are being employed.

D01.27.09, Corrosion, F. Lutze, Chairman, discussed results of the recent round-robin test. General conclusions were that modifications to the General Motors test did not have a significant effect on the results. Test results on painted steel showed agreement between the four labs; however, test results on painted galvanized showed wide variation among the four labs. It was emphasized that laboratory test results on painted galvanized did not agree with outdoor exposure (and field vehicles). Results of an American Iron and Steel task force meeting were presented. These, together with SAE work, indicates that the current indoor cyclic corrosion tests are not appropriate for evaluating the performance of painted galvanized and that new tests were needed. Four tests to be evaluated by AISI were reviewed. The chairman will work with SAE and AISI to minimize duplication and maximize use of resources.

A report of the direction of SAE and AISI and response to a joint project will be presented in January 1988.

D01.27.10, Accelerated Outdoor Weathering, M.P. Morse, Chairman, reviewed the present objective of the task group, determining the applicability and precision of the accelerated weathering methods in D 4141, "Accelerated Outdoor Exposure Tests."

The chairman also reviewed the results obtained from Florida 5° South black box exposure of a series of 12 automotive coatings. These results showed that after 12 months' exposure, the agreements in the rankings of the coatings of both gloss loss and color change by the exposures at four Florida locations were only fair. After 18 months' exposure, the agreement in the rankings of the coatings by four locations were fairly good, while after 24 months' exposure, the agreement in rankings was excellent. Thus it is concluded that, to determine the "true" gloss loss and color change performance of coatings, they should be exposed for 24 months on the 5° South black box in Florida.

J. Robbins III reported on his latest efforts to establish the correlation and precision of results obtained by exposures of the 12 coatings on EMMA-NTW (Night Time Wetting). The results he has obtained on his latest version of EMMA-NTW are in poor agreement with the Florida rankings of these coatings.

It was agreed that the evaluation of EMMA-NTW cycle conditions should be continued. A set of backed panels will be exposed on the "original" EMMA-NTW cycle during the winter months and a set of unbacked panels will be exposed on this cycle during the summer months. There is a possibility that the combination of these two exposure conditions can produce the desired acceleration of gloss loss and color change.

Since there were some small formulation changes made in the 12 coatings in producing additional sets of panels for testing, it was agreed that two sets of the latest coated panels will be exposed on 5° South black box racks at South Florida Test Service and at Sub-Tropical Testing. The panels are to be rated for gloss loss and color change after 12 months and after 24 months exposure periods. All ratings are to be performed by South Florida Testing Service.

D01.27.12, Detergent Testing. B.L. Williamson, Chairman, discussed negatives and comments from the 86-4 ballot. A negative by G. Stearns was found to be persuasive since a safety caveat should be required for the procedure. A negative by H.E. Ashton was found to be "not persuasive" since lack of information from a study being done should not be grounds for a negative vote. Ashton's comments were discussed and several will be employed in the document.

A negative by D. Fairley was found to be "not persuasive" since the title was not required to be changed and the fact that various documents were indeed listed which allowed a person to perform various tests and measurements when using this method. Comments from H.A. Wray were discussed and were found to be useful for this document. Once the editorial changes are made, the document will be reballoted.

Comments from K. Tator were discussed. These were in regard to the required panel size. It was decided that the size should be left as is and any variation from this should be between the buyer and seller.

D01.27.16, Chalking, J.S. Robbins, Jr., Chairman, reviewed substantive changes suggested in the re-drafting of D 4214, "Evaluating Degree of Chalking." Reasons for suggested changes are: (1) The Kronos-Titan Photographic Reference Standard is no longer available from ASTM as an adjunct; (2) Suggested changes in Methods A, B, and C in the use of "finger tip" instead of finger; (3) The development of a preparation section and additional procedure text with illustrations covering preparation and worksheet form for computations in Method D; and (4) To comply with editorial comments in the previous draft received from H.E. Ashton.

Several new suggestions will be incorporated into a new draft of the Standard for Sub. D01.27 ballot.

One comment was received regarding the use of the Helmet 100 Instrument. The testing of the Erickson & Helmet devices prior to the last meeting showed those devices did not detect initial chalking, and, therefore, were not considered in the redraft of this standard. However, the group will continue to pursue and investigate any new chalking methods developed.

D01.27.17, Evaluation of Weathering Effects, A.S. Allen, Chairman, reported that D 772, "Evaluating Degree of Flaking (Scaling) of Exterior Paints," is due for revision. The chairman read some differences between ASTM and ISO Standards. He asked for any terminology or report forms labs may use and pointed out some areas that need work. The chairman will also get ISO and TNO illustrations. When the data comes in a revision will be drafted based on input from several sources.

D01.27.29, Substrates, B.L. Williamson, Chairman, discussed a negative by D. Jacobs on D 609 (Preparation of Steel Test Panels), and on D 3891 (Preparation of Glass Panels). Safety caveats and solvent substitutions will be added. Comments from J. Reid regarding solvent replacement will be incorporated into D 3891. The group decided that D 609 and D 2201 (Galvanized Test Panels) should be withheld from the ballot until they are completely upgraded. A negative from a Mr. Ribar on D 1734 (Concrete and Masonry Panels) was discussed. This standard will be withheld and Ribar and additional people will be asked to update it. H.E. Ashton had comments on D 3891 which were unavailable. These will be discussed with him.

SUBCOMMITTEE D01.28 BIODETERIORATION

M.C. McLaurin, Chairman

D01.28.01, Package Stability, W.B. Woods, Chairman, reported that test coatings and cultures will be distributed for the evaluation of a serial dilution procedure for determining the effectiveness of in-can preservatives. There will be five participants in this study.

D01.28.02, Rapid Method for Detection of Enzymes, L.A. Burmeister, Chairman, reviewed Draft 1 (October 13, 1986) of the method and a few editorial changes were made concerning target solution viscosities and mixing conditions.

A new enzyme source was proposed for use in round-robin testing (Sigma Chemical Co., Cellulase No. C-7377). A final round-robin (using the enzyme system) will be sent out to seven cooperators to test a higher viscosity CMC 7H3S (~2800 cps) as the substrate. Previous round-robins have used material with a viscosity around 1600 cps. This round-robin should be completed and a precision statement generated prior to the January meeting.

D01.28.04, Resistance of Paint Films to Attack by Algae, W.B. Woods, Chairman, reported that unpreserved acrylic paint and cultures of *Oscillatoria* will be supplied to the five participants in a collaborative study. The initial objective is to obtain reproducible failure of the test coating on a wood substrate.

D01.28 — Stewards were assigned to each of the Sub. D01.28 Standards. A letter from A. Leathers was discussed. It will be forwarded to the steward of the test method involved for comment and reply.

DIVISION 30 PAINT MATERIALS

SUBCOMMITTEE D01.31 PIGMENT SPECIFICATIONS

C.W. Fuller, Chairman

A.M. Snider, Jr. of PPG Industries, Inc. asked if ASTM had a method for minor quantities of asbestos or tremolite using x-ray diffraction and/or optical microscopic methods. This is in reference to a cosmetic (CTF method) standard. He, a member of Sub. D01.21, was asked by a Cyprus Mines Corp. person. He was informed that D 605 and D 717 did not cover this. However, it is a good request, which will be followed up.

J.C. Weaver requested that an individual be assigned as steward to each of the Sub. D01.31 Standards to make review both more personal and easy to follow. This can be accomplished using our task group numbers. The Standards for 1987 review are on the #3 ballot.

Certain analytical Standards for Sub. D01.31 are handled by Sub. D01.21. These have been requested to be transferred to Sub. D01.31. The suggestion was accepted by Sub. D01.21 and the transfer of jurisdiction was approved at this meeting. Included are D 185, D 280, D 1208, and D 2448.

SUBCOMMITTEE D01.33 POLYMERS & RESINS

R.A. Orr, Chairman

D01.33.01, Varnishes, H.A. Ball, Chairman, had received a negative on D 1643, "Gas Checking and Draft Test of Varnish Films," from H.E. Ashton which pointed out that this method was recommended for withdrawal in June 1982, but was never balloted.

It was balloted finally on Ballot D0101 (87-1) and received negatives from J.C.

Weaver and E. Clegg, both of which were accepted. It was then advanced to Society ballot as revised to include the comments by Ashton.

D01.33.12, Urethanes, R.A. Orr, Chairman, met jointly with Sub. D01.21.53, Trace Levels of Monomers in Paints, to consider both GC and LC methods for trace monomers in urethanes. D 3432, "Free TDI in Urethane Prepolymers and Coating Solutions by GC," per negatives by T. Rends and H.E. Ashton on the 86-3 ballot. This method covers only one monomer as original round-robin data was poor when other monomers were analyzed. This was due mainly to sample problems rather than analytical technique. It was decided to maintain this GC method and submit it to Society ballot. The joint task group then addressed LC methods for TDI, MDI, and HDI. Three methods provided by task group members will be circulated for review and comments, then a round-robin utilizing a unified method and various levels of the three monomers will be performed. The minutes of Sub. D01.21.53 show more details on this method.

D01.33.14, Alkyds, R.A. Orr, Chairman, discussed four negatives from the 86-3 ballot concerning use of benzene in methods D 563, D 1306, D 1397, and D 2689. The negative voter is developing data showing toluene as a suitable solvent. However, this data was not to be ready until early July. He withdrew his negatives with the editorial addition of benzene warnings and that other solvents are appropriate and precision data will be added to these methods when available. These methods will be revised and sent to Society ballot.

R.E. Lowrance discussed the need for a method of analysis on silicone content in alkyds. He volunteered to submit a proposed method and to chair a task group on the subject.

J.C. Weaver discussed the need for an alkyd publication to include standards, an update of STP 500's alkyd section, and a manuscript by Herman Lanson. The group voted in favor of this. Chairman Orr will coordinate.

D01.33.18, Rosins, C.M. Winchester, Chairman, received negative ballots by D.F. Zinkel on both D 1469 and D 1542 on the 87-1 ballot on the premise that Sub. D01.34 was studying them. His negative was withdrawn when it was explained that this was not the case, and that the methods were under Sub. D01.33 jurisdiction.

D01.33.20, Shellac, J.W. Martin, Chairman. In the absence of the chairman, R.A. Orr reported that action on negatives on the 87-1 ballot by H.E. Ashton and comments by J.R.S. Reid on four methods on the 87-1 ballot was postponed until the January meeting.

D01.33.23, Epoxy Resins, R.A. Orr, Chairman, reported that methods D 1652, "Epoxy Content of Epoxy Resins," and D 1726, "Hydrolyzable Chloride Content. . ." have been submitted for Society ballot with no results reported to date. New versions of these methods were also presented for discussion. For D 1652, the new method as a Method A, the HBR titration, and as Method B, the perchloric acid titration, which generate HBR *in situ* via addition of TEAB. For D 1726, Method A is the HCL titration of KOH saponified resin, while Method B is a silver nitrate titration after saponification. Five labs have volunteered for the round-robin.

D 1847 on Total Chloride received one comment on the D-1 87-1 ballot. This will be incorporated into the method, which will then be submitted to Society ballot.

J. Peters reported progress on D 1544, "Gardner Color Scale." Data from glass producers' method improvement would be economically unfeasible for improvements of Table 1 luminous transmittance and transmittance tolerance data. Peters will write negative voter, W.N. Hale, and resolve this prior to January. At that time the negative vote of H.E. Ashton will also be resolved.

Two negatives were received on the 87-2 ballot on D 1545, "Viscosity of Transparent Liquids by Bubble Time Test." One negative was accepted as editorial. J. Peters' negative requested deletion of all of section 3.3 and a statement referring to a number tube comparison since those tubes are no longer manufactured. It was agreed that this method would be rewritten and reballoted at subcommittee level prior to the January meeting.

D01.33.24, Nitrogen Resins, J.H. Smith, Chairman, reported that editorial comments were received from H.E. Ashton on balloting of both D 1013, "Total Nitrogen in Resins and Plastics," and D 1198, "Solvent Tolerance of Amine Resins." They will be incorporated into the methods, which will then be submitted for Society ballot.

The method for determining free formaldehyde in nitrogen resins has been put into a usable form. It will be sent to potential round-robin cooperators for their comments and to solicit their participation.

D01.33.25, PVC and PVB Resins, J.J. Brezinski, Chairman. In the absence of the chairman, R.A. Orr reported that there were no negatives on the balloting of D 1396 for reapproval. He also reported that D 3680, "Test for VC2 Monomer Content of PVC Resins," recently transferred from Committee D-20 to D-1, needs reviewing.

D01.33.26, Water-Reducible Polymers, J.J. Brezinski, Chairman, will review two subcommittee negatives and comments from five voters on the balloting of the New Test Method for "Nonvolatile Content of Latices" and resolve them by the next meeting.

D. Waldrep volunteered to submit by letter prior to the January meeting a new version of the determination of filtered solids content of a latex for group review.

D01.33.27, Phenolic Resins. Five members present discussed possible task group chairman candidates and identified manufacturers of the products. Chairman Orr will contact these individuals and companies for support of this task group. The seven methods and nine negatives were not discussed.

SUBCOMMITTEE D01.34
NAVAL STORES

C.M. Winchester, Chairman

D01.34.01, Resin Solutions, C.M. Winchester, Chairman, reported that the results of the round-robin on the proposed method, "Preparation of Ink Resin Solutions by Waring Blender" showed poor repeatability and reproducibility. A second round-robin was organized based upon a blender method to be supplied by J. Daust, FRP Corp., in which the procedure is based upon a standard time rather than a standard peak temperature. The cooperators will also prepare samples using a hot plate method to be supplied by W. Martens, Superior Varnish & Drier. To minimize the lab time required for this round-robin, duplicate samples will be run on only one day. E. Hoffman, of Hercules, and J. Gharia, of S.C. Johnson, will investigate the effect of time and temperature on the hot plate method for preparing resin solutions containing ARLO. The suppliers of the solvents and resins of the first round-robin will be asked for materials for the second round-robin.

D01.34.02, Dilutability, J.W. Daugherty, Chairman, reported that results of a second round-robin, in which cooperators used standard solvent samples, showed better reproducibility and repeatability than did the first round-robin, in which cooperators used their own solvent samples. The lowest variance was obtained with samples cut in ARLO and titrated with hexane. The highest variance was obtained with samples cut in Magiesol 47 and titrated with that solvent. The dilutability procedure will be documented and circulated to the subcommittee for comments prior to the next meeting.

D01.34.03, Gelability, A.N. Scarlatti, Chairman, organized a round-robin to determine whether different labs can produce similar gelled vehicles from a standard resin solution. Chairman Scarlatti will provide the cooperators with the test procedure and with a resin cut in ARLO. He also agreed to arrange for samples of the two gellants, AOA and AIM, to be used in the procedure. The gelled vehicles will be prepared by the cooperators, who will also run rheological measurements, and send both to Scarlatti, for standard measurements, and to G. Bien for tack stability tests.

D01.34.04, Cellulose Compatibility, L. Drucker, Chairman, reported that a method for determining the degree of compatibility between EHEC and metal resinates may be of interest to the liquid ink industry. Neither Sub. D01.34 or D01.56 currently has sufficient participation from this industry segment to pursue development of a procedure. J. Gharia, of S.C. Johnson, will survey the industry to solicit interest both in the development of a compatibility test and in participation in ASTM.

D01.34.05, Naval Stores, H. Ault, Chairman, reported that C.M. Winchester, Sub. D01.34 Chairman, attended the Spring meeting of the Pulp Chemicals Association, where a meeting of this group was held. H. Ault agreed to chair this group, which will continue to meet with the Pulp Chemicals Association, and to be the steward of the 22 methods for naval stores already on the books.

D01.34.06, Vehicle Tack, W.R. Martens, Chairman, met jointly with Sub. D01.56.06. J. Cichon, Acme Printing Inks, has compiled statistical results from the round-robin. It was agreed to recalculate the statistics for the electronic and mechanical inkometers separately, and to limit the precision statement to the 60 sec tack readings. Stability will be handled as a separate study. Chairman Martens will develop the precision statement. T. Sayers, Hallmark Cards, is chairing Sub. D01.56.06 and will include the vehicle tack writeup as part of the revision of D 4361, "Test Method for Apparent Tack of Printing Inks by the Inkometer."

SUBCOMMITTEE D01.35 SOLVENTS, PLASTICIZERS AND CHEMICAL INTERMEDIATES

L.R. Thurman, Chairman

D01.35.10, Solvents-Hydrocarbons and Ketones, R.L. Hinrichs and S.A. Yuhas, Jr., Co-Chairmen, reported that three identical negative votes cast by J.M. Nielson on separate items (D 1363, D 1720, D 2804) in which he objected to the removal of the precautionary statements pertaining to solvent hazards were found to be "not persuasive," *provided that* the following statement could be added in the Scope, "Consult suppliers' Material Safety Data Sheets for specific hazard information."

After discussion, it was decided to revise D 740, "Specification for Methyl Ethyl Ketone," to include D 3729, "Specification for Methyl Ethyl Ketone (99.5 grade) for Urethane Coatings." D 740 will include a regular and urethane grade with the appropriate statements for urethane grade as they appear in D 3729. D 3729 will be withdrawn upon approval of the new D 740.

D01.35.20, Reactive Monomers — Vinyl Monomers and Aldehydes, T.H. Golson, Chairman, discussed various submitted GC methods to determine dimer in Acrylic Acid. A draft method was distributed for further review and revision. At the next meeting, the objective will be to prepare a method for ballot. This could be a three-part method: (a) saponification, (b) dimer only, (c) detailed inpurities. The precision and bias for (b) and (c) can be determined during balloting, if practical.

D01.35.30, Chemical Intermediates, J.R. Morrison, Chairman, discussed the distillation specification for 2-ethylhexanol. It was decided to specify an initial boiling point of 182° minimum, a dry point of 186° maximum and a distillation range of 2°C maximum. The proposed 2-ethylhexanol specification and the 2-ethylhexanol GC method will be submitted to Sub. D01.35 letter ballot concurrently. The GC method will be submitted to a round-robin evaluation.

D01.35.40, Plasticizers and Ester Solvents, R.L. Smith, Chairman, received a negative vote by J.M. Nielson on D 3131, "Standard Specification for Isopropyl Acetate (99% Grade)," for the same reason as in Sub. D01.35.10 previously mentioned and was voted "not persuasive" for the same reason given there.

The manufacturers of tricresyl phosphate will be contacted to determine if the test methods and specification are relevant and should be maintained.

D01.35.50, Liaison, L.P. Field, Chairman, reported that the Government Procurement Document had passed the Sub. E-46.10 ballot and was to go to the main committee ballot in August. The Quality Provisions Guide did not pass subcommittee ballot. Discussions at this meeting resulted in the opinion that Sub. D01.35 members could not support the Government Procurement Requirements Guide, if it comes to Society ballot.

The Task Group chairman has applied for membership to E-46 and will apply to D-2 in order to obtain information about revision of Standards from minutes and ballots.

Old Business — A discussion was held on the General Safety Statements used in Sub. D01.35 Standards. It was determined that the following statement will be sufficient for both test methods and specifications:

"Refer to Suppliers' current Material Safety Data Sheet for specific hazard information when handling material(s) referenced in this Standard."

New Business — Vice-Chairman T.H. Golson will assist the Task Group chairmen in new Standards development. Presently Standards for the Specification and Test Method of 2-Ethylhexanol and the GC Analysis of the Dimer Content of Acrylic Acid are being developed. A method to determine "Clear and Colorless" properties of materials will be investigated. Development of test methods and specifications for plasticizers used in coatings will be considered.

Sub. D01.35 has many Standards associated with Tricresyl Phosphate; however, no active member is very familiar with these Standards. New member interest in these Standards is being sought.

