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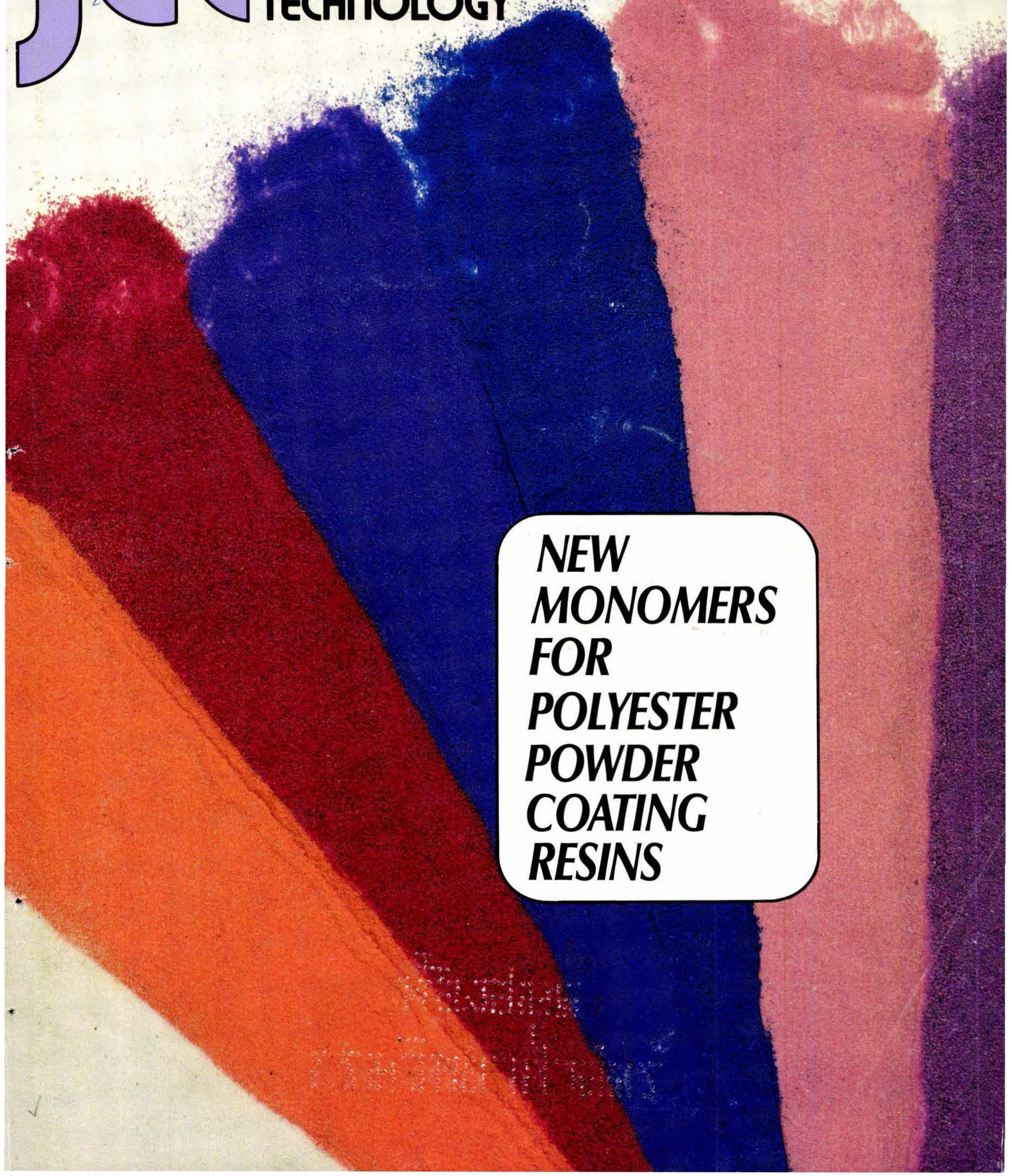

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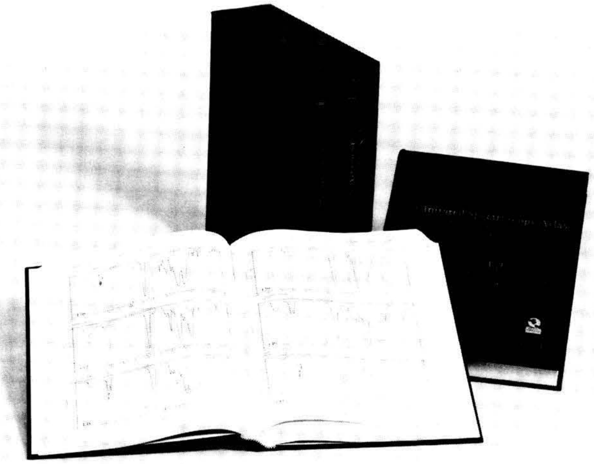

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VOLUME 65 NUMBER 826

## Technical Articles

## Federation News

## Departments

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jct 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 submission of original research papers, review papers, and papers under the special headings *Open Forum* and *Back to Basics*, and *Letters to the Editor*. All manuscripts will be assumed to be previously unpublished writing of the authors, not under consideration for publication elsewhere. When review papers contain tables or graphs from copyrighted articles, the authors will be required to obtain permission for use from the copyright holders. When the organization with which the authors are affiliated requires clearance of publications, authors are expected to obtain such clearance before submission of the manuscript. 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. 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 regional meetings or symposia of the Constituent Societies.

***Papers in which proprietary products or processes are promoted for commercial purposes are specifically nonacceptable for publication.***

## SUBMISSION OF MANUSCRIPTS...

### ...for the Journal

Four complete copies should be sent to the Editor, Journal of Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422. The cover letter should address copyright, clearance, and release issues discussed above and should specify paper category: *Original Research, Reviews, Open Forum*, or *Back to Basics*.

*Letters 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 previously.

### ...for Roon Foundation Award Competition

Ten complete copies of the manuscript are required, and should be submitted to the Chairman of the 1994 Roon Awards Committee, N. Bradford Brakke, Lilly Industries, Inc., 521 W. McCarthy St., Indianapolis, IN 46225. (For complete details, see "Roon Awards" section of the Journal in January 1993 issue.)

## 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).

Authors are encouraged to consider submissions in several categories and to prepare their manuscripts accordingly. The categories are:

*Original Research Papers:* The main technical content of the Journal of Coatings Technology will continue to be original research papers. Editors support the trend in scientific writing to a direct, less formal style that permits limited use of personal pronouns to avoid repititious or awkward use of passive voice.

*Review Papers*: Papers that organize and compare data from numerous sources to provide new insights and unified concepts are solicted. Reviews that show how advances from other fields can beneficially be applied to coatings are also desired. Reviews that consist mainly of computer searches with little attempt to integrate or critically evaluate are not solicited.

*Open Forum:* Topics for this category may be nontechnical in nature, dealing with any aspect of the coatings industry. The subject may be approached informally. Editors encourage submission of manuscripts that constructively address industry prolbems and their solutions.

*Back to Basics:* Papers that provide useful guides to Federation members in carrying out their work are solicited. Topics in this category are technical but focus on the "how to" of coatings technology. Useful calculations for coatings formulation and procedures that make a paint test more reproducible are examples of suitable topics. Process and production topics, i.e., paint manufacture, will also be reviewed in the *Back to Basics* category.

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 originally composed for oral presentation will have to be revised or rewritten by the