DIVISION 40 PAINT PRODUCTS APPLIED ON SITE

SUBCOMMITTEE D01.41 PURCHASE OF PAINTS AND RELATED COATINGS

H.A. Ammlung, Chairman

D01.41.02, D 3927, Standard Guide for State and Institutional Purchasing of Paint, W.V. Moseley, Jr., Chairman, reported no negatives or comments had been received on the recent Society ballot for reapproval. H.E. Ashton questioned the disposition of his negatives on the initial and latest concurrent subcommittee and main committee ballots. It was noted that there had been a number of negatives and substantial comments on the initial draft that attempted to incorporate the Weighted Cost of Active Ingredients method. These were found persuasive and it was decided that the best approach would be to delete the Weighted Cost of Active Ingredients sections for future study and to submit D 3927 with minor editorial changes for reapproval. This action was approved at the June 1986 meeting in Louisville. Two comments, but no negatives, were received on this revision prior to the January 1987 meeting in Tampa. It was approved for Society ballot at that time. It was felt that most of his comments had been answered with the deletion of the sections in question, but the chairman agreed to review his files and report at the next meeting.

D01.41.05, Latex Flat Wall Paint Specification, I.V. Bloom, Chairman, reported that the proposed D 4717, "Latex Flat Wall Paint Specification," was on the June 1987 Society ballot. To date, no negatives or comments were received, but balloting will not close until July 22, 1987. Several members reported rumors that a number of flat interior latex paints had failed to meet specification requirements. M.E. McKnight said this was puzzling since her work during development of the requirements would indicate this should not have happened. In addition, some requirements were later modified at the request of industry representatives. Since specifics were lacking, members were asked to submit any data they might have on tests of commercial products vs D 4717. These will be discussed at the next meeting as a basis for future revision.

Old Business — Work on revision of the scope of Sub. D01.41 was deferred at the request of the chairman until the impact of the proposed property/cell concept for paint specifications within D-1 could be determined.

New Business — Suggestions to expand the work on D 4717 to include other interior and exterior latex paints drew little response from the membership.

The chairman then introduced J.J. Brezinski and asked him to discuss his proposal that D-1 adopt the property/cell concept for paint and coatings specifications. He reviewed its past use in Committee D-20 on Plastics and noted that plastic compounds and paints had several common characteristics.

Such a specification lists key properties or performance characteristics on the left margin of the table and cell limits for these properties across the top. The table does not establish any specific performance level per se and, thus, it is not a performance specification. It does provide a user or buyer with a means of specifying the quality level he desires. It also provides broad flexibility both as to classes of products and variations within classes.

For government procurement it would be possible to include specific requirements by code plus packaging within a CID or some similar document. Establishment of such a table for a paint product would require input from people knowledgeable in the technology of the paint class.

Industry members expressed concern about how such a document might be used and its impact on the paint industry. It was reemphasized that incorporation of the property/cell matrix within a specification did not establish any specific level of performance, but would provide a means of identifying practical limits on key properties where desirable. This is lacking in current guide specifications. This could be studied at a future meeting. M.E. McKnight was appointed to head up this task group and all members were asked to provide input.

SUBCOMMITTEE D01.42 ARCHITECTURAL PAINTS

L. Schaeffer, Chairman

D01.42.01, Soil and Stain Removal — Mechanical Test, T.J. Sliva, Chairman, discussed a proposal submitted by the chairman to amend and expand upon D 3450, "Test Method for Washability Properties of Interior Architectural Coatings," to incorporate the use of both stains and soilants in the procedure. After discussion of the proposal, it was decided to run a round-robin using one each of the four basic types of discolorant: water-borne stain, water-borne soilant, solvent-borne stain, and solvent-borne soilant. The test paints will be an interior flat and a semi-gloss. The results of the round-robin will be reported at the January meeting.

D01.42.02, Soil and Stain Removal — Practical Test, C.C. Tatman, Chairman, reviewed comments from three respondents and one negative received on the 87-1 ballot. The comments were editorial in nature and were incorporated into the procedure. The negative was reviewed with the voter and was withdrawn on the basis of appropriate revisions in the method. The edited document will be reballoted.

D01.42.04, Wet Adhesion, W. Vanderslice, Chairman, restated the scope of the group and distributed copies of a proposed test method. In the ensuing discussion, suggestions were made and accepted for important changes in the method relating to test surface, spreading rate, water immersion, and film removal. The chairman is to evaluate the test in a preliminary way and then send out samples and equipment for the first round-robin. Eight cooperators have signed up.

D01.42.05 (Tentative), Chalky Surface Adhesion, is an old task group which bogged down several times and is now on inactive status. However, the subject continues to be of importance and interest. A proposal has been made to revive the group on the basis of a test concept involving artificial chalk surfaces and intercoat adhesion tests. Anyone interested in, or having suggestions on, this subject should contact R.M. Schiller, M.A. Bruder & Sons, 600 Reed Road, P.O. Box 600, Broomall, PA 19008.

D01.42.09, Color Acceptance, L.A. Burmeister, Chairman. The immediate objective of this group is to develop a method for measuring color changes in paints caused by application-related shear stress. The test would relate to both practical application performance and color development. A proposed method (Draft #1, June 1987) was reviewed and several variations were suggested, on which basis it was decided to initiate a round-robin using a variety of application procedures including brushouts, drawdowns, and roller applications, to determine which procedure yields the most meaningful and reproducible results. Color measurements will be made by each cooperator and the test panels then sent to the chairman for remeasurement on a single instrument. The chairman welcomes any ideas and suggestions in connection with this method.

D01.42.13, Brushability (Brush Drag), L.A. Burmeister, Chairman, reviewed the latest draft (Draft #4, Jan. 1987) of the proposed method. Several editorial changes were recommended whereby the scope would be expanded to include gloss as well as flat paints. Also, the ranking system will be revised to more closely reflect that used in the ASTM Standardized Scoring Scheme and the precision statement will likewise be revised to use more typical ASTM terminology. A new draft is to be available for review before the next meeting.

D01.42.16, Practical Hiding Power, J.F. Price, Chairman. In the absence of the group chairman, L. Schaeffer chaired the meeting. Panels were circulated showing texture and design for a proposed paint test chart that would be used as a standard surface for the work of this group. The surface will have a relatively coarse texture, referred to as "tooth," so that a roller will encounter sufficient friction to avoid slippage. The printed design will have six stripes (one black and five shades of gray) on a white background. The size will be about 11 × 17 inches, with provision for larger sizes if that should be considered necessary in the future. The task group felt that the proposed texture and design would be satisfactory. This opinion will be reported back to the group chairman, who will prepare a detailed test procedure for review at the next meeting.

D01.42.18, Block Resistance. Due to the resignation of the chairman, the meeting was chaired by L. Schaeffer. In view of the slow progress of this group, the question was raised for discussion as to whether the property of block resistance was of real significance and interest in connection with architectural coatings. Each of the members present was asked to state his opinion on this question. There was unanimous agreement that it was of importance. Most do run some sort of block resistance test in their own labs, although in all cases the test has an informal and subjective character which will not readily translate into a precise ASTM format and procedure. It was agreed that the development of such an ASTM test was a desirable and important objective.

It was decided to continue the group and to seek a new chairman. The previous chairman, U. Jackson, agreed to cooperate to the maximum that his time will permit.

D01.42,19, Wet-to-Dry Hiding Change, L. Schaeffer, Chairman, submitted data on hiding measurements that were made using a single clearance applicator on charts with multiple shades of gray. He also presented a report and exhibits showing sharply improved results using a special multi-clearance applicator on black and white charts. This special applicator has eight clearances in geometric progression. On application, the clearances corresponding to the hiding end point could be readily observed. Of the three paints used in preliminary tests, one decreased in hiding by 70%, another increased by 50%, and a third showed virtually no change. The group agreed to run a round-robin on the basis of the special applicator. It is expected results will be available for discussion at the next meeting.

D01.42.20, Water Repellency of Wood, V. Scarborough, Chairman, distributed copies of the revised test method. Comments from the Forest Products Laboratory and representatives from the wood industry were discussed and will be incorporated into the method. A round-robin will be initiated which will evaluate the relative influence in this method of end and flat grain surfaces, as well as the effectiveness of different repellency treatments. Results will be reported at the next meeting.

Old Business—H.E. Ashton submitted a precision statement for the new Standard, D 4707 on Roller Splatter Resistance. The subcommittee voted to approve the statement, but, because of its late submission, it cannot be included in the first publication of the Standard. It will be incorporated in due course by the customary ballot procedure, under the stewardship of J.F. Price, chairman of the originating task group.

New Business:

Supervision of Sub. D01.42 Standards—S. LeSota agreed to chair an editorial task group (Sub. D01.42.21) for the purpose of maintaining an orderly and timely review schedule for Sub. D01.42 Standards. Each of these Standards should have a "steward" to make the actual review. A Standards review schedule was distributed showing the full list of Sub. D01.42 Standards and indicating names of those with stewards and those without as yet. Additional volunteer stewards will be needed.

Federal Test Methods Standard 141-C—The question of converting still active methods from FTMS 141-C into ASTM format was discussed. A list of such methods within the scope of Sub. D01.42 was distributed. The ultimate objective would be for each of them to be formally discarded by the government in favor of a specific ASTM Standard, or discarded altogether if determined to be of inadequate value. It would be logical for a volunteer from the government to make a review for that purpose.

SUBCOMMITTEE D01.45 MARINE COATINGS

L.S. Birnbaum, Chairman

D01.45.06, Dynamic Testing:

Group A, Rotating Drum Method, D. Laster, Chairman, discussed three negatives and three sets of comments received on Item 61 of the 87-2 ballot. Some of these recommendations were considered of merit and will be incorporated. However, by vote of the members present, a number of these recommendations were also determined either not persuasive or not related to the item balloted. Time did not permit preparation of reason(s) and explanation(s) for the "not persuasive" action(s) for submission to the Main Committee at this meeting. Mr. Laster will modify Item 61 (showing changes adopted), prepare documentation of action(s) taken on negative votes, and will forward to D-1 ASTM Staff Manager.

Group B, Flowing Water Method, T. Dowd, Chairman, discussed a negative and a set of comments received on Item 60 of the 87-2 ballot. Additional time and information is needed to resolve the negative received from C.D. Stevens. Chairman Dowd was unable to contact Stevens prior to the meeting. Mr. Ellor of Ocean City Research will also be contacted concerning these negatives. If appropriate, a meeting of the group or subcommittee will be convened in the Washington, D.C. area.

D01.45.07, Antifouling Ratings, C. Perez, Chairman. No negatives or comments were received for reapproval of D 3623, "Method of Testing Antifouling Panels in Shallow Submergence," Item 62 of the 87-2 ballot. However, since Item 55 of the 87-1 ballot covered a revision of D 3623, Item 62 of the 87-2 ballot was withdrawn. Action on Item 55 of the 87-1 ballot was deferred.

D01.45.08, Organotin Release Rate, L. Birnbaum, Chairman, received too late input on modifications to Draft #7 for preparation of a revised Draft #8 by the chairman for this meeting.

Copies of the M & T proposed analytical simplification protocol were distributed to the attendees for review with the request that comments be forwarded to the chairman for action.

Attention was invited to D 3980, "Standard Practice for Interlaboratory Testing of Paint and Related Materials," as a reference document for consideration in any future round-robin.

Dr. Janet Anderson, Organotin Special Manager, EPA, advised that the EPA was not in a position to release data on results of the Data Call In at this time, but was hoping that some data could be shared with ASTM within the next few months. She also indicated that, based on information obtained as a result of the Data Call In, EPA had a number of comments covering protocol including holding tank, carbon filter system, staggering of test starting times, size of tank, clarification of calculations, and others. The chairman will follow up on this. Dr. Anderson also advised the EPA was hoping to promulgate some type of regulatory action on organotin AF's by the Fall of 1987. Options cover the range from no changes to a complete ban and could involve such criteria as release rates (daily and cumulative), types of paints, active ingredient composition, and an interview with water quality criteria.

There was a general discussion covering state regulations (both existing and proposed), possible differences between federal and state regulations and enforceability of regulations.

D01.45.09, ASTM Specification Conversion, D. Sowell, Chairman. In the absence of the chairman, L. Birnbaum chaired the meeting. As liaison with F-25.02, L. Birnbaum advised of actions taken on the two specifications recently balloted by Sub. F-25.02 (tank coatings and aluminum heat-resistance paints). Because there were so many changes recommended by the Sub. F-25.02.07 Task Group during their meeting in February, 1987, Mr. O'Sullivan, Todd Pacific Shipyards, will prepare new drafts.

substrate, method of application, and method of reporting test results. Contact will be made with the Brick Institute to clarify questions on the substrate to be used. The chairman will revise his draft and submit it to the five cooperators who have agreed to participate in a round-robin. The results will be distributed at the January 1988 meeting.

D01.47.02, Acid Deposition, V. Scarborough, Chairman. In absence of the chairman, T.J. Sliva chaired the meeting. Two published test protocols for the determination of resistance to acid rain of masonry substrates were submitted for evaluation. A discussion on the test protocols followed. Members were referred to upcoming articles in various periodicals relating to acid rain.

D01.47.03, Active Solids Determination of Silanes, and Siloxanes, T.J. Sliva, Chairman, submitted a test protocol. Discussions with manufacturers of silanes and siloxanes disclosed common ground on the method to be used. The addition of a water:isopropyl alcohol: hydrochloric acid solution to an aliquot of silane/siloxane will be used to effect hydrolysis and condensation reactions. After a one-hour polymerization period, the samples will be oven-dried for various periods. A discussion followed concerning the size of the aliquot, reaction vessel, time in oven, and the necessity for stirring. The test method will be revised and six cooperators have agreed to participate in a round-robin. The results will be reported at the January meeting.

D01.47.04, Surface Preparation, F. Gale, Chairman, presented a review of manufacturers' recommendations for surface preparation. A discussion on the various surface conditions which affect the success of masonry water repellent treatments followed. It was decided that the focus of the task group would be to develop a standard guide for surface preparation for water repellent treatment of stone masonry. It was suggested that existing guides for preparing masonry surfaces could be used as models and referenced in the guide.

D01.47.05, Permeability of Treated Masonry Substrates, N. Weiss, Chairman. F. Gale chaired the meeting in the absence of the chairman. Test methods used in NBS Technical Bulletin 883 and BS 6477 for determination of Water Vapor Transmission on Masonry Surfaces were distributed and discussed. It was decided to evaluate data on treated substrates compared to untreated substrates to determine a percent retention of water vapor permeability. A further comparison with data obtained with ASTM E 96 will be made. A proposed test protocol will be developed and presented at the next meeting.

D01.47.06, Adobe, W. Zimmt, Chairman. In the absence of the chairman, the meeting was cancelled.

D01.47.07, Alkali Resistance of Masonry Treatments, A. Sanders, Chairman, gave a brief outline of the procedure and will submit a formal test protocol at the January meeting.

New Business—P. Semach, of Goldschmidt Chemical, has agreed to begin setting up a guide for the testing of water repellents. A compilation of manufacturers' application rates, drying times, and other relevant data will be started.

A request was made to look into various test methods for determination of freezing and thawing and chemical resistance of masonry water repellents. All suggestions and information should be forwarded to Frances Gale, ProSoCo, Inc., P.O. Box 1578, Kansas City, KS 66117.

Due to the large volume of material under consideration by the subcommittee at this time, a majority of the members present requested that an interim meeting be held before the January meeting. The time and location of this meeting had yet to be decided.

DIVISION 50 PAINTS FOR FACTORY APPLICATION

SUBCOMMITTEE D01.51 POWDER COATINGS

D.J. Wilverding, Chairman

Two samples of powder were distributed for round-robin testing along with procedures for testing for specific gravity by two different methods: (1) by dissolving the powder in an active solvent and (2) by dissolving it in a non-solvent. At present, there are three cooperators. Other cooperators will be sought by mail contact with powder manufacturers and users.

D3451, "Practices for Testing Polymeric Powders and Powder Coatings," was submitted for reapproval with revisions.

A suggestion was made and agreed upon to develop a compilation of all specifications and test methods for powder coatings along with other specific significant information on the subject.

SUBCOMMITTEE D01.52 FACTORY-COATED WOOD BUILDING PRODUCTS

S.B. Schroeder, Chairman

D01.52B, Hardboard, S.B. Schroeder, Chairman, discussed progress being made in the round-robin testing of the proposed methods, "Cobb Ring" for water permeability of primed siding and a Triton X-100 edge wick test for measurement of surfactant resistance. Five of the six cooperating hardboard companies have sent out their sample boards and two of the cooperating labs have completed five/six of the required testing. No major difficulty was encountered by either test lab. It is hoped that all testing can be completed prior to the next meeting.

D01.52.13, Prefinished Siding, K. Kruse, Chairman, discussed a proposal from DSET Laboratories to reevaluate a modified EMMAQUA test for prefinished hardboard. Several members will check their inventories of retains of weathered and unweathered samples which could be provided to DSET. These samples with associated weathering data will be sent to Chairman Kruse.

D01.52—Revisions to a number of methods were discussed and several negatives on reapproval of existing methods were resolved.

D 2336, "Practices for Specifying Properties from Liquid Through Cured State for Coatings Factory-Applied to Wood Products." This was reapproved by both Sub. D01.52 and D-1 on the 87-1 ballot.

D 2366, "Method for Accelerated Testing of Moisture Blister Resistance of Exterior House Paints on Wood." This method was approved for withdrawal on the 87-1 ballot. It has been superseded by D 4582.

D 2691, "Method for Microscopic Measurement of Dry Film Thickness of Coatings on Wood Products." This method was balloted for withdrawal on the 87-1 ballot. It received negatives from D.F. Zinkel, J.C. Weaver, and C.M. Winchester on the grounds that a replacement method is not yet available. This argument was found persuasive. A new task group, Sub. D01.52.15, (R. Matejka, Chairman) has been formed to develop a replacement. It was agreed to ballot the method for reapproval until the new method is completed.

D 3719, "Method for Dirt Collection on Coated Exterior Panels." This method was reapproved on the 87-1 ballot.

D 2921, "Method for Qualitative Tests for the Presence of Water Repellents and Preservatives in Wood Products." This method was also balloted for withdrawal. It received a negative from D.F. Zinkel who suggested that it be retained until a quantitative method for water repellents was available. This method is being developed by Sub. D01.42. The negative was found persuasive. The method will be balloted for interim reapproval.

D 2830, "Method for Durability and Compatibility of Factory-Primed Wood Products with Representative Finish Coats." This method was balloted by Sub. D01.52 for reapproval. It received a lengthy negative from H.E. Ashton which was found generally persuasive. A complex issue regarding tape adhesion testing is involved and caused a long discussion. This method and some alternate means of resolving the tape adhesion issue will be sent out to the full Sub. D01.52 membership for input prior to the next meeting.

D 2793, "Method for Block Resistance of Organic Coatings on Wood Substrates." This method is under study for replacement by a revised D 3003. In the interim, it has been reapproved on the 87-1 ballot.

D 4144, "Method for Estimating Package Stability of Coatings for Ultraviolet Curing." This method was approved for reapproval on the Sub. D01.52 ballot. It will be submitted for D-1 ballot.

SUBCOMMITTEE D01.55 FACTORY-APPLIED COATINGS ON PREFORMED PRODUCTS

J.M. Behrle, Acting Chairman

Acting Chairman Behrle reported that on the recent 87-1 ballot the proposed "Guide for Testing of Industrial Water-Reducible Coatings" received comments from two voters. They were judged editorial and were recommended to be incorporated. This Standard is now ready for Society ballot.

D2571, "Practice for Testing Wood Furniture," and D 3023, "Determination of Resistance of Factory-Applied Coatings on Wood Products to Stains and Reagents," are due for review. Suggested revisions were considered and approved. The changes in D 2571 are editorial. One change in D 3023 is substantive, although minor.

D 333, "Testing of Clear and Pigmented Lacquers," received a lengthy negative in 1986. A number of revisions were discussed and more suggested. It was approved for Sub. D01.55 ballot.

D 2091, "Print Resistance of Lacquers," received a number of comments on a 1986 letter ballot. These have been approved and incorporated. Since none are substantive, a concurrent Sub. D01.55/D-1 letter ballot on the editorial changes should be possible.

The prospects for developing a method for testing wet and dry heat resistance of furniture coatings were discussed. There are methods for such tests developed by BSI. C.M. Winchester will draft a proposed method. The rationale is that D 2091 is usually used to evaluate the print resistance of coatings under conditions of packaging, shipping, and warehousing rather than conditions of service.

E.A. Praschan presented some comments from D.C. Gleason on D 3170, "Chip Resistance of Coatings." They involve the Safety Caveat and Significance and Use Statement. They were approved for revision.

It is anticipated that the Executive Subcommittee will consider placing some of the work on Transfer Efficiency in Sub. D01.08 under Sub. D01.55. (See following report on Provisional Subcommittee on Transfer Efficiency.)

PROVISIONAL SUBCOMMITTEE ON TRANSFER EFFICIENCY

J.J. Brezinski, Chairman

The meeting on June 16, 1987 in Dearborn, MI, was the second meeting of this subcommittee. The first meeting was held on May 13, 1987 in Warren, MI, in conjunction with an NPCA meeting.

Transfer Efficiency Determination-Laboratory Conditions—The scope and limitations section of the planned "Proposed Practice for the Determination of the Relative Transfer Efficiency of Spray-Applied Coatings Under Laboratory Conditions," developed at the May 13th meeting, was approved with several editorial changes.

The test method description prepared by CENTEC Corporation was judged to be a suitable starting point for the development of an ASTM Practice. Suggestions for modifications were exchanged at the meeting.

A task group was formed to restructure the CENTEC Corp. document into ASTM format. The members are: R. Diem (Chairman), D. Hemming, M. Tersillo, and H. Fujimoto.

Transfer Efficiency-Production Conditions—The title, scope, and limitations sections were developed and agreed to. The title is "Practice for the Determination of the Transfer Efficiency Under Production Conditions for Spray Application of Automotive Coatings-Weight Basis."

The documents developed at General Motors Laboratories will be used as a basis for the draft ASTM Practice.

A task group organized to do this consists of E.A. Praschan (Chairman), C.D. Lega, J.M. Reinke, and J.J. Brezinski.

With the formation of the two task groups, it was decided to request dissolution of the Provisional Subcommittee on Transfer Efficiency and to transfer the task group jurisdictions to Sub. D01.55. The new task group designations would be Sub. D01.55.08, Transfer Efficiency-Laboratory Conditions, and Sub. D01.55.09, Transfer Efficiency-Production Conditions.

Drafts of the two ASTM documents are to be prepared and approved by the chairmen of Sub. D01.55 and D01.08 in time for distribution this Fall as a joint Sub. D01.55/D01.08 letter ballot.

SUBCOMMITTEE D01.56 PRINTING INKS

J.M. Fetsko, Chairman

D01.56.01, Fineness of Grind, J. Cichon, Chairman, reported the subcommittee ballot of the revised D 1316, "Fineness of Grind of Printing Inks by the NPIRI Grindometer" elicited one negative. After the suggested revisions are made, the method will be submitted for D01 ballot.

D01.56.02, Lightfastness of Prints, J. Daugherty, Chairman, distributed prints and ΔE values resulting from exposure to sunlight, carbon arc, xenon lamp, and HPUV. It was apparent that the same ΔE endpoint cannot be used for all colors. C. Sherman, Chairman of Sub. D01.26, will investigate the matter.

D01.56.04, Viscosity of Paste Inks, J.M. Fetsko, Chairman, distributed copies of proposed revisions for D 4040, "Viscosity of Printing Inks and Vehicles by the Falling Rod Viscometer." After the precision statement is enlarged to cover parameters of non-Newtonianism, the revised method will be ballotted. Data from fall-time runs will be sent to laboratories having computer programs in order to compare calculated results.

D01.56.06, Ink Tack, Toni Sayers, new Chairman, reported that a joint meeting was held with Sub. D01.34, Naval Stores. W. Martens distributed results of a round-robin on tack and tack stability of vehicles. After the data are re-analyzed by instrument, it was suggested that the scope of D4361, "Apparent Tack of Printing Inks by the Inkometer" be enlarged accordingly.

D01.56.09 Tinting Strength, J.M. Fetsko, Chairman, discussed a round-robin for the proposed new test method, "Relative Color and Strength of Printing Inks by Visual and Instrumental Evaluation." Five sets of finished inks, each consisting of two unknowns and a standard, will be prepared. Fifteen laboratories have agreed to run from two to five sets.

D01.56.10, Water-Pickup of Litho Inks, G. Bien, Chairman, reported that 12 laboratories participated in a round-robin on the single point part of the proposed new method. It was suggested that a design change in the Duke Tester may have contributed to variability in the results. The matter will be explored with the cooperating laboratories.

D01.56.11, Nonvolatile Content of Printing Inks, B. Blom, Chairman, reported that the new method passed the Society ballot and has been assigned the number D4713.

D01.56.12, Viscosity of Liquid Inks, J. Cichon, Chairman, reported that a joint meeting was held with Sub. D01.24.19, "Viscosity by Efflux Cups," C. Scoff, Chairman, discussed plans for a round-robin which will include calibration with two standard oils as well as measurement of sample temperature.

D01.56.14, Setting of Heatset Inks, A. Urdea, Chairman, distributed copies of a proposed procedure for evaluating relative setting of inks by the Silvaco Tester. The subcommittee suggested several changes, after which the instructions will be checked out by a cooperating laboratory.

D01.56.17, Practice for Printing Inks, A. Scarlatti, Chairman, distributed a list of ASTM test methods that are applicable to printing inks. After additions are incorporated, he will prepare the practice as per ASTM protocol.

D01.56.18, Heat Resistance of Prints, B. Blom, Chairman, reported that five laboratories tested two inks, one of which had failed in the IBM 3800. Both inks passed all the lab tests. Blom will discuss the results with UARCO.

D01.56.20, Technical Liaison, B. Blom, Chairman, reported that ANPA will certify the color and strength of newsinks from color coordinates measured on letdowns. In response to requests from a printers association, NAPIM has prepared a booklet, "Test Methods for Printing Inks," which describes the status of various ASTM tests under the jurisdiction of Sub. D01.56.

D01.56.21, Print Abrasion with the CAT, G. Bien, Chairman, reported that only one company sent in its test method. He will contact others known to have the instrument in order to determine whether there is interest in development of a standardized test.

D01.56.22, Drying of Sheet-Fed Inks, G. Bien, Chairman, distributed a list of in-house procedures. Other companies were requested to send him their testing methods.

D01.56.24, Grit Determination, M. Fuchs, Chairman of this new task force, will collect available methods for determination of grit by wash-out.

SUBCOMMITTEE D01.57 ARTISTS' PAINTS

J.T. Luke, Chairman

Sub. D01.57 and its task groups met at the Hyatt Regency Hotel in Atlanta, GA, on May 12-13, 1987, just prior to the National Art Material Association (NAMTA) Convention. This enabled European and Japanese company representatives to participate in the subcommittee work and to attend the convention on the same trip to this country.

Planning Meeting—It was agreed that Sub. D-01.57 will meet with Committee D-1 in January 1988 at Clearwater Beach, FL. The Spring meeting will be held in conjunction with the 1988 NAMTA convention in New York in May 1988.

T. Takigawa of Turner Colour Works, Ltd. agreed to chair a task group to develop a quality standard for artists' gouache paints. Company representatives agreeing to serve on the group include S. Yamauchi and K. Hirota, of Holbein Works Ltd., of Japan; J. Smeur, of Royal Talens, in Holland; P. Hesse, of H. Schmincke & Co., in Germany; S. Lynton and W. Upchurch, of Newton, in England; and A. Graham and J. Branewie, of M. Grumbacher Inc., in the United States. B. Crooks, of Maimeri, in Italy and L. Bresnahan, of LeFranc & Bourgeois, in France earlier expressed an interest in the proposed standards.

A preliminary draft of the standard closely paralleling D 4302, the quality standard for artists' oil and acrylic paints, written by Takigawa, was discussed as to what further requirements would be needed. Six additional items were agreed upon.

It was agreed that to accommodate overseas members, the new task group, Sub. D01.57.11 on Gouache Paints, would not hold a session in January, but would meet in the Spring each year when Sub. D01.57 meets with NAMTA.

D01.57.01, Drawdowns of Artists' Paints, T. Pamer, Chairman, reviewed a draft of a proposed method for preparing specimens of artists' paints for colorimetric measurement (or other occasions where a specimen at complete hiding is needed) and a supporting study submitted by C.C. Tatman and M. Dudley. Their conclusion was that, in spite of difficulties with the slow drying artists' paints, it is possible to make drawdowns at complete hiding using a .004 in. applicator and three cross-hatched coats for transparent acrylic paints and a .0015 in. applicator and two or three cross-hatched coats for oil paints. In an air-dry cabinet, the oil specimen took 48 hours to dry between coats, while the acrylic paint took 24 hours.

Tatman and Dudley had been supplied with only two paints by A. Spizzo—a Hansa Yellow Light acrylic paint and a Green Earth oil paint. The Green Earth may not be as transparent as some other oil paints and may not be as slow a drier as other oil paints, but the study results are encouraging.

D01.57.02, Lightfastness of Pigments, E.T. Vonderbrink, Chairman, distributed the revisions in D 4303 necessary to include coverage of the resin-oil paints. These include: definitions of oil, alkyd, and resin-oil artists' paints; a formula for a mixing white to be used to dilute paints when preparing specimens for lightfastness tests; and the use of D 1640 to control drying time between coats and before instrumental color measurement of specimens.

Last year, there was a question whether the dark storage time during shipment of specimens following lightfastness tests and before measurement may cause the oil in oil, alkyd, and resin-oil paints to yellow sufficiently to affect the readings. It would be expected that yellows may appear to have faded a smaller amount than they actually have, blues may appear grayer and there may be a shift in hue in other colors.

P. Staples and T. Vonderbrink have looked into the problem. It is Staples' recommendation that the subcommittee do nothing about the problem since exposing the samples to light in order to bleach out the yellow might also affect the measurement. The subcommittee has now seen the results of two sets of lightfastness tests where alkyd and resin-oil paints were exposed in South Florida and shipped to Europe for measurement. In both instances, the test results on specific pigments were very similar to those from those pigments in a set of tests where the specimens were measured immediately after being removed from exposure.

D01.57.03, Tinting Strength, T. Pamer, Chairman, considered the editorial comments and replies by F. Billmeyer and J.T. Luke received on the Sub. D01.57 ballot on the "Test Method for Determining the Relative Tint Strength of Chromatic Paints." It was agreed to clarify the description of drying time in 7.5 by referring to D 1640. C.C. Tatman had pointed out a possible confusion due to the wording in 7.6 on how to determine whether a drawdown is at complete hiding. It was agreed to clarify this paragraph using wording suggested by Billmeyer. It was also agreed to drop a modifying clause in 5.8 since it could be construed as implying that if paints from different companies do not have equal tinting strength, the cause is contamination or adulteration of the paint.

D01.57.04, Specification for Artists' Paints, A. Spizzo, Chairman, presented copies of the changes in D 4302 that are necessary to include resin-oil paints in the standard. These embodied the agreements reached in earlier meetings: definitions of oil and resin-oil paints, and these should be identical to the definitions given in D 4303; an additional section listing the information that must be included on the label of resin-oil paints; a description of the resin-oil vehicle including the limitation that the gum or resin can total no more than 10% maximum by weight of the vehicle solids; and reduction of the requirement for a nonvolatile content of 97% for oils to 09% for weight for the resin-oils.

Luke distributed copies of a report from H. Mueller giving the results of lightfastness tests from pigments that H. Schmincke & Co. uses in their resin-oil line. The tests were conducted in accordance with requirements of D 4303. One set of specimens was exposed in South Florida behind glass to 1260 MJ/m^2 of irradiation. In the Xenon arc exposure, the specimens were exposed for 154 hr at 2270 W/m^2 irradiance to reach the same 1260 MJ/m^2.

There were five pigments for which one of the test results was borderline or where the results were sufficiently different from those of earlier tests to raise questions about the form of the pigment used. For the borderline pigments, Schmincke will need to indicate whether they should be assigned to the poorer of the two relevant lightfastness categories, or whether the company wants to run a third test. In the case of PY 1 and PY 3, inquiries need to be made of the supplier. It was agreed to add a column for resin-oil paints in Table 1 of D 4302 and to list all the pigments tested by Mueller, with the exception of the borderline five, which can be added later.

A question has arisen about instances where Cadmium Oranges are supplied by the manufacturer that consist of mixtures of Cadmium Red (PR 108) and Cadmium Yellow (PY 35 and PY 37) since Table 1 of D 4302 specifies that Cadmium Orange must be PO 20. A. Graham agreed to study the color gamut of the mixtures compared with that of PO 20 to see if there is any advantage to using PO 20 instead of mixtures of PR 108 and PY 37. Permanence should be comparable.

D01.57.07, Physical Properties, H. Brown, Chairman, reported on a survey of adhesion between acrylic grounds and oil paint films in contemporary paintings. The Ralph Mayer Center sent questionnaires to approximately 100 conservators of contemporary painting. As of May 1, 1987, the Center had received 33 completed returns. Of these, 14 reported adhesion failures. The Center sent questionnaires to approximately 150 well-known painters in the United States. As of the same date, 38 completed returns were received, three of which reported adhesion failures. National Artists Equity Association (NAEA) printed the questionnaire in their December newsletter, which reaches approximately 5000 members. As of May 1, the Center received 28 completed questionnaires from NAEA members, of which two reported adhesion failures. It is not surprising that conservators report the most failures since it is their job to repair damaged paintings. The Mayer Center will continue to receive questionnaires for the next few months and then they will be analyzed.

D01.57.08, Labeling for Toxicity, J.T. Luke, Chairman. C. Jacobson, Compliance Officer of the U.S. Consumer Product Safety Commission (CPSC), described the Advisory Opinion #309 issued by the CPSC staff. The Opinion states (1) that CPSC under the Federal Hazardous Substances Act (FHSA) has the authority to regulate labeling for chronic health hazards; (2) that FHSA regulations preempt state regulations; and (3) that D 4236 meets the labeling requirements of FHSA. Jacobson pointed out that CPSC does not challenge state regulations. Companies themselves must contest state regulations that differ from CPSC regulations; however, CPSC might join a suit as a friend of the court.

Regarding the issue raised by the U.S. Public Interest Group (USPIRG) that educational materials are exempted from the FHSA regulation banning hazardous products [1500.85 (a)(4)] and therefore, small children are exposed to hazardous materials in school, Jacobson pointed out the section in the FHSA regulations stating that this exemption applies only to materials used by children old enough to read and comprehend warning labels [1500.3 (b)(15)(i)(B)]. Therefore, CPSC can ban hazardous art and craft materials for pre-school, kindergarten, and grade school children.

CPSC already divides consumers by age, i.e., under age three, the main problem is ingestion, under age eight, it is sharp objects, while everyone is subject to inhalation hazards. CPSC will follow up complaints and injuries due to art and craft materials as it does for other consumer products. Jacobson reminded the subcommittee that state departments of education are free to ban more substances for use in schools than are banned by CPSC regulation or state regulations.

The chairman read a statement from P. Gilbert of USPIRG requesting that the subcommittee drop the phrase underlined below in the D 4236 definition of art material or art material products as: "any raw or processed material, or manufactured product, marketed or represented by the producer or repackager as *intended for and* suitable for use by artists and craftspeople." USPIRG considers that this phrase makes it more difficult to prove that a product is an art material. Chairman Luke had canvassed the health departments of five states with laws that reference D 4236 and all responded that the inclusion or exclusion of the phrase would make no difference in their judgments on whether a product is, or is not, an art material. They base their judgments on whether the product is sold frequently in art materials stores and how it is advertised and marketed. C. Jacobson showed the subcommittee a number of places where the phrase is used in sections of FHSA. It was voted to leave the definition unchanged.

It was voted to send the revisions proposed in January out for ballot. These changes expand the standard to cover art and craft materials marketed to people of any age. The word "users" is substituted for the term "art or crafts people" throughout and wording limiting the standard to coverage of adult art materials is dropped. It was also decided to adapt wording from FHSA regulations to clarify the requirements for labeling kits that contain one or more products that present health hazards, and to include this change in the ballot.

D01.57.09, Watercolors, E.T. Vonderbrink, Chairman, presented the current situation regarding the tests for lightfastness of pigments used in watercolors. He passed out a sheet showing the watercolors contributed by different manufacturers covering 73 pigments that are used in watercolors. The chairman had also prepared a definition of watercolor and a list of qualities, in addition to lightfastness, that might be covered in the standard. These include fineness of dispersion, consistency, separation of vehicle, rewetting on a palette, ease of breakdown, inerts, and flow. Artists present emphasized the frequent problem of purchasing pan watercolors that are very difficult to dissolve in water to make a wash. There is a problem in distinguishing between watercolors that are poorly formulated and those that have been stored too long.

D01.57.10, Consumer Evaluation, H. Brown, Chairman, distributed a draft of

D-1 Chairman J.C. Waever advised that, to his knowledge, no DOD funds for paint spec revisions for meeting new VOC requirements had been allocated to date. He also discussed problems being experienced with participation of government technical representatives in ASTM activities because of lack of funds.

D01.45 Meeting—Votes were taken on actions concerning negatives in the dynamic test ballots as recommended by the task groups with results being recorded by Dowd and Laster for subsequent documentation.

Chairman Birnbaum recommended Theodore Dowd for appointment as Sub. D01.45 Vice-Chairman to replace William Briggs.

SUBCOMMITTEE D01.46 INDUSTRIAL PROTECTIVE PAINTING

K.A. Trimber, Chairman

D01.46.02, Surface Preparation, K.A. Trimber, Chairman, reported that Draft #4 of a proposed "Method For Conductimetric Analysis of Water-Soluble Ionic Contamination of Blasting Abrasives" has been prepared based on the results of the Sub. D-01.46 ballot received and discussed during the January 1987 meeting. Because the changes were substantial, the draft will be submitted to another Sub. D01.46 ballot.

In addition, the group is soliciting volunteers to participate in a round-robin to establish the precision of the proposed test method. Conductance tests will be required on six lots of abrasives. The laboratory test procedure and report forms can be obtained from K.A. Trimber or W.C. Johnson, KTA-Tator, Inc., 115 Technology Drive, Pittsburgh, PA 15275 (412-788-1300). It is hoped that the tests can be completed prior to the next meeting.

D01.46,03, Repainting, L.M. Smith, Chairman, reported that Draft #1 of a newly proposed "Standard Practice for Testing Coating Compatibility" was prepared by the chairman. The proposed Standard provides for the application and testing of candidate coating systems to be used for maintenance painting (e.g., test patch applications). The draft will be circulated for subcommittee comment prior to the next meeting.

D01.46.04, Pull-Off Adhesion, A. Cunningham, Chairman, reported that two different round-robins have been conducted previously in an attempt to establish the precision of the Elcometer and Fisher 2000 units addressed in D 4541, "Standard Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers." The results of the round-robins were submitted to H.E. Ashton to determine if enough data was available to establish a precision statement. The data was not received in time for a complete review prior to the meeting, but Ashton indicated that he was uncertain as to whether enough data had been generated to properly establish the precision. The data will be reviewed completely and a decision made in time for the next meeting.

Despite the outcome of the analysis, plans are underway for conducting a new round-robin test. The intent is to compare the Elcometer with other Fisher units, and a newly-developed English unit, HATE. In addition, it is expected that the round-robin will be coordinated with Sub. D01.23 in their investigation of D 3359, "Measuring Adhesion by Tape Test." The intent will be to conduct the various pull-off and knife adhesion test methods on the same test panels to establish the precision of both methods as well as a comparison of the test results between them. Investigation is currently underway to select appropriate coated coupons, with the round-robin test variables to be agreed upon at the next meeting. The intent is that all round-robin testing be completed so that the data can be ready in time for the next scheduled revision of D 4541 in 1989.

D01.46.10, Condition Assessment, M.E. McKnight, Chairman, discussed Draft #3 of the proposed "Standard Guide for Assessing the Condition of Aged Coatings on Steel Surfaces and the Condition of the Surface of the Underlying Substrate." Previous drafts already have been circulated to the group with all comments incorporated into the latest version. The group recommended that Draft #3 be submitted to Sub. D01.46 ballot.

D01.46.11, Zinc-Rich Coatings, R.H. Wakefield, Chairman, reported that the Draft #5 of the proposed "Standard Test Method for Measuring the Degree of Cure for Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub" received one negative vote (C. Stevens) and one comment (B. Appleman). The negative involved the title, suggesting that the method assesses the degree of cure, whereas it was Stevens' opinion that the method addresses MEK resistance. It was agreed that the title should be changed to read "Measuring the MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub," followed by associated editorial changes throughout the text. However, the scope (1.1) will indicate that a correlation has been found between the cure of some inorganic zinc-rich coatings and the MEK resistance.

A second portion of the negative refers to the use of a control by rubbing the surface with cheesecloth that had not been saturated with MEK in order to assess the effect of burnishing. It was agreed that this should be included in the method.

Finally, the negative requested a definition of "moderate" pressure when conducting the test. The definition could not be agreed upon, and it was decided that moderate pressure should remain. Stevens was contacted by phone and agreed to withdraw the negative based on the editorial changes. However, due to the significance of the changes, it was agreed that the method should be resubmitted to D-1 ballot.

The comment by Appleman involved the reference to the report in footnote 1. He indicated that the report is available from SSPC, and that the U.S. Dept. of Transportation reference is no longer valid. The reference will be changed to SSPC.

SUBCOMMITTEE D01.47 MASONRY COATINGS

F. Gale, Chairman

The new officers of Sub. D01.47 are: Chairman—Frances Gale, ProSoCo, Inc.; Vice-Chairman—Richard C. Herring, Gloucester Co., Inc.; and Secretary—Thomas J. Sliva, D/L Laboratories.

A discussion on the scope of Sub. D01.47 led to the following revision:

"To develop test methods for the evaluation of short and long term performance of water repellent treatments applied to masonry surfaces.

"To develop guides for (a) evaluating the conditions of masonry substrates to establish the need for treatment, (b) surface preparation, and (c) application of treatments."

It was also agreed that several of the task group titles would be revised.

Copies of various NBS, DIN, BSI, Federal, and ASTM standards relating to water repellency were distributed for consideration as a basis for future task groups.

A. Sanders, of Wacker Silicone Corp., made a presentation entitled, "The Chemistry of Silicone Masonry Water Repellents."

D01.47.01, The Efficiency of Water Repellent Materials on Brick, R.C. Herring, Chairman, submitted copies of BS 6477, "British Standard Specification of Water Repellents for Masonry Surfaces," and a draft test method for determining water absorption of treated specimens based on this standard. A discussion followed about the choice of the proposed "Practice for Visual Determination of the Relative Lightfastness of Artists' Materials" and discussed minor changes suggested by the Inter-Society Color Council Project Committee #37, Artists' Materials, at its April meeting. This standard addresses the need for artists to be able to check the lightfastness of materials such as colored pencils, pastels, and other coloring and colored materials. It uses ISO Blue Wool Standards as controls in tests that expose materials to light coming through a window. Some other sections were read and discussed. It was agreed that this standard is ready for ballot.

D01.57.11, Gouache Paints, T. Takigawa, Chairman. P. Hesse agreed to write a definition for gouache paints. It was further agreed that the chairman, with the support of other task group members, would have recommendations on the following by the May 1988 meeting:

(1) *Opacity Test* — It was suggested that mass tone drawdowns be made on the unlacquered side of half black, half white Leneta or Morest charts, that these drawdowns be measured and, depending on the ratio of measurements for the paint over the black and over the white substrate, the paints be assigned to one of a few (say three or four) opacity categories.

(2) *Lightfastness Test* — (a) Instructions are needed on how to prepare specimens; (b) What exposure tests should be required since exposure in South Florida is inappropriate? Possibilities include exposure in Xenon arc apparatus and/or fluorescent apparatus, as described in D 4303, or exposure to natural light indoors; (c) How many exposures should be required?; (d) Must every paint be tested in each manufacturer's vehicle, or can pigments be tested in a generic gouache paint?; (e) Should the paints be diluted with white before testing and, if so, how much?; (f) How should lightfastness categories be established?

(3) *Strength Determination* — Since gouache paints are often used in the mass tone and are less frequently intermixed with other paints, the strength of the color in mixtures with white may be less important than that the mass tone have a suitably high chroma. It was suggested that mass tone drawdowns be made and measured, then the measurement converted to a Munsell notation. A minimum chroma designation would be set for each pigment. In this case, how would the more transparent paints be handled, i.e., multiple measurements be taken on one sample and averaged, or the specimen coated until opaque before measurement? This last alternative would result in much lower chroma figures for the paint.

DIVISION 60
PAINT APPLICATION

SUBCOMMITTEE D01.61 PAINT APPLICATION TOOLS

F.B. Burns, Chairman

D01.61.01, Paint Brushes, T.J. Sliva, Chairman, presented the results of the recently completed, revised round-robin testing on paint brush discharge characteristics performed by six cooperators. The results indicated much improved interlaboratory correlation between cooperators. A discussion was held on improvements of the test method. It was decided to initiate another round-robin reducing application time, and using a more porous substrate to accentuate differences in results. A recommended practice for proper use of a paint brush was discussed briefly. It will be rewritten for potential future inclusion.

D01.61.04, Nomenclature and Definitions, R. Dean, Chairman. R. Summerlin chaired the meeting in the absence of the chairman. She submitted a compilation of 53 terms and definitions as potential ASTM Standard Definitions and 16 potentials for "Description of Terms" for use in specific standards to be developed. The group reviewed approximately 70% of the first group and this information will be refined by the chairman for distribution prior to the next meeting, along with the remainder of the terms. It was also suggested that the final results be communicated to the American Brush Manufacturers Association.

D01.61.05, Bulk Density of Filaments and Bristles, T. O'Brien, Chairman, will have as its objective the development of a standard test method for use by paint brush producers and filament and bristle suppliers to measure this important parameter. The chairman requested that suggested methods be sent to him for consideration at the next meeting.

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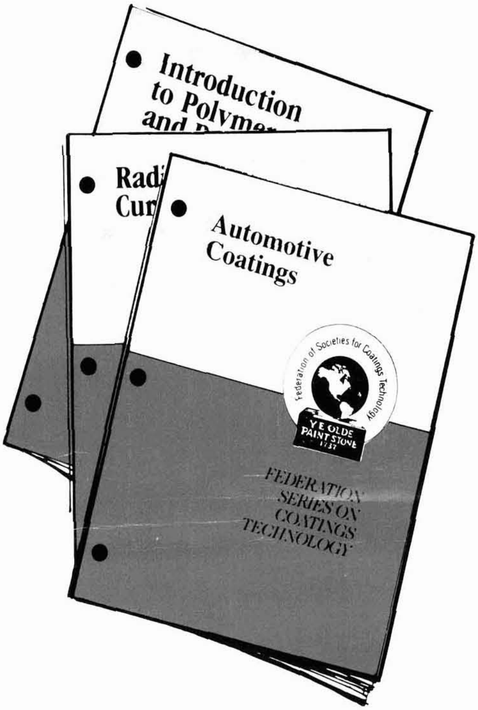

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Society Meetings

BALTIMORE............... MAY

"High Speed Dispersion"

The slate of officers for 1987-88 is: President—Edward B. Countryman, of Bruning Paint Co.; Vice-President—Helen L. Keegan, of Valspar Corp.; Secretary—Donald Hilliard, of Unocal; Treasurer—Gary Morgereth, of McCormick Paint Works Co.; and Society Representative—Joe Giusto, of Lenmar, Inc.

The evening's first speaker was Edwin "Stretch" Kaufman, of George A. Milton Can Co. A member of the New York Society, Mr. Kaufman spoke on "HIGH SPEED DISPERSION AND BLADES."

The speaker presented a history on the progression of mills in the coatings industry from 1940 to the present. Slides displayed blending, grinding, as well as new and worn blades. Mr. Kaufman gave a detailed explanation on how to detect a worn blade. The speaker pointed out that some blades are made of polyvinyl plastic and are not resistant to all types of solvent. He followed with an explanation on the vortex of a batch of paint in the various stages of grinding as it "wets out."

The following points were made during the question and answer period:

(1) better mixing occurs when the vortex goes beyond the blade;

(2) large toothed blades are recommended for high viscosity material and an epoxy system;

(3) high toothed blades are preferable when using a high viscosity, high solids material;

(4) slowing down the speed and lifting the blade closer to the surface of an air-entrapped, warmed up, puffy material will deaerate the paint;

(5) experience with Teflon blades has been poor; and

(6) plasma (chrom/ox.) blades are good for abrasive material.

The evening's second speaker was Society member Bill Sickels, of Lenmar, Inc. Mr. Sickels presented a talk on the true meaning of Flag Day.

HELEN L. KEEGAN, *Secretary*

GOLDEN GATE............ JUN.

"Hazard Evaluation"

A moment of silence was dedicated to the memory of Society member Paul Laliberte who died recently.

Society officers for 1987-88 are, as follows: President—Timothy Donlin, of Pacific Coast Chemical Co.; Vice-President—David Filson, of Clorox Corp.; Secretary—Gordon Pioch, of Triangle Coatings, Inc.; Treasurer—Ernest J. Soldavini, of Nuodex, Inc.; and Society Representative—Barry Adler, of Royell, Inc.

Distinguished Service Awards were presented to Ernest "Bud" Harmon, of Borden Chemical Co., and Robert T. Miller, of Frank W. Dunne Co., for their many years of service to the Society and the coatings industry.

Zoltan Saary, of Chevron Research Co., will be the instructor for the "Introduction to Coatings Technology" course for 1987-88.

The meeting's guest speaker was Richard Jackson, of the California Department of Health Services. Dr. Jackson is in charge of the Hazard Evaluation Section for toxic chemicals in air, water, food, and pesticides.

The speaker covered areas of concern which affect everyone, including: occupational hazards for California farm workers; consumer products, such as art supplies for children; a response to Proposition 65; and handling communication of risk information to the public.

Dr. Jackson noted that pesticides are a one billion dollar industry in California, but there is a lack of data on the toxicity of chemicals used as pesticides. Presently, there has been no EPA occupational health-hazard assessment for the 300,000 agricultural workers and the 10,000 workers engaged in mixing, blending, and applying pesticides.

According to the speaker, public credibility and trust of a product is important. Dr. Jackson concluded by saying that the market place will have a big influence on the type of pesticides that will be allowed to be used on foods. He continued, stating that the market place will have an influence on the type of pesticides allowed as consumers and the large food chain stores refuse to purchase or sell foods that use pesticides harmful to one's health.

ERNEST J. SOLDAVINI, *Secretary*

PACIFIC NORTHWEST..... MAY

Federation Visit

In attendance were Federation President Carlos E. Dorris, of Jones-Blair Co., Fed-

NEW YORK SOCIETY OFFICERS for 1987-88 are (l-r): Secretary—Arthur A. Tracton, of Tile Council of America, Inc.; President—John W. Burlage, of Pacific Anchor Chemical; Vice-President—Irwin H. Young, of Jesse S. Young Co., Inc.; and Treasurer—Roger P. Blacker, of Whittaker, Clark, & Daniels, Inc.

eration Executive Secretary Robert F. Ziegler, and Director of Field Services Thomas Kocis.

President Dorris reviewed the work of various Federation Committees, especially the Educational, Corrosion, and Professional Development Committees.

Officers for 1987-88 are: President—Yvon Poitras, of General Paint Corp.; Vice-President—John H. Daller, of McCloskey Corp.; Secretary—Emil Iraola, of Olympic Home Care Products Co.; Treasurer—John Berghuis, of NL Chemical Canada, Inc.; and Society Representative—Carlton Huntington, of Chemical Distributors, Inc.

John H. Daller, *Secretary*

PIEDMONT .... JUN.

25-Year Pins

Gary L. Water, of Sadolin Paint Products, Inc., was elected Treasurer for 1987-88.

Twenty-five Year Pins and Certificates were mailed to the homes of Gerard M. Currier and Charles H. Gifford, of Reliance Universal, Inc., and James Drum, of Nationwide Research Corp.

Barry F. York, *Secretary*

Constituent Society Meetings and Secretaries

BALTIMORE (Third Thursday—Snyder's Willow Grove, Linthicum, MD). Donald Hilliard, Unocal, 1500 Carbon Ave., Baltimore, MD 21226. Virginia Section—Fourth Wednesday, Ramada Inn-East, Williamsburg, VA.

BIRMINGHAM (First Thursday—Strathallan Hotel, Birmingham, England). D.A.A. Wallington, Macpherson Drynamels Ltd., Westgate, Aldridge, West Midlands WS9 8YH England.

CHICAGO (First Monday—meetings alternate between Como Inn in Chicago and Sharko's West in Villa Park). Kevin P. Murray, DeSoto, Inc., 1700 S. Mt. Prospect Rd., Des Plaines, IL 60018.

CDIC (Second Monday—Sept., Jan., Apr., June in Columbus; Oct., Dec., Mar., May in Cincinnati; and Nov., Feb. in Dayton). N. Jay Huber, Jr., Paint America Co., 1501 Webster St., Dayton, OH 45404.

CLEVELAND (Third Tuesday—meeting sites vary). Ilona Nemes-Nemeth, Sherwin-Williams Co., Cleveland Technical Center, 601 Canal Rd., Cleveland, OH 44113.

DALLAS (Thursday following second Wednesday—Executive Inn. Near Lovefield Airport). Steve Stephens, Ribelin Sales, Inc., P.O. Box 461673, 3857 Miller Park Dr., Garland, TX 75046.

DETROIT (Second Tuesday—meeting sites vary). Liana Callas Roberts, A.T. Callas Co., 1985 W. Big Beaver, Suite 308, Troy, MI 48043.

GOLDEN GATE (Monday before third Wednesday—Alternate between Francesco's in Oakland, CA and Leaning Tower Restaurant in S. San Francisco). Gordon Pioch, Triangle Coatings, Inc., 1930 Fairway Dr., San Leandro, CA 94577.

HOUSTON (Second Wednesday—Look's Sir-Loin Inn, Houston, TX). James Ignatow, O'Brien Corp., P.O. Box 14509, Houston, TX 77221.

KANSAS CITY (Second Thursday—Cascone's Restaurant, Kansas City, MO). Nick Dispensa, Davis Paint Co., P.O. Box 7589, N. Kansas City, MO 64116.

LOS ANGELES (Second Wednesday—Steven's Steak House, Commerce, CA). James F. Calkin, E.T. Horn Co., 16141 Herron Ave., La Mirada, CA 90638.

LOUISVILLE (Third Wednesday—Executive West Motor Hotel, Louisville, KY). Raymond L. Mudd, Porter Paint Co., Coatings Div., P.O. Box 1439, 400 S. 13th St., Louisville, KY 40201-0439.

MEXICO (Fourth Thursday—meeting sites vary). Arturo Ita, Pinturas Aurolin, S.A. De C.V., Poniente 150 No. 750, Nueva Industrial Vallejo, 02300 Mexico, D.F.

MONTREAL (First Wednesday—Bill Wong's Restaurant). M. Marc Lemieux, Reichhold Canada Inc., P.O. Box 120, St. Therese, Que., Canada J7E 4J1.

NEW ENGLAND (Third Thursday—LeChateau Restaurant, Waltham, MA). Joseph T. Schrody, Unocal Chemicals Div., 90 Cumberland Rd., Leominster, MA 01453.

NEW YORK (Second Tuesday—Landmark II, East Rutherford, NJ). Arthur A. Tracton, Hempel Technology, Foot of Curie Ave., Wallington, NJ 07057.

NORTHWESTERN (Tuesday after first Monday—Jax Cafe, Minneapolis, MN). Mark W. Uglem, Hirschfield's Paint Mfg., Inc., 4450 Lyndale Ave., N., Minneapolis, MN 55412. Winnipeg Section (Third Tuesday, Marigold Restaurant)—Neil Webb, Phillips Paint Products Ltd., 95 Paquin Rd., Winnipeg, MB, Canada R2J 3V9.

PACIFIC NORTHWEST (Portland Section—Tuesday following second Wednesday; Seattle Section—the day after Portland; British Columbia Section—the day after Seattle). Emil Iraola, Olympic Home Care Products Co., 1141 N.W. 50th, Seattle, WA 98107.

PHILADELPHIA (Second Thursday—Williamson's, GSB Bldg., Philadelphia, PA). Orville E. Brown, M.A. Bruder & Sons Inc., 52nd & Grays Ave., Philadelphia, PA 19143.

PIEDMONT (Third Wednesday—Howard Johnson's, Brentwood Exit of I-85, High Point, NC). Forrest G. Fleming, Reliance Universal, Inc., P.O. Box 2124, High Point, NC 27261.

PITTSBURGH (Second Monday—Montemurro's, Sharpsburg, PA). James Lore, Watson Standard Co., P.O. Box 11250, Pittsburgh, PA 15238.

ROCKY MOUNTAIN (Monday following first Wednesday—Bernard's Arvada, CO). Paul D. Silva, Kwal Paints, Inc., 3900 Joliet St., P.O. Box 39485, Denver, CO 80239.

ST. LOUIS (Third Tuesday—Salad Bowl). Howard Jerome, Mozel Equipment Co., 4003 Park Ave., St. Louis, MO 63110.

SOUTHERN (Gulf Coast Sections—Third Thursday; Central Florida Section—Third Thursday after first Monday; Atlanta Section—Third Thursday; Memphis Section bi-monthly on Second Tuesday; Miami Section—Tuesday prior to Central Florida Section). Kenneth W. Espeut, Carter Coatings Corp., 6110 Gunn Hwy., Tampa, FL 33624.

TORONTO (Second Monday—Cambridge Motor Hotel). Roy A. Donnelly, St. Lawrence Chemical Co. Ltd., 321 Humberline Dr., Rexdale, Ont., Canada M9W 5T6.

WESTERN NEW YORK (Third Tuesday—meeting sites vary). Marko K. Markoff, 182 Farmingdale Rd., Cheektowaga, NY 14225.

Federation Membership Anniversaries

25-Year

Cleveland

George B. Dreka, Byk-Chemie USA
Ted McNamara, Seegott, Inc.
Victor G. Sandorf, Harshaw/Filtrol Partnership
Helen Skowronska, Retired
M. Janice Tillman, Sherwin-Williams Co.

Los Angeles

John M. Benson, McKesson Chemical Co.
John G. Locke, Angeles Chemical Co., Inc.
Walter L. Standridge, W L S Coatings, Inc.

Piedmont

Charles H. Clifford, Reliance Universal, Inc.
Gerard M. Currier, Reliance Universal, Inc.
James Drum, Nationwide Research Corp.

Future Society Meetings

Baltimore

(Oct. 14)—"Collaborative Testing Services, Inc."—Charles G. Leete, Collaborative Testing Services, Inc.

(Nov. 18)—Joint Awards Meeting with Baltimore Paint & Coatings Association.

(Jan. 21)—"Vinyl Acrylics in Rheology Modified Coatings"—Richard Caldwell, Reichhold Chemicals.

Birmingham

(Nov. 5)—"Practical Applications of Computer Color Match Prediction"—Ashley Anderson, Pacific Scientific Limited. Also Annual Ladies' Lecture.

(Dec. 3)—Dr. A. Learner Memorial Lecture—"Instrumentation in a Paint Laboratory"—Luigi Cutrone, Tioxide Central Laboratories.

(Jan. 7)—"Thin Film Powder Coatings"—Sid T. Harris, Consultant.

(Feb. 4)—"The Future of the Paint Industry—A Personal View"—Quintin Knight, ICI Paints.

(Mar. 3)—"Fundamentals of Water-Based Technology"—G. Brown, Harlow Chemical Co. Also, Annual Ladies' Night Dinner Dance.

(Apr. 7)—"Improving Dispersion of Pigments"—Allen Cowley, ICI Chemicals and Polymers Limited.

(May 5)—59th Annual General Meeting.

Louisville

(Oct. 21)—"The Truth About Accelerated Weathering"—Donald M. Grossman.

(Nov. 18)—"Environmental Systems"—Michael J. Schmutzer, Disti, Inc.

(Dec. 5)—Joint LSCT/Louisville Paint & Coatings Association Christmas Dance.

(Jan. 20)—FSCT Officers and Past Presidents' Night.

New York

(Oct. 13)—"Blocked Isocyanates in Coatings"—Terry Potter, Mobay Chemical Corp.

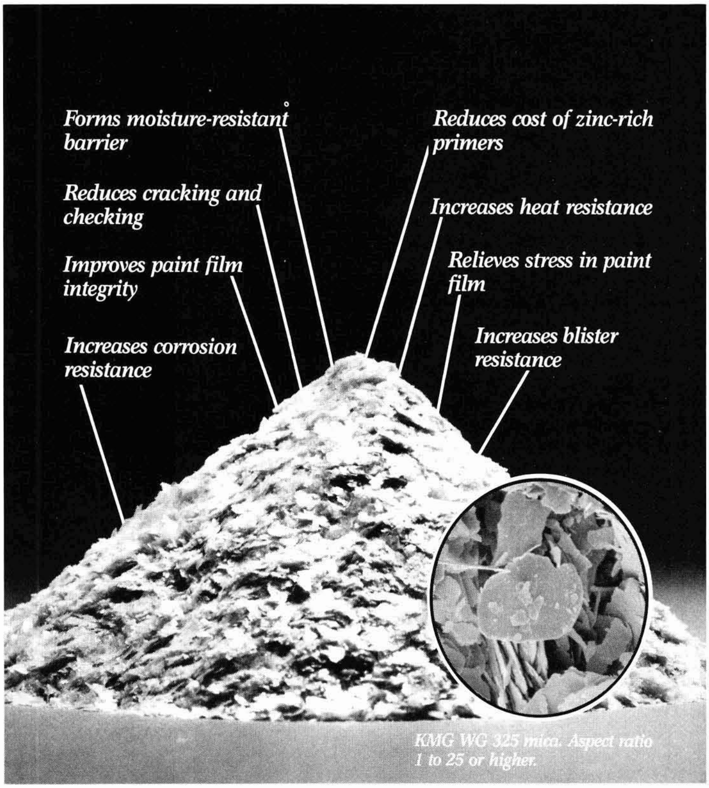
(Nov. 10)—"Calculation of VOC Data as Required by EPA"—Joseph Weinberg, Permuthane, Inc.

Western New York

(Oct. 20)—"Silicones for Today's Paint Industry"—Robert W. Vash, Byk-Mallinckrodt, Carriage House Restaurant, Cheektowaga, NY.

(Nov. 17)—"The Case for In-House Solvent Recovery"—Michael Schmutzer, Disti, Inc., Ramada Inn, Williamsville, NY.

(Dec. 15) "Powder Coating Technology and Formulation"—Tim Sandor, Pratt & Lambert, Inc., Saratoga Restaurant, Buffalo, NY.



Federation of Societies for Coatings Technology

AUDIO/VISUAL PRESENTATIONS

(All A/V presentations include slides, cassette tapes and scripts)

CAUSES OF DISCOLORATION IN PAINT FILMS

Some of the common causes of paint discoloration, such as mildew, sulfide staining, dirt retention, and staining by cedar or redwood, are illustrated on houses and on painted panels. Chemical tests for distinguishing between these types of stains are shown. A test for distinguishing efflorescence and chalking of paint films is also described. 15 minutes (37 slides) **$40**

THE SETAFLASH TESTER

Produced by the Technical Committee, Birmingham Paint, Varnish and Lacquer Club

The Setaflash Tester offers the capability to quickly ascertain the flash point of a volatile product. This presentation describes the equipment and explains the procedures for determining flash point in two temperature ranges (ambient to 110° C; 0° C to ambient) by both the flash/no-flash method and the definitive method. 54 minutes (134 slides) **$100**

FEDERATION TRAINING SERIES ON TEST METHODS*

Volume II

(3 Lessons) Lessons vary from 7 to 11 minutes (79 slides . . . **$70**

Lessons	***Producing Society***
A Simple Method to Determine Microbiological Activity	Philadelphia
A Salt Spray (Fog) Testing Cabinet	Golden Gate
Wet Film Thickness Gages	Golden Gate

*Volume I not available at this time.

HIGH SPEED DISPERSION

Produced by the Manufacturing Committee, Montreal Society for Coatings Technology

The program covers theoretical and practical techniques used for dispersion in paint plants, showing laboratory test equipment and plant scale manufacturing procedures. 20 minutes (60 slides) **$65**

INTRODUCTION TO RESIN OPERATIONS

Produced by the Manufacturing Committee, Toronto Society for Coatings Technology

This presentation has been developed to assist in the selection and training of resin plant operators, and focuses on basic concepts of manufacture and the role of a resin operator. 12 minutes (58 slides) **$65**

A BATCH OPERATED MINI-MEDIA MILL

Produced by the Manufacturing Committee, New York Society for Coatings Technology

This presentation describes the design and operation of a batch operated mini-media mill, and was developed to assist in the training of plant personnel to operate such equipment. 8½ minutes (51 slides) **$60**

NOW AVAILABLE!

OPERATION OF A VERTICAL SANDMILL—(Produced by the Manufacturing Committee, Kansas City Society for Coatings Technology). This program describes the design and operation of a vertical sandmill, to assist in the training of plant personnel to operate such equipment. 14 minutes (73 slides).............................. **$75**

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People

William G. Setser has joined Indurall Coatings, Birmingham, AL, as Technical Director. A member of the Houston Society, he will be responsible for research, development, quality assurance, and purchasing for the firm.

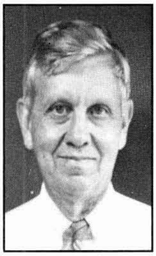
SCM Chemicals, Baltimore, MD, has named **Ray Horath** and **Sandra Mitton** Sales Representatives for the Chicago area. In this position, Mr. Horath and Mrs. Mitton will be responsible for the direct sales and support of all SCM Chemicals products. Mr. Horath is a member of the Chicago Society.

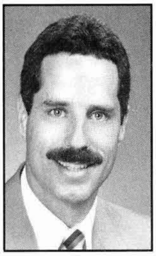
Midland Division, The Dexter Corp., Waukegan, IL, has promoted **Louis J. Sharp IV** to Director of Research and Administrative Services. He joined Midland in 1981 as Manager of Polymer Chemistry and in 1985 was promoted to Manager of Polymer and Analytical Chemistry. A member of the Chicago Society, Dr. Sharp will have responsibility for managing quality assurance, office services, and research/administrative services of the company.

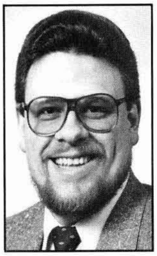
In addition, **Peter M. Stone** has been named Manager, Eastern Region, Building Products, for the firm.

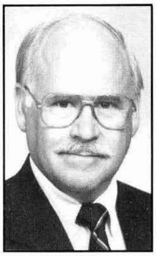
Mark S. DiLorenzo has been appointed Technical Manager of Universal Pigments Dispersions in the Color Division of Harshaw/Filtrol Partnership, Cleveland, OH. His duties will include providing technical service assistance to paint manufacturers to ensure pigment compatibility, as well as other services as needed.

Neil W. Nelson has joined E.C.C. America, Inc., Atlanta, GA, as Product Specialist—White Pigments. His new duties include the marketing of the company's line of ultra fine ground calcium carbonates and high brightness coating clays to the paper industry. He was previously employed by Air Products & Chemicals, Inc.


W.G. Setser


R. Horath


T.J. Jewell


C.W. Vallette

Thomas J. Jewell is President of the newly formed Research Data Access Corporation, Greenville, PA. His eight years of experience in the coatings industry included serving as Laboratory Director for Jamestown Paint & Varnish Co. Mr. Jewell is a member of the Cleveland Society.

Manville, Filtration & Minerals Div., Lompoc, CA, has announced two promotions: **C.N. (Hap) O'Neil** to Manager of National Accounts and Distribution Sales and **Bill Hernandez** to District Sales Manager. Mr. O'Neil is responsible for coordinating and expanding sales of all filtration and minerals products through distributors and the firm's new distribution centers. Mr. Hernandez is responsible for sales of all filtration and minerals products in the western U.S.

Pierce & Stevens Corp., Buffalo, NY, has appointed **John B. Pallasch** to the position of Vice-President, Research. In his nearly 27 years of service to the firm, Mr. Pallasch most recently served as Technical Director/Central R&D Laboratories.

Mississippi Lime Co., Alton, IL, has promoted **David Viox** to the position of Marketing Manager for precipitated calcium carbonate.

Hercules Incorporated, Wilmington, DE, has appointed **Charles W. Vallette** Accounts Manager for Coatings and Additives in Southern California and the 11 western states. Mr. Vallette joined Hercules in 1966 and most recently served as Accounts Manager for Pulpex® synthetic pulps in San Francisco.

Joseph A. Pagliarulo has been named Regional Sales Manager, Chemicals, for USI Chemicals Co., Cincinnati, OH. He will assume responsibility for the sale of the company's wet chemicals, including ethyl alcohol, acetic acid, vinyl acetate monomer, ethyl ether, ethylene oxide, ethylene glycol, and industrial coolants and cleaners in the eastern U.S.

In a series of managerial appointments, Eastman Chemical Products, Inc., Kingsport, TN, has named **Harris P. Cantrell**—Chemical Product Manager for solvents, etylene oxide and Texanol® ester alcohol; **Matt J. Russ**—Market Manager for inks, resins, and chemical specialties; and **George Tipton, Jr.**—District Marketing Manager (Cleveland area) Field Marketing, Chemicals.

In addition, **Collis L. (Bud) Burns, Jr.**, **Richard Plummer**, and **James Romano** have been appointed Marketing Representatives with the Chemicals Field Marketing Div.; **Thomas J. Stevens** has been named a Marketing Specialist; and **Damon Warmack** has assumed the position of Product Specialist for the company.

The Chemicals & Metals Department of Dow Chemical U.S.A., Midland, MI, has appointed **Robert W. Simmons** to the position of Product Marketing Manager for fine organics, and **Jeff Sullivan** to the post of Product Marketing Manager for chlorinated solvents.

The Chemical Products Division of Cargill, Inc., Minneapolis, MN, has elected **John Sweeney** President. He succeeds **Jim Lawrence**, who, as Senior Vice-President of the division, will concentrate on business development in the areas of acrylics, emulsions, adhesives, and paper resins.

Sanncor Industries has appointed **Jeffrey F. Michaels** to the position of Plant Manager at the company's headquarters in Leominster, MA. He will be responsible for the manufacturing, engineering, and safety of the operations.

Clifford Q. Schneider has been appointed Vice-President, Marketing, for Reichhold Chemicals, Inc., White Plains, NY. In this new corporate position, he will assist Reichhold divisions and subsidiaries with identification of marketing opportunities and the development of products and processes to meet those opportunities.

Burton C. Anderson (left), Research Director of Dupont's Fabricated Products Experimental Station Laboratory, presents a commemorative plaque to Joseph A. Vasta, in recognition of 35 years of continuous service to E.I. du Pont de Nemours & Co., Inc. Mr. Vasta, a member of the Philadelphia Society, is Chairman of the FSCT Educational Committee and serves on its Professional Development and Publications Committees. He also serves as a Trustee of the Coatings Industry Education Fund and as a member of the JCT Editorial Review Board.

The Canadian Paint and Coatings Association recently elected **J.J. (Jerry) Castiglia**, of Pratt & Lambert Inc., Chairman of the Board. **W.R. (Bill) MacInnis**, of Interlab Paints, was elected Vice-Chairman of the Board.

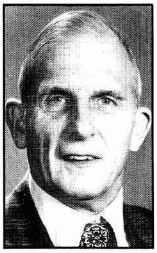
Richard F. Gentile has been appointed Product Manager of the Pharmaceutical Division of Colorcon, Inc., West Point, PA. In his new position, Mr. Gentile will be responsible for the commercial development and sales support for products distributed to the pharmaceutical industry.

Marco Wismer Is Recipient of 1987 Tess Award

The Polymeric Materials: Science and Engineering Division, of the American Chemical Society has announced that the recipient of the 1987 Roy W. Tess Award in Coatings is **Marco Wismer**, of PPG Industries, Inc.

The $1000 Tess Award, funded by a grant from Dr. and Mrs. Tess, recognizes outstanding contributions in the area of coatings science, technology, and engineering. Dr. Wismer is recognized for his achievements in a variety of areas including cathodic electrodeposition, radiation-curable coatings, polyols for urethane foams, fire retardant foams, organic foams, and others.

Dr. Wismer is a major contributor in the fields of coating science and technology, as evidenced by over 100 U.S. and foreign patents, and through his significant body of journal publications and book chapters.



M. Wismer

Dr. Wismer graduated from the Zurich Oberrealschule and received his M.S. and Ph.D. Degrees in Chemistry from the Swiss Federal Institute of Technology. Until he retired in June of this year, he served as Vice-President of Science and Technology at PPG; a position he held since 1984. He is a member of the Executive Board of the international review journal, *Progress in Organic Coatings*.

Dr. Wismer is a member of the Pittsburgh Society. He was a Trustee of the Paint Research Institute from 1977-81.

Morton Chemical Division, Powder Coating Group, Chicago, IL, has promoted **Ron Creech** to Manager, Technical Service. Based in Glenside, PA, Mr. Creech will be responsible for scheduling technical service field calls, line audits, and directing all the technical service group's activities. He will report to **Steven L. Kiefer**, Manager, Sales Support.

Obituary

Thomas G. DiManno, Specialty Products Manager for NL Chemicals, died on July 5. He was 38 years old. Mr. DiManno joined NL Chemicals in 1978 as a Marketing Analyst and was appointed to the company's sales force in 1982. He was promoted to his most recent position in 1985. Mr. DiManno was a member of the Cleveland Society.

Missouri-Rolla Schedules Fall Coatings Short Courses

The University of Missouri-Rolla, Rolla, MO, is sponsoring two coatings short courses this fall. The "15th Introductory—Paint Formulation" is slated for October 19-23, and "Introduction to Polymer Chemistry" will take place November 2-6.

The "Paint Formulation" introductory short course is for persons interested in the basic fundamental concepts of paint formulating science. Topics to be discussed include:

- basic raw materials and their influences on the performance characteristics of the finished coating;
- manufacturing and testing of coatings in the laboratory to meet the state requirements of the job to be done with a minimum of inventory complication;
- limitations of plant production equipment and the variables that can be introduced when a coating is moved from the laboratory to the plant;
- systems for reducing variables in screening series; and
- modern chemical instrumentation as applied to coatings.

The "Polymer Chemistry" short course is designed to give a strong overview of polymer chemistry from synthesis to kinetics to analysis. The course will benefit persons in the plastics, resins, or coatings industry; in quality control of polymeric materials; or those who use polymers. The course will cover the physical and chemical properties of polymers, and the methods of preparation of polymers. Special emphasis is placed on how changes of chemical structure affect the physical properties of these polymer systems. The use of instrumentation to evaluate and test polymers is covered. Insight into the selection of a given polymer system for a specific application will be given.

In addition, UMR has announced its preliminary spring 1988 schedule of coatings short courses. Tentative courses and course dates are as follows: "Maintenance Coatings for Contractors and Paint Inspectors"—February 22-24; "56th Introductory Short Course—The Basic Composition of Coatings"—February 29-March 4; "16th Introductory—Paint Formulation"—March 14-18; "Conformal Coatings"—April 18-20; and "Physical Testing of Paints and Coatings"—May 16-20.

For additional information, write the Coatings and Polymer Science Program, Dept. of Chemistry, UMR, Rolla, MO 65401-0249.

Water-Borne Symposium Proceedings Available

Proceedings of the 1987 Water-Borne and Higher-Solids Symposium, held in New Orleans, LA, on February 25-27, 1987, are now available. The publication contains 30 papers which discuss the chemistry, formulation, and new developments in water-borne and higher-solids coatings. The symposium is cosponsored by the Southern Society for Coatings Technology and the Department of Polymer Science at the University of Southern Mississippi. Proceedings may be ordered for $65 ($70 foreign orders) from Gordon L. Nelson, Dept. of Polymer Science, Southern Station Box 10076, Hattiesburg, MS 39406-0076.

NACE Schedules Regional Conference

The South Central Region of the National Association of Corrosion Engineers (NACE) is sponsoring a special conference for November 16-18, at the Lafayette Hilton and Towers, Lafayette, LA. A wide gamut of corrosion subjects directly related to the industry's corrosion problems will be discussed.

Many of the technical papers to be presented by experts of the industry will address the four methods of controlling corrosion: chemical corrosion inhibitors, cathodic protection, protective coatings and linings, and materials selection. Other topics to be included in the technical program are offshore structures, failure analysis, corrosion monitoring, elastomers, and environmental concerns. A special session will be devoted to the use of computers in corrosion work.

Tabletop exhibits will present the latest products, services, and techniques in the prevention and control of corrosion.

A detailed conference brochure can be obtained by contacting the Conference General Chairman, Mark Gautreaux at (318) 837-3578.

CALL FOR PAPERS

PRA 8th International Conference
Biodeterioration of Coatings and Substrates
Nov. 7-9, 1988 Amsterdam, The Netherlands

The Paint Research Association invites interested persons to submit papers for its Eighth International Conference, to be held on Nov. 7-9, 1988, in Amsterdam. The theme of the conference is "Biodeterioration of Coatings and Substrates."

Biodeterioration problems facing coatings manufacturers and users fall into four broad categories: biodeterioration of paint films by fungi or algae; viscosity loss, gassing, and malodors in water-borne paints; protection of wood by paints, stains, and preservatives; and marine fouling of ships and small boats (with associated toxicity problems).

From its inception, the PRA has been actively involved with all four problems and numerous papers have been published and presented at international gatherings. In association with the Dutch Paint Research Institute (TNO), PRA is organizing this conference to cover all four topics, including test methods, toxicity of biocides, and legislation.

To insure the broadest possible program, PRA is calling for contributions relating to original experimental work from coatings manufacturers/users, raw material suppliers, research institutes, and the academic world. The Organizing Committee will select 16 papers on the basis of their merit and relevance.

Those interested in presenting a paper must submit a summary (200-300) words to: Mr. Dip Dasgupta, Head of Information Dept., Paint RA, Waldegrave Rd., Teddington, Middlesex TW11 8LD England.

Society of Manufacturing Engineers Announces Fall Slate of Programs

The Society of Manufacturing Engineers (SME), Dearborn, MI, is sponsoring the following four programs this fall:

"INDUSTRIAL PAINTING PROCESSES"

The program is scheduled for October 27-29, at the Doubletree Hotel, Anaheim, CA. The three-day program will present modern industrial coatings, their applications, and a thorough discussion of California environmental regulations.

Topics scheduled include: paint principles, finishing processes and their current applications, production problems, paint defects, and the alternatives to California EPA regulations. Special focus will be on the painting industry located in the west.

Ron Joseph, President, Ron Joseph & Associates Inc., will be the program instructor.

"SPRAY APPLICATIONS FOR FURNITURE FINISHING"

The one-day program is scheduled for November 16, at the Holiday Inn-High Point, High Point, NC. The program will focus on the advantages and limitations of the most popular spray finishing systems used today by the wood/metal furniture industries.

Subject matter to be examined includes: coatings materials, fundamentals of finishing systems, compressed air and airless atomization, electrostatics, airless rotary belt, disk, plural component spray, preventive maintenance, and future trends in wood and metal finishing.

The program instructor is Jerry Hund, Training Instructor, Binks Manufacturing Co.

"SPRAY APPLICATIONS WORKSHOP FOR FURNITURE FINISHING"

The three-day, hands-on workshop is slated for November 17-19, at the Holiday Inn-High Point. The program will involve hands-on and problem solving exercises, and focus on the advantages and limitations of the most popular spray finishing systems used today by the wood/metal furniture industries.

Issues scheduled for discussion include: coatings materials, fundamentals of finishing systems, hands-on and problem solving exercises, spray booths, operator techniques, preventive maintenance, and future trends in wood and metal finishing. The hands-on exercises will take place at the Lilly Paint Co.

Jerry Hund will also be the instructor for this program.

For more information on these programs, contact Jim Warren, SME, Special Programs Div., One SME Dr., P.O. Box 930, Dearborn, MI 48121.

"FINISHING AUTOMOTIVE PLASTICS"

The Association for Finishing Processes of SME is sponsoring this educational clinic to be held November 18-20, at the Hyatt Regency Dearborn, Dearborn, MI.

The clinic and tabletop exhibits will examine the difficult task of finishing plastics for the automotive industry. Various substrates will be discussed with special attention given to their properties and finishability. New coatings and application processes will also be examined.

The event will be co-chaired by Mackenzie K. Endo, Manufacturing Development Engineer, Ford Motor Co., and Judy M. Riede, Senior Technical Engineer, Paint Systems, CPC Group, General Motors Corp.

Persons interested in attending, exhibiting, or obtaining further information should contact Diane Korona, The Association for Finishing Processes of SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121.

Du Pont Schedules Seminar On Hazardous Waste, Nov. 3-5

The Du Pont Co., Wilmington, DE, has announced a new seminar that will help companies comply with the Resource Conservation and Recovery Act (RCRA) and improve their hazardous waste management programs. The "Fundamentals of RCRA" is scheduled for November 3-5, in Houston, TX.

The two- and one-half day seminar is directed at those responsible for hazardous wastes. It offers a practical understanding of the amended law, which increased pressures on manufacturers of all sizes to better manage, generate, treat, store, and dispose of hazardous wastes.

Classes will be kept under 30 people, so that individual situations can be explored. Topics to be covered include: characterizing wastes and reporting findings to the EPA; cradle-to-grave responsibility; packaging and labeling; small vs "regular" generator status; accumulation vs storage; shipping hazardous wastes; management issues, including waste minimization, contingency planning, emergency preparedness, training, inspections, inventory control, and permitting; facilities for treatment, storage, and disposal; closure and related responsibilities; and disposal methods.

For information write the Du Pont Co., P19-1105, P.O. Box 4500, Greenville, DE 19807.

38th SSPC Conference to Feature Symposium On Field Reliability of Protective Coatings

The Steel Structures Painting Council will sponsor its 38th National Conference and Protective Coatings Exposition in Orlando, FL, on November 2-5.

The theme of the conference symposium, "Improving the Field Reliability of Protective Coatings," will focus on new developments, techniques, and materials that can improve the reliability of field-applied coatings systems. Twenty presentations have been scheduled, including sessions on "Coatings Management Programs," "Advances in Quality Assurance, Application, and Inspection," "Advances in Surface Preparation Techniques," and "Improvements in Field Applied Low VOC Coatings."

In addition to nine informal technical seminars, the SSPC in planning tutorial sessions, taught by recognized industry leaders, on practical techniques for users to get the most from their coatings investment. These tutorials include "Developing Inspection Programs" and "Estimating Painting and Protection Costs."

Over 1000 people are expected to attend this year's conference. Groups represented include plant owners, maintenance engineers, coatings and corrosion engineers, paint and raw materials manufacturers, equipment suppliers, consultants, regulatory agencies, and universities.

At the Protective Coatings Exposition held concurrently with the symposium, more than 100 exhibits will feature the latest developments in equipment, materials, and services dedicated to protective coatings and linings for industrial maintenance.

For additional information, contact SSPC, Nov/87 Conference, 4400 Fifth Ave., Pittsburgh, PA 15213-2683.

Corrosion/88, Mar. 21-25 To Feature Over 350 Papers

More than 5000 people are expected to visit the 1988 Materials Performance & Corrosion Show to be held March 22-24, 1988, at the Cervantes Convention & Exhibition Center, St. Louis, MO. The show is held in conjunction with Corrosion/88, the international conference sponsored by the National Association of Corrosion Engineers (NACE).

This show is devoted to products, services, and techniques to help reduce the billions of dollars lost each year in the United States due to corrosion.

Corrosion/88, to be held March 21-25, will feature over 350 technical papers on corrosion topics.

Advances in Polyurethanes Seminar, Nov. 9-10

The two-day seminar, "Advances in Polyurethanes," will be held Nov. 9-10 in Atlanta, GA. The seminar is designed to show how current technologies can be applied to the manufacture of specialty polyurethane products.

The first day's topic will be the basic research of elastomers and coatings. Kurt Frisch, Polymer Institute, Univ. of Detroit, will explain the chemistry, materials, and compounding techniques for formulating polyurethane coatings for specific end uses.

The second segment of the seminar will focus on polyurethane elastomers in coatings sealants and adhesives. Product formulations, manufacturing processes, and marketing/applications will be presented for many specialty areas.

Subjects of instruction will include: new developments in raw materials; formulations and structure property relationships in polyurethane cast elastomers and thermoplastics; RIM and polyurethane elastomers; IPN-RIM systems; coating systems; high-temperature-resistant isocyanate coatings; crosslinked specialty elastomers; polyurethane coatings; sealants; and adhesives.

Other seminar speakers include: C. Schollenberger, Consultant, B.F. Goodrich; Norman W. Gill, of the Tennant Co.; Robert Evans, of Case Western Reserve Univ.; and Denis J. Zalucha, of S.C. Johnson & Son, Inc.

For a full description and registration information, write Program Div., Technomic Publishing Co., Inc., 851 New Holland Ave., Box 3535, Lancaster, PA 17604.

Don't Miss The
Convention
Follow Up Issue
In
December

Polymeric Products

A 20-page booklet provides detailed information on surfactants, dispersants, and resins. The literature includes the chemical compositions, properties, mechanism descriptions, benefits, and applications on each product. For a copy of "Hypermer® Polymerics for Industrial Chemicals," contact Loretta Ianni, ICI Specialty Chemicals, Wilmington, DE 19897.

Color Data Processing System

A product bulletin highlights a new color matching and quality control system designed to measure and control the color of pigmented materials. The color data processing system utilizes high resolution color graphics to display the effects of metamerism and color change. Software programs include colorimetry, quality control, tolerance determination pass/fail, laboratory and production formulation and correction. For more details on the Pigmenta color data processing system, contact Larry Backus, Ahiba, Inc., 4123-D Rose Lake Dr., Charlotte, NC 28217.

Multifunctional Surfactants

The physical properties and performance characteristics of surfactants which function as wetting agents, viscosity stabilizers, and defoamers in a variety of water-based systems are highlighted in a new data sheet. The publication includes information regarding surface tension, dynamic surface tension, solubility, and formulation guidelines. The Surfynol® 400 series data sheet is available from Air Products and Chemicals, Inc., Performance Chemicals Div., Allentown, PA 18195.

Rust Preventive

Current formulation and application information for a line of rust preventive concentrates used to make high-performance protective coatings is summarized in a bulletin. The literature describes nine product systems available in diluent oil, water, and mineral spirits carriers. In addition, composition data, application suggestions, and formulating tips are provided for each product category. A copy of "SACI® Product Composition and Formulating Review" is available from Witco Corp., Sonneborn Div., 520 Madison Ave., New York, NY 10022-4236.

Protective Coatings/Linings

A 104-page, soft-bound book which contains over 30 questions and multiple responses to problems that have occurred in the protective coatings/linings field is in print. Question and response subjects include problems dealing with surface preparation, coatings application, inspection and testing, coating materials, and coatings failure. For a copy of "Problem Solving Forum," write KTA-Tator, Inc., 115 Technology Dr., Pittsburgh, PA 15275.

Epoxy Resin

A new, low viscosity product for use by epoxy resin formulators is the subject of a technical product bulletin. The epoxy resin is a 700 cps liquid epoxy blend which does not contain conventional monoepoxide diluents. Additional data on CMD 50735 epoxy resin is available from G.L. Smith, Interez, Inc., 10100 Linn Station Rd., P.O. Box 37600, Louisville, KY 40233.

Adhesive Emulsion

A technical bulletin describes a new borax-compatible, polyvinyl acetate homopolymer emulsion which can be compounded with starches, dextrins, or reactive pigments like zinc oxide. The emulsion can be used as a heat sealing adhesive, in paper and foil adhesives, in fire retardant mastics, and in caulks. Write W.R. Grace & Co., Organic Chemicals Div., 55 Hayden Ave., Lexington, MA 02173 for details on Daratak® 92L.

Formulating Program

A new computer program designed for the ever changing, formulating environments of the coatings, printing inks, pesticides, and other related industries is featured in a product bulletin. The program provides information about solvents and solvent blend properties, such as solubility parameters, density, surface tension, viscosity, flash point, and solvent compatibility as well as formulation cost in a variety of currencies. The computer program can store physical and performance properties information on over 500 solvents and plasticizers in combination with 450 resins/polymers and 500 pesticides. For further data on SPARC, the Exxon Chemical Solubility Parameter Calculator, write Exxon Chemicals, Performance Products Group, P.O. Box 3272, Houston, TX 77253-3272.

Rate Viscometer

An innovative approach to the field of low viscosity, low shear rate testing has been introduced in literature. The viscometer offers low shear rate measurements in three configurations. In a manual mode, it will display 30 values of shear rate, shear stress, and temperature for viscosity calculations. Adding a speed programmer and recorder allows automatic plots of flow curves, yield points, and thixotropy. Interfacing with an IBM computer provides a fully automatic viscometer, including viscoelastic measurements, calculations, regression models, and plots. For details on the Haake RV20, contact Mike Delancy, Product Manager, or Barbara L. Naser, Advertising/Sales Promotion Coordinator, at Haake Buchler Instruments, Inc., 244 Saddle River Rd., Saddle Brook, NJ 07662-6001.

Dual Scrub Tester

Recently released literature announces a new heavy-duty, dual scrub tester for testing paints, coatings, wall finishes, polishes, etc., in order to examine washability and related properties that affect wear resistance of surfaces. For details on heavy-duty Model 902HD87, write the Paul N. Gardner Co., Inc., P.O. Box 10688, Pompano Beach, FL 33061-6688.

Mechanical Convection Oven

Recently released literature highlights a mechanical convection, bench top laboratory oven with special controls for precise operation. Designed for the testing, curing, and conditioning of various materials at precise temperatures, the oven has a temperature range of 35°C-280°C and operates on 115V, 50/60Hz. For further data, write Hotpack Corp., 10940 Dutton Rd., Philadelphia, PA 19154.

Rotating Electrodes

A product bulletin highlights several new electrode accessories for corrosion measurements and other electrochemical studies. The electrodes permit electrochemical studies of user-defined electrode materials. For more facts on the RDE011 Rotating Cylinder Electrode and RDE001 Quick-Change Rotating Disk Electrode, write EG&G Princeton Applied Research, Electrochemical Instruments Div., CN 5206, Princeton, NJ 08543-5206.

Reactive Diluent

Recently released literature introduces a reactive diluent developed for cationic initiated radiation curable coatings, inks, and adhesives. The reactive diluent functions as a viscosity diluent and adds flexibility to epoxy compositions. Additional information, literature, and samples of Rapi-Cure™ DVE-3 can be obtained from the Acetylene Chemicals Dept., GAF Chemicals Corp., 1361 Alps Rd., Wayne, NJ 07470.

Particle Size Analyzer

Recently released information highlights a particle size distribution analyzer in the range of a few microns down to submicron size. In addition to particle size distribution, the analyzer measures molecular weights and diffusion coefficients by light scattering or high resolution size distributions with a disc centrifuge. For more data, write Scientific Systems Sales Corp., 333 Jericho Turnpike, Jericho, NY 11753.

Solvent Recovery System

A new series of solvent recovery systems which are continuous, on-line separation units featuring an internal scraper for self-cleaning operation are highlighted in an announcement. Recovery systems are available in 70, 110, 175, and 300 gallon capacities. Once on-line, the units handle anywhere from 70-300 gallons continuously as part of the production process. Information on the Auto-Scrape Series can be obtained from Disti Environmental Systems, Inc., 525 Blvd., Kenilworth, NJ 07033.

Coating and Lining Manual

A training manual which contains detailed information on: inspecting prepared steel surfaces; measuring surface profile; inspecting defects in coated surfaces; monitoring environmental conditions; measuring dry film thickness; and holiday (pinhole) testing is available. The "Bechtel Coating and Lining Inspection Training Manual" is available from KTA-Tator, Inc., 115 Technology Dr., Pittsburgh, PA 15275.

Pump Seal

Literature describes easy-to-replace internal pump seals on single and double gear joints. The seals are one-piece integrated units. For more data, write Netzsch Inc., 119 Pickering Way, Exton, PA 19341-1393.

Color Difference

The manual "Prismatic Displays of Measured Color Difference" which gives a set of color difference examples is now available. Colors in eight areas of the full spectrum found in the coil coatings industry were arbitrarily chosen as reference points. Mac Adams units of color differences are also included in the manual. For additional information, contact Wendy E. Webber, National Coil Coaters Association, 1900 Arch St., Philadelphia, PA 19103.

Polybutenes

A 32-page, full-color brochure filled with details of the physical and chemical properties of polybutenes has recently been published. Polybutenes are viscous, nondrying liquid polymers. Major uses include adhesives, caulks and sealants, a cling additive for stretch wrap film, a rubber additive, and an impact modifier for thermoplastics. For a copy of Bulletin 12-L, write Amoco Chemical Co., Dept. MCODE-JCT-BLOK, Mail Code 4106, 200 E. Randolph Dr., Chicago, IL 60601.

Surfactant

A data sheet depicts the physical and surface properties of a surfactant suitable for use in a variety of systems, including paints, inks, dyes, paper coatings, adhesives, agricultural chemicals, construction cements, and metalworking lubricants. For information regarding Surfynol® 104's stability, solubility, defoaming, wetting, and formulating capabilities, write Air Products and Chemicals, Inc., Performance Chemicals Div., Allentown, PA 18195.

Chlorinated Polyolefins

A new brochure which features solution properties for chlorinated polyolefins and typical formulas for various primers and stir-in formulations is available. For a copy of "Eastman® Chlorinated Polyolefins in Coatings for Selected Plastics," publication GN-360A, write Eastman Chemical Products, Inc., Coatings Chemicals, P.O. Box 431, Kingsport, TN 37662.

Filter Cartridge

Information is available on a pleated filter cartridge for aggressive solvents and corrosive fluids. The membrane filter is manufactured with a very uniform pore size distribution and high solid volume. For more details on the BTTR pleated membrane filter cartridge, write Filterite, 2033 Greenspring Dr., Timonium, MD 21093.

Volume Autosampler

A recently released product bulletin describes an on-column, large sample volume autosampler which provides from 10-100 times the normal sample volumes available for capillary GC applications. For additional data on the Carlo Erbra AS 550, contact Bob Bove, Product Manager, Gas Chromatography, or Barbara L. Naser, Advertising/Sales Promotion Coordinator, at Haake Buchler Instruments, Inc., 244 Saddle River Rd., Saddle Brook, NJ 07662-6001.

Batching Controllers

A technical bulletin features batching controllers able to provide the necessary functions to automatically sequence and control batching, mixing, or blending processes. For complete specifications on the Series 9000 Models C and D, write Advanced Process Controls, Inc., 500 W. Cummings Park, Woburn, MA 01801.

Viscosity Conversion Tables

A comprehensive set of viscosity conversion tables is now available. Readings from commonly used cups are readily converted to equivalent centipoise values. A copy of the tables is available from Norcross Corp., 255 Newtonville Ave., Newton, MA 02158.

Scratch Hardness Tester

Recently released literature describes a scratch hardness tester which determines the resistance of coating materials to surface scratching and is of particular use for furniture or vehicle lacquers. The new tester is capable of making scratch hardness measurements in accordance with ASTM D3363, using constant pressure and lead pencils having various hardnesses. A descriptive leaflet is available from the Paul N. Gardner Co., P.O. Box 10688, Pompano Beach, FL 33061-6688.

Planetary Mixers

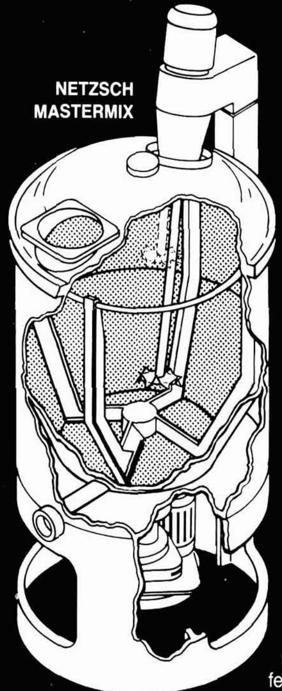
A six-page brochure which describes a line of vacuum double planetary mixers is now available. Nine models are examined, including the five liter model and the 2,350 liter model. A specifications section of the literature includes: overall dimensions, tank volumes, mixing capacity/orbit speeds, blade speeds plus main motor hp, hydraulic lifting motor hp, and hydraulic lifting reservoir capacity. For a copy of the brochure, write Premier Mill Corp., 220 E. 23rd St., New York, NY 10010.

Amido-Amine Hardener

Information is available on a new liquid amido-amine hardener for room-temperature curing of epoxy resins which exhibits a wide mix ratio, permitting variations of resultant epoxy properties. The hardener also offers low viscosity (400-700 cps at 25°C) and high reactivity. To obtain further details on Epotuf® epoxy hardener 95-445, contact Billy K. Winters, Manager Coating Applications, Reichhold Chemicals, Inc., Chemical Coatings Div., P.O. Box 1433, Pensacola, FL 32596.

Urethanes

Two revised and updated technical bulletins on urethanes used by automotive, packaging, aerospace, plastic, roofing, construction, furniture, textile, defense, marine, and flooring industries are available. For copies of "Urethane Adhesives, Coatings and Elastomers," #487, or "Tenacious Urethane Prepolymers, Polyols and Moisture Curing Resins for High Solids Adhesives and Coatings," #465, write Synthetic Surfaces Inc., P.O. Box 241, Scotch Plains, NJ 07076.



Portable Shipping Containers

A 20-page, four-color brochure describing a complete line of portable shipping containers for liquids and flowable dry materials is now available. The brochure describes each model in the line, details their features and benefits, provides specifications, and illustrates special application units. Further information on Jumbo portable shipping containers is available from Clawson Tank Co., 4701 White Lake Rd., Clarkston, MI 48016-0350.

Ketones

A four-page brochure which briefly describes five ketones is available. "Ketones from Eastman®," Publication M-258 on methyl amyl, methyl n-propyl, methyl isoamyl, methyl isobutyl, and C-11 ketones is available from Eastman Chemical Products, Inc., P.O. Box 431, Kingsport, TN 37662.

Resin Modifier

A new highly reactive resin solution designed to improve cure response and certain film properties when used as an additive in thermoset coatings systems is featured in a bulletin. The resin modifier may be considered as a performance additive in primers and topcoats for coil, automotive, flexible substrate, and plastic coatings. For technical data on Modacure™, write MTS Coatings Dept., 730 Worcester St., Springfield, MA 01151.

Cellulosic Resins

A brochure is available on cellulosic resins in metallic pigmented automotive OEM basecoats. The low molecular weight resins are suited for use in higher-solids automotive basecoats for clear-on-base coatings systems. The brochure also includes application information and tables on basecoat composition, solvent composition, and formulation constants, as well as a listing of raw materials and suppliers. For a copy of Publication GN-366 or more information, write Eastman® Chemical Products, Inc., Coatings Chemicals, P.O. Box 431, Kingsport, TX 37662.

Polyurethane Catalyst

A highly stabilized, 100% active stannous octoate and a host of tertiary amine catalysts including several proprietary specialty blends aimed at specific processing are featured in a product bulletin. For supplemental data, write Cosan Chemical Corp., 400 14th St., Carlstadt, NJ 07072.

Viscometer Software

A technical data sheet features application-oriented software which allows for complete characterization of a material's flow properties in a matter of minutes. The software offers a multitude of different test modes that provide maximum flexibility for the operator to fully analyze their material. For more facts on the RV20 viscometer software, contact Mike Delancy, Product Manager, or Barbara L. Naser, Advertising/Sales Promotion Coordinator, at Haake Buchler Instruments, Inc., 244 Saddle River Rd., Saddle Brook, NJ 07662-6001.

Urethane Formulary

A guide to starting formulations for various kinds of coatings based on a line of aqueous urethane dispersions is being offered. The formulary covers paint, architectural coatings, floor care, textile, automotive polish, leather finish, and auto rubber coating. For a copy of "Witcobond® Urethane Dispersions Formulary," write Witco Corp., Organics Div., Dept. U, 2701 Lake St., Melrose Park, IL 60160-3041.

High-Solids Resins

Three new high-solids resins, based on patented technology, which enable paint and coatings formulators to meet or exceed the EPA guidelines for VOC content have been introduced in a technical data sheet. The high-solids resins fall within EPA guidelines of no more than 3.5 pounds of solvent per gallon of paint or coatings formulation. For more details on Styresol® 13-510, Styresol® 13-520, and Lustrasol® 13-511 high-solids resins, contact Billy K. Winters, Manager Coating Applications, Reichhold Chemicals, Inc., Chemical Coatings Div., P.O. Box 1433, Pensacola, FL 32596.

Filter Cartridges & Housings

A 28-page guide which aids in the selection of the correct filter cartridge and housing for an application is available. The guide, which lists over 330 chemical compounds, provides chemical compatibility with: 14 different filter media used in filter cartridges; five different materials used in cartridge center cores; eight different gasket or O-ring materials used for cartridge sealing; and nine different materials used for cartridge filter vessels. For a copy of "Chemical Compatibility Guide," write Filterite, 2033 Greenspring Dr., Timonium, MD 21093.

Superfine Grinding

Literature describes totally enclosed, pressurized grinding mills designed to produce superfine grinds down to submicron sizes in liquids and pastes. Applications include the processing of coatings, inks, pigment dispersions, graphite, plastics, dyes, magnetics, specialty food products, and a wide range of other materials. For more data on John Small Media Mills, write Netzsch Inc., Grinding & Dispersion Dept., 119 Pickering Way, Exton, PA 19341-1393.

Ink-Jet Coder System

Literature describes the redesign of a non-contact, ink-jet coder system making it more compact and easier to upgrade or expand. The new, compact design allows the system to fit into areas where the space is limited. For more details on the Telemark system, contact Charles Sellberg, Telemark Product Manager, Diagraph Corp., 13789 Rider Trail North, St. Louis/Earth City, MO 63045.

Measurement System

Literature describes a computer-assisted, electrochemical impedance measurement system. The system is based on the IBM Personal System/2™ Model 30 computer. Features include multiple plot formats, equivalent-circuit modeling aids, and automatic repetitive measurement capability. For particulars on the Model 378-1 Electrochemical Impedance System, contact Marc Rothstein, Marketing Manager, EG&G Princeton Applied Research, Princeton, NJ 08543.

Surfactants

An 18-page, four-color brochure details a full range of surfactants and specialty products for use in the personal care, textiles, agrichemicals, food, metalworking, and plastics industries. For a free copy of "ICI: Your One of a Kind Resource for Surfactants, Specialty Products and Innovations," contact Loretta Ianni, ICI Specialty Chemicals, Wilmington, DE 19897.

Polyester Intermediates

A four-page brochure which lists the structural features and performance characteristics of a line of polyester intermediates is available. For a copy of "Eastman® Polyester Intermediates and Their Performance Characteristics," Publication N-307, write Eastman Chemical Products, Inc., P.O. Box 431, Kingsport, TN 37662.

Mercury Porosimeter

A mercury porosimeter which provides data acquired from either scanning or stepwise (truly equilibrated) intrusion is highlighted in recently released literature. Any number of pressure points up to 250 in as many as 10 pressure tables may be selected. The instrument measures a range of pore openings from 360 μm diameter on the low pressure side to 0.003 μm diameter on the high pressure side. For details on the AutoPore II 9220, write Micromeritics, One Micromeritics Dr., Norcross, GA 30093-1877.

CLASSIFIED ADVERTISING

PIGMENT DISPERSION SPECIALIST

Join the laboratory group of a small division of Multi-National Chemical Company.

We are looking for a specialist in the dispersion of pigments (white, black, organics, silicas, and clays) to be responsible for developing new pigment dispersion products, improving existing plant process, and productivity improvements. It is expected you would be the coordinator for all colorant related activities of this business group, including the use of color computer methods for quality control and coloring matching, and selection and identification of improved raw materials and intermediates.

In this position, you would have substantial contact with our manufacturing facility. Practical knowledge of pigment dispersion grinding equipment is important. It is expected the qualified candidate will have at least 3 years of experience related to dispersing of pigment type materials and familiarity with laboratory bench work and scaling up into commercial manufacturing equipment.

As part of a major international chemical company, salary and fringe benefits would be fully competitive, and opportunities to grow within this business group or with many of the other business groups within the corporation should be excellent for an aggressive, highly motivated individual.

Please forward your resume along with salary history. Be assured of confidentiality. Reply to: JCT, Box 832, 1315 Walnut St., Philadelphia, PA 19107.

An Equal Opportunity Employer M/F.

* * * * *

SALES REP

"Florida Chemical Distributor" seeking experienced and technical oriented Sales Representative to sell raw materials to paint, inks, and allied industries. Will pay relocation expenses. Excellent compensation package and benefits. Send resume to: JCT, Box 800, 1315 Walnut St., Philadelphia, PA 19107.

Book Review

PARTICLE SIZE DISTRIBUTION

Edited by
Theodore Provder

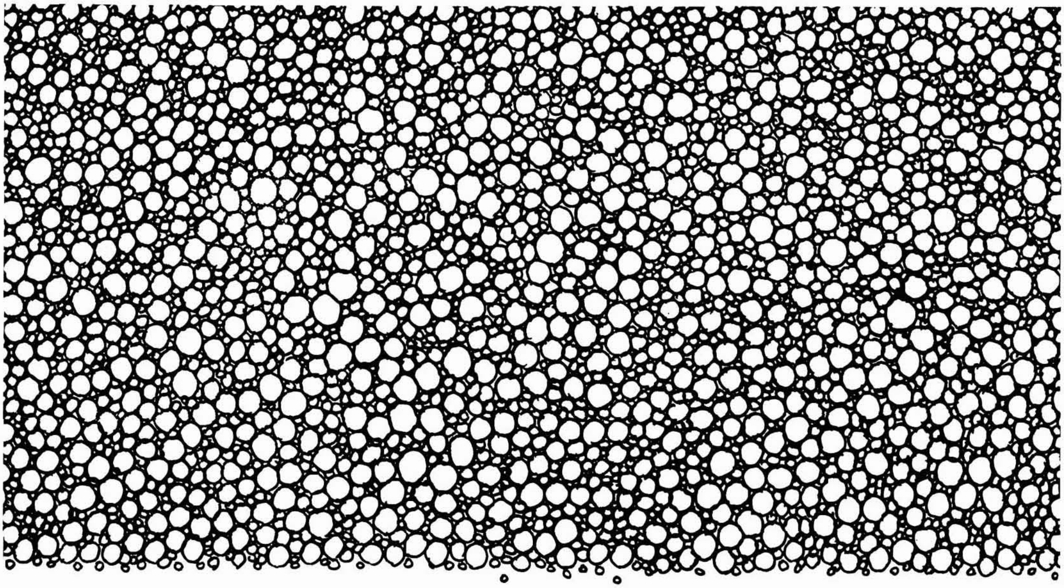
Published by
American Chemical Society
Washington, D.C. (1987)
$59.95, 308 pages

Reviewed by
Ray A. Dickie
Ford Motor Company
Dearborn, MI 48121

Particle size and size distribution analysis have, as the editor points out in his introduction to this excellent volume, undergone a renaissance over the past five years largely as a result of advances in electronics and computer technology. Instrumental methods, that until recently were confined to academic and large industrial research laboratories, have been transformed into relatively low cost, easy to use commercial instruments. The present volume describes recent advances in four areas of size distribution analysis: image analysis, photon correlation spectroscopy and other light scattering methods, disc centrifuge photosedimentometry and sedimentation field-flow fractionation, and column chromatography methods.

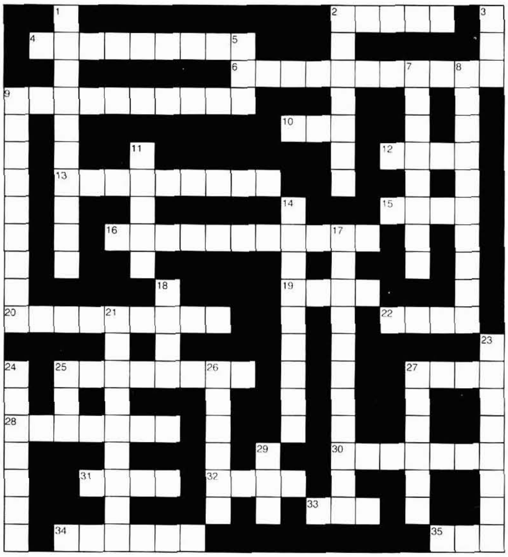
The book is based on a symposium sponsored by the American Chemical Society Division of Polymeric Materials Science and Engineering; the papers thus reflect current research activities. Greatest emphasis is placed on photon correlation/light scattering methods (nine of 20 chapters). Several chapters detail the hazards and pitfalls of various methods of size distribution analysis. The chapters describing limitations of photon correlation techniques should be especially valuable to researchers and technologists interested in this technique.

The editor has provided a brief preface which sets the individual chapters in context, and has provided references to recent overall reviews of the literature. The book could have been improved by the incorporation of a longer introductory chapter outlining the development of size distribution techniques and identifying current critical issues. Even without such an introductory treatment, the book will be a valuable reference source on current research in particle size and size distribution analysis.



CroSSLinkS

by Earl Hill



Solution
to be published
in
November issue

No. 20

ACROSS

2. Optical effect pattern, M____
4. Water surface tension modifier, A____
6. Reddish-blue vat dye pigment
9. Small voids
10. Non ____ ____ (Abr.)
12. Sp. Gr. balance
13. Black pigment
15. Scale for hardness testing
16. Interference color
19. A material joining place
20. Micaceous silica mineral
22. Size grading of natural resins
25. Aqueous enemy of paint
27. Close relative of 20 across
28. Plain wallpaper edge
30. Hydrocarbon solvent, N____
31. Admixture with white pigment
32. Common extender pigment
33. Aliphatic ketone (Abr.)
34. Unusable resin, heat generated*
35. Theory of particle light scattering

DOWN

1. Cluster of pigment particles
2. Test for coating flexibility
3. Color attribute
5. "Near" (Org. Chem. Prefix)
7. Science of flow
8. Blue-black aniline dye (Pl.)
9. Chemical element, light metal
11. Printing ink institute (Abr.)
14. Paper used by the "daily rag"
17. Film formation, H_2O base
18. Painter's m____
21. To be attacked by oxygen
23. White deposit on fresh concrete
24. Mason's art
25. Small metric length unit
26. To thin, as a varnish
27. M____L alcohol
29. Slip additive

*A most unusual word to be in the Paint/Coatings Dictionary. Probably known mainly to senior citizen paint chemists.

Coming Events

FEDERATION MEETINGS

For information on FSCT meetings, contact FSCT, 1315 Walnut St., Philadelphia, PA 19107 (215-545-1506).

1987

(Oct. 5-7)—65th Annual Meeting and 52nd Paint Industries' Show. Convention Center, Dallas, TX.

1988

(May 17-20)—Federation "Spring Week." Seminar on the 17th and 18th; FSCT Society Officers Meeting on the 19th; FSCT Board of Directors Meeting on the 20th. Orlando Marriott Hotel, Orlando, FL.

(Oct. 19-21)—66th Annual Meeting and 53rd Paint Industries' Show. McCormick Place, Chicago, IL.

1989

(Nov. 8-10)—67th Annual Meeting and 54th Paint Industries' Show. Rivergate, New Orleans, LA.

SPECIAL SOCIETY MEETINGS

1988

(Feb. 3-5)—Southern Society. 15th Annual Water-Borne and Higher-Solids Coatings Symposium. New Orleans, LA. (Dr. Gordon L. Nelson, Chairman, Dept. of Polymer Science, University of Southern Mississippi, Southern Station Box 10076, Hattiesburg, MS 39406-0076).

(Mar. 16-18)—Southwestern Paint Convention of Dallas and Houston Societies. Hyatt Regency Hotel. Houston, TX. (J. Harrell, Buckman Labs., 5127 Wightman Ct., Houston, TX 77069).

(Apr. 13-15)—Southern Society. Annual Meeting. Omni Hotel, Charleston, SC. (Scott McKenzie, Southern Coatings Co., P.O. Box 160, Sumter, SC 29150).

(Apr. 28-30)—Pacific Northwest Society. Annual Symposium. Vancouver, B.C., Canada. (Yvon Poitras, General Paint Corp., 950 Raymur Ave., Vancouver, B.C., Canada V6A 3L5).

(May 11-12)—New England Society. 1988 Tech Expo. Sheraton Boxboro Hotel, Boxboro, MA. (G. Pollano, Polyvinyl Chemicals, 730 Main St., Wilmington, MA 01887).

1989

(Mar. 13-15)—Western Coatings Societies Symposium and Show. Disneyland Hotel, Anaheim, CA. (Andy Ellis, NL Industries, Inc., 200 N. Berry St., Brea, CA 92621).

(Apr. 5-7)—Southern Society. Annual Meeting. Hyatt Regency Westshore, Tampa, FL.

OTHER ORGANIZATIONS

1987

(Oct. 14-16)—SURTEC '87 Berlin. International Congress Center, Berlin. (Gabriela Thal, 1625 K St., N.W., Suite 500, Washington DC 20006).

(Oct. 15-16)—"The Fundamentals of Color" Seminar sponsored by Macbeth, a Division of Kollmorgen Corp. San Jose, CA. (Jeanne Dolan or Karen Degnan, Macbeth, Little Britain Rd., P.O. Box 230, Newburgh, NY 12550-0382).

(Oct. 19-20)—"The Fundamentals of Color" Seminar sponsored by Macbeth, a Division of Kollmorgen Corp. Grand Rapids, MI. (Jeanne Dolan or Karen Degnan, Macbeth, Little Britain Rd., P.O. Box 230, Newburgh, NY 12550-0382).

(Oct. 19-21)—"Fundamentals of Adhesion: Theory, Practice and Applications." Course sponsored by the State University of New York, West Point, NY. (A.V. Patsis, Chemistry Dept., State University of New York, New Paltz, NY 12561).

(Oct. 19-23)—"Corrosion Control: Design Parameters for Coatings." Course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(Oct. 19-23)—15th Introductory—"Paint Formulation." Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Coatings and Polymer Science Program, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401-0249).

(Oct. 20-21)—Industrial Color Technology Seminar sponsored by the Educational Service Division, Applied Color Systems, Inc. Lancaster, PA. (Bobbie Deel, Applied Color Systems, Inc., 2848-M Carolina Center, Highway I-85, Charlotte, NC 28208).

(Oct. 20-22)—"Process Hazards Management" seminar sponsored by the Du Pont Co., New Orleans, LA. (Du Pont Safety Services, Barley Mill Plaza, P19-1104, Wilmington, DE 19898).

(Oct. 20-22)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Stamford, CT. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Oct. 22-23)—60th Anniversary Conference of Japan Society of Colour Material, Tokyo, Japan. (Japan Society of Colour Material, 9-12, 2-chrome, Iwamoto-cho, Chiyoda-ku, Tokyo 101, Japan).

(Oct. 26-28)—National Paint and Coatings Association 100th Anniversary Annual Meeting. Washington Hilton, Washington, D.C. (Karen Bradley, NPCA, 1500 Rhode Island Ave., NW, Washington, D.C. 20005).

(Oct. 26-28)—Paint Research Association's 7th International Conference on Water-Borne Coatings. Penta Hotel, Heathrow Airport, London, England. (PRA, Waldegrave Rd., Teddington, Middlesex TW11 8LD, England).

(Oct. 26-28)—"Principles of High Performance Composites." Course sponsored by the State University of New York, West Point, NY. (A.V. Patsis, Chemistry Dept., State University of New York, New Paltz, NY 12561).

(Oct. 26-29)—"Introduction to Coatings Technology." Course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(Oct. 27-28)—Industrial Color Technology Seminar sponsored by the Educational Service Division, Applied Color Systems, Inc. Detroit, MI. (Bobbie Deel, Applied Color Systems, Inc., 2848-M Carolina Center, Highway I-85, Charlotte, NC 28208).

(Oct. 27-28)—Paint Volatile Organic Compounds (VOC) Workshop sponsored by ASTM Standards Technology Training. Philadelphia, PA. (Julie Shelton, ASTM, 1916 Race St., Philadelphia, PA 19103).

(Oct. 27-29)—Managing Environmental Risks Conference sponsored by The Association Dedicated to Air Pollution Control and Hazardous Waste Management. Capitol Holiday Inn, Washington, D.C. (Barbara Yunk, Meetings Dept., APCA, P.O. Box 2861, Pittsburgh, PA 15230).

(Oct. 27-29)—"Measurement of Appearance" Workshop sponsored by HunterLab. Sheraton Hotel, Tysons Corner, VA. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Oct. 27-29)—"Industrial Painting Processes" Program sponsored by the Society of Manufacturing Engineers. Doubletree Hotel, Anaheim, CA. (Jim Warren, SME, Special Programs Div., One SME Dr., P.O. Box 930, Dearborn, MI 48121).

(Oct. 28-29)—Tenth Resins and Pigments Exhibition, Penta Hotel, London Heathrow Airport, England. (Polymers Paint Colour Journal, Queensway House, 2 Queensway, Redhill, Surrey RH1 1QS, England).

(Oct. 28-30)—"Electroactive Polymers: Materials, Properties and Applications of Electrically Conductive, Ionically Conductive and Redox Polymer Systems." Course sponsored by the State University of New York. West Point, NY. (A.V. Patsis, Chemistry Dept., State University of New York, New Paltz, NY 12561).

(Nov. 2-5)—38th Steel Structures Painting Council Conference and Protective Coatings Exposition, Sheraton World Hotel, Orlando, FL. (SSPC, 4400 Fifth Ave., Pittsburgh, PA 15213-2683).

(Nov. 2-6)—"Introduction to Polymer Chemistry." Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Coatings and Polymer Science Program, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401-0249).

(Nov. 3-5)—"Fundamentals of RCRA" Seminar sponsored by the Du Pont Co. Houston, TX. (Du Pont Co., P19-1105, P.O. Box 4500, Greenville, DE 19807).

(Nov. 4-5)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Cincinnati, OH. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Nov. 4-5)—"Painting Processes: Industrial Paint Application Technology." Course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(Nov. 7-11)—10th International Congress on Metallic Corrosion sponsored by Central Electrochemical Research Institute on behalf of International Corrosion Council. Madras, India. (Dr. V.I. Vasu, Chairman, ICMC Organizing Committee, Director CERI, Karaikudi 623006, Tamil Nadu, India).

(Nov. 9-10)—"Advances in Polyurethanes" Seminar sponsored by Technomic Publishing Co., Inc., Atlanta, GA. (Program Div., Technomic Publishing Co., Inc., 851 New Holland Ave., Box 3535, Lancaster, PA 17604.)

(Nov. 11-12)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Richmond, VA. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Nov. 16)—"Spray Applications for Furniture Finishing" Program sponsored by the Society of Manufacturing Engineers. Holiday Inn-High Point, High Point, NC. (Jim Warren, SME, Special Programs Div., One SME Dr., P.O. Box 930, Dearborn, MI 48121).

(Nov. 16-17)—"The Fundamentals of Color" Seminar sponsored by Macbeth, a Division of Kollmorgen Corp. Cherry Hill, NJ. (Jeanne Dolan or Karen Degnan, Macbeth, Little Britain Rd., P.O. Box 230, Newburgh, NY 12550-0382).

(Nov. 16-18)—Special Conference on Corrosion sponsored by the South Central Region of the National Association of Corrosion Engineers. Lafayette Hilton and Towers, Lafayette, LA. (NACE, P.O. Box 218340, Houston, TX 77218).

(Nov. 16-21)—SF China. The 1988 International Surface Finishing & Coatings Exhibition. Shanghai Exhibition Center, Shanghai, P.R., China. (Sino Trade Promotions, 15/F, Flat A, Wing Cheong Commercial Bldg., 19-25 Jervois St., Central, Hong Kong).

(Nov. 17-19)—"Spray Applications Workshop for Furniture Finishing" sponsored by the Society of Manufacturing Engineers. Holiday Inn-High Point, High Point, NC. (Jim Warren, SME, Special Programs Div., One SME Dr., P.O. Box 930, Dearborn, MI 48121).

(Nov. 18-19)—Industrial Color Technology Seminar sponsored by the Educational Service Division, Applied Color Systems, Inc. Atlanta, GA. (Bobbie Deel, Applied Color Systems, Inc., 2848-M Carolina Center, Highway I-85, Charlotte, NC 28208).

(Nov. 18-20)—"Finishing Automotive Plastics" Clinic sponsored by The Association for Finishing Processes of SME. Hyatt Regency Dearborn, Dearborn, MI. (Diane Korona, SME, Special Programs Div., One SME Dr., P.O. Box 930, Dearborn, MI 48121).

(Nov. 19-20)—"The Fundamentals of Color" Seminar sponsored by Macbeth, a Division of Kollmorgen Corp. Hempstead, NY. (Jeanne Dolan or Karen Degnan, Macbeth, Little Britain Rd., P.O. Box 230, Newburgh, NY 12550-0382).

(Nov. 20-22)—40th Annual Show and Convention of National Decorating Products Assn., McCormick Place, Chicago, IL. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Nov. 23-27)—Pacific Corrosion '87—Fifth Asian-Pacific Corrosion Control Conference in conjunction with the 27th Australasian Corrosion Association Conference. Hilton Hotel, Melbourne, Australia. (Bloomsbury Conference Services, P.O. Box 2368, Richmond, 3121, Australia).

(Dec. 2-3)—Industrial Color Technology Seminar sponsored by the Educational Service Division, Applied Color Systems, Inc., San Francisco, CA. (Bobbie Deel, Applied Color Systems, Inc., 2848-M Carolina Center, Highway I-85, Charlotte, NC 28208).

(Dec. 7-8)—Regional "Color and Appearance" Seminar sponsored by HunterLab. San Francisco, CA. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Dec. 7-11)—"Fundamentals of Chromatographic Analysis." Course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242).

(Dec. 10-11)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Los Angeles, CA. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

1988

(Jan. 20-21)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Atlanta, GA. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Jan. 24-27)—ASTM Committee D-1. Holiday Inn, Clearwater Beach, FL. (Brent Backus, ASTM, 1916 Race St., Philadelphia, PA 19103).

(Jan. 27-28)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Seattle, WA. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Feb. 12-14)—Southern Decorating Products Show sponsored by the National Decorating Products Association. Georgia World Congress Center, Atlanta, GA. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

(Feb. 22-24)—"Maintenance Coatings for Contractors and Paint Inspectors." Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Coatings and Polymer Science Program, Dept. of Chemistry, UMR, Rolla, MO 65401-0249).

(Feb. 25-26)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Stouffer Resorts Hotel, Orlando, FL. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Feb. 29-Mar. 2)—Finishing Technologies '88 sponsored by Coatings Magazine. Constellation Hotel, Toronto, Ont., Canada. (CM, 86 Wilson St., Oakville, Ont., Canada L6K 3G5).

(Feb. 29-Mar. 4)—"56th Introductory Short Course—The Basic Composition of Coatings." Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Coatings and Polymer Science Program, Dept. of Chemistry, UMR, Rolla, MO 65401-0249).

(Mar. 5-6)—Canadian Decorating Products Show sponsored by the National Decorating Products Association. Constellation Hotel, Toronto, Ont., Canada. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

(Mar. 14-18)—"16th Introductory—Paint Formulation." Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Coatings and Polymer Science Program, Dept. of Chemistry, UMR, Rolla, MO 65401-0249).

(Mar. 16-17)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Dallas, TX. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Mar. 21-25)—Corrosion/'88 sponsored by the National Association of Corrosion Engineers. Cervantes Convention & Exhibition Center, St. Louis, MO. (Allison Mayer, NACE Exhibits Dept., P.O. Box 218340, Houston, TX 77218).

(Mar. 26-27)—Western Decorating Products Show sponsored by the National Decorating Products Association. Brooks Hall, San Francisco, CA. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

(Mar. 30-31)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Denver, CO. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Apr. 5-7)—Electrocoat '88. Drawbridge Inn and Convention Center, Ft. Mitchell, KY. (Products Finishing, 6600 Clough Pike, Cincinnati, OH 45244).

(Apr. 9-10)—Eastern Decorating Products Show sponsored by the National Decorating Products Association. World Trade Center, Boston, MA. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

(Apr. 13-14)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Cleveland, OH. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Apr. 18-20)—"Conformal Coatings." Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Coatings and Polymer Science Program, Dept. of Chemistry, UMR, Rolla, MO 65401-0249).

(Apr. 20-21)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Minneapolis, MN. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(Apr. 20-27)—Surface Treatment '88 at Hannover Fair Industry '88. Hannover Fairgrounds, Hannover, W. Germany. (Joachim Schafer, Hannover Fairs USA Inc., 103 Carnegie Center, P.O. Box 7066, Princeton, NJ 08540).

(Apr. 24-28)—RadTech '88. New Orleans Fairmont Hotel, New Orleans, LA. (John Waxman, Director of Conferences and Exhibits, RadTech International, 60 Revere Dr., Suite 500, Northbrook, IL 60062).

(May 2-4)—International Seminar sponsored by the Ford Motor Co. Dearborn, MI. (A. Golovoy, Ford Motor Co., Box 2053/SRL 3198, Dearborn, MI 48121).

(May 4-5)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Boston, MA. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(May 9-11)—Federation of Scandinavian Paint and Varnish Technologists. 12th Congress, Helsinki, Finland. (Arja Saloranta, Tikkurila Oy, PB 53, SF 01301 Vanda, Finland).

(May 16-20)—"Physical Testing of Paints and Coatings." Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Coatings and Polymer Science Program, Dept. of Chemistry, UMR, Rolla, MO 65401-0249).

(May 18-20)—"Measurement of Appearance" Workshop sponsored by HunterLab. Sheraton Hotel, Tysons Corner, VA. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(May 24-25)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Charlotte, NC. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(June 1-2)—Regional "Color and Appearance" Seminar sponsored by HunterLab. Cherry Hill, NJ. (Hunter Associates Laboratory, Inc., 11491 Sunset Hills Rd., Reston, VA 22090).

(June 5-11)—American Chemical Society. 195th National Meeting and Third Chemical Congress of North America. Toronto, Ont., Canada. (B.R. Hodson, ACS, 1155—16th St. NW, Washington, D.C. 20036).

(June 13-17)—International Conference on Composite Interfaces II. Case Western Reserve University, Cleveland, OH. (Professor H. Ishida, General Chairman, ICCI-II, Dept. of Macromolecular Science, Case Western Reserve University, 10900 Euclid Ave., Cleveland, OH 44106-1727).

(June 15-16)—Surfex '88. Oil and Colour Chemists' Association. Harrogate International Conference Center, Yorkshire, England. (R.H. Hamblin, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex HA0 2SF England).

(Sept. 18-24)—XIXth Congress of FATIPEC. Aachen, Germany. (C. Bourgery, FATIPEC Secretary General, 76 Blvd. Pereire, 75017 Paris, France).

(Sept. 25-30)—"Polymers in Information Storage Technology" Symposium sponsored by the American Chemical Society. Los Angeles, CA. (K.L. Mittal, IBM-Corporate Technical Inst., 500 Columbus Ave., Thornwood, NY 10594).

(Oct. 2-4)—Canadian Paint and Coatings Association. 76th Annual Convention. Ottawa, Ontario. (CPCA, 515 St. Catherine St. W., Montreal, Que. H3B 1B4 Canada).

(Oct. 10-13)—15th International Naval Stores Meeting. Intercontinental Ritz and Meridien Hotels, Lisbon, Portugal. (Manco L. Snapp, Jr., Arizona Chemical Co., Panama City, FL).

(Oct. 18-21)—12th World Congress on Metal Finishing, INTERFINISH 88. Palais des Congres, Paris, France. (SEPIC INTERFINISH, 17 rue d'Uzes, 75002 Paris, France).

(Nov. 4-6)—41st Annual Show and Convention of National Decorating Products Association. McCormick Place, Chicago, IL. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

(Nov. 7-9)—Paint Research Association. Eighth International Conference. Amsterdam, The Netherlands. (Mr. Dip Dasgupta, Head of Information Dept., Paint RA, Waldegrave Rd., Teddington, Middlesex TW11 8LD England).

(Dec. 3-9)—Chemtech China '88. China International Exhibition Centre, Beijing, China. (SHK International Services Ltd., 22/F., 151 Gloucester Rd., Hong Kong).

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(Apr. 8-9)—Eastern Decorating Products Show sponsored by the National Decorating Products Association. World Trade Center, Boston, MA. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

(Apr. 11-14)—"Advances in Corrosion Protection by Organic Coatings" Symposium. Christ's College Cambridge, England. (David Scantlebury, Corrosion and Protection Centre, UMIST, P.O. Box 88, Manchester, U.K. or Martin W. Kendig, Rockwell International Science Center, Thousand Oaks, CA 91360).

(June 21-24)—Oil and Colour Chemists' Association Biennial Conference. Grosvenor Hotel, Chester, England. (Mr. Christopher Lacey-Day, Director, OCCA, Priory House, 967 Harrow Rd., Wembly, Middlesex HA0 2SF, England).

(Sept. 25-30)—American Chemical Society. 196th National Meeting. Los Angeles, CA. (B.R. Hodson, ACS, 1155—16th St. NW, Washington, D.C. 20036).

(Nov. 18-20)—National Decorating Products Show sponsored by the National Decorating Products Association. McCormick Place, Chicago, IL. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

Advertisers Index

'Humbug' from Hillman

He had just graduated from the school for advanced training at the FBI. He was overwhelmed with the excitement of having achieved his goal in the field of espionage with all its secrecy and high drama.

And now, here he was in the office of the Director, about to start on his first secret assignment. The instructions were simple but impressive. Caution and secrecy were of prime importance. All action was to be unobtrusive and names used were not to be memorable.

"Travel to New York City via Amtrak. No plane reservations. A quiet arrival. Take the subway downtown and walk to 114 Greene St.—a fairly large apartment house. Go to the apartment of Jones. To establish your identity, repeat the code phrase, "The sun is down, the moon is up." You will be given an envelope. Return here immediately."

He followed the instructions faithfully. However, when he arrived at the house, he found that there were three Joneses listed on the directory. Nonplussed for a moment, he then realized that the code phrase would identify the proper Jones as well as himself. He went to the door marked Jones on the ground floor and rang the bell. The door opened slowly and he was surprised to see a little old lady peering up at him. He quietly but distinctly spoke the phrase, "The sun is down, the moon is up."

The little old lady looked at him quizzically, hesitated, and then smiled brightly, "Oh, you must want Jones the spy. Apartment 3S—third floor, rear."

Family Incident

Some months ago, reliable and respected Delta Airlines was cited with several unfortunate incidents of pilot error which received wide publicity. During that time, my son, Donald, who is a nervous but frequent flyer, had occasion to fly from Miami to Boston.

As he settled into his first class (by virtue of his frequent flying) seat, he noted that his neighbor was a pleasant looking, middle-aged lady, who smiled cordially in acknowledgment to his greeting. As the time for take-off approached, Don, nervously repeating a current joke, observed that on Delta he was a "frequent survivor." The lady smiled and giggled a little. Encouraged, he remarked as they roared aloft that he hoped, in light of the recent incidents of Delta landings at wrong airports or runways and being lost over the ocean, that the pilot would safely find his way to Logan Airport.

"Oh," the pleasant lady questioned, "have they had problems at Delta?" Somewhat surprised by her ignorance of the incidents, Don confirmed the news but hastened to assure her (and perhaps, himself) that he was certain that the pilot would stay alert and manage to find the airport. The lady smiled.

Just then the cockpit door opened and the Captain emerged to greet the passengers up front. He bent down toward the pleasant little lady. "Hi Mom," he said and kissed her.

Jay Adams thought that those who are interested in medical affairs would find this quote apropos from a *Wall Street Journal* article by Bridget O'Brian.

"Those who complain about indecipherable prescriptions ought to consider what doctors' writing is like when it's legible:

"Sudden death, though fortunately it is rare, is frequent," wrote one physician in the *British Medical Journal.* A colleague outdid even that in the *U.S. Journal of Medicine:* "All seven of the patients who died never completely recovered."

Ralph H. Huff, envious of the faltering erudition of Sal Sanfilippo, joins our Department of Obscure Recipes with the following from the *Abstract Review* of December 1930. Humbug believes that anyone who reads *Abstract Reviews* from 1930 should get credit in ridiculous columns such as this and/or should be carefully watched by a full time attendant.

Dr. Barbara Lauren, who has been highly touted by Humbug for her distinguished legal research, supplies the following from "Murphy's Law, Book Two":

(1) If you are given an open book exam, you will forget your book.

Corollary: If you are given a take home exam, you will forget where you live.

(2) *Law of Life's Highway*: If everything is coming your way, you're in the wrong lane.

(3) *Femo's Law of Automotive Engineering*: If you drop something, it will never reach the ground.

(4) *First Law of Kitchen Confusion*: Multiple function kitchen gadgets will not perform any function adequately.

(5) *Van Roy's Law*: An unbreakable toy is useful for breaking other toys.

(6) *Farmers' Credo*: Sow your wild oats on Saturday night and on Sunday pray for crop failure.

(7) *Ruby's Principle of False Encounters*: The probability of meeting someone you know increases with someone you don't want to be seen with.

(8) *Benedict's Principle*: Nature always sides with the hidden flaw.

—Herb Hillman
Humbug's Nest
P.O. Box 135
Whitingham, VT 05361

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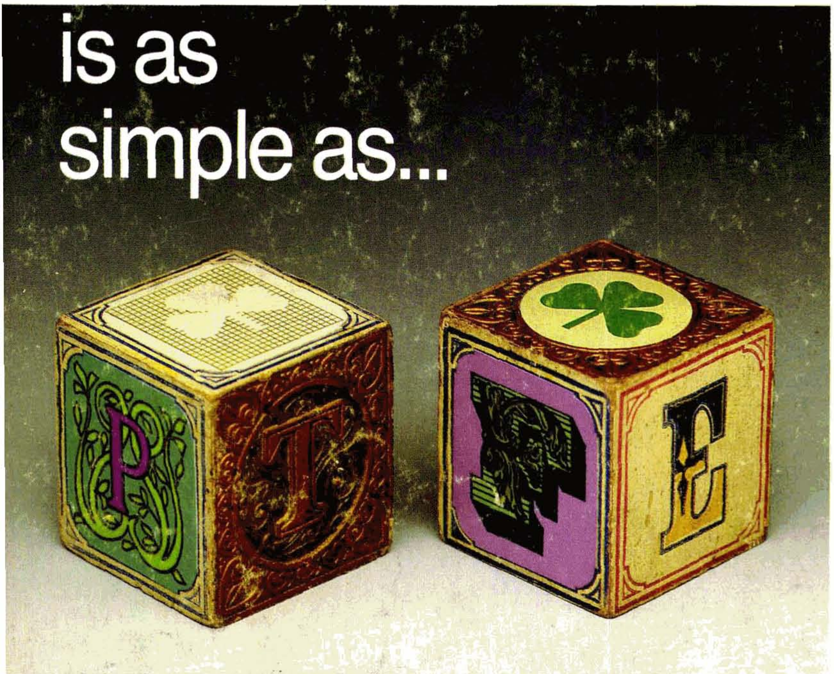

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When you need PTFE in your formulation, talk with our coatings-experienced staff. They'll make a recommendation, send you samples and data.

Coatings Application	Desired Performance Qualities	PTFE Product
Container, solvents	can mobility and spin-neck abrasion resistance	SST-3D
Container, water	can mobility and spin-neck abrasion resistance	SST-3H
Ultra-thin clears	abrasion resistance with clarity retention	SST-1MG
Coil, protective	corrosion inhibition and falling sand resistance	SST-2SP5
Coil, decorative	taber abrasion and metal-mark resistance	FluoroSLIP 515
Powder coating	durability	FluoroSLIP 525
General industrial	fabrication protection	SST-2
Printing inks	rub-resistance	SST-3
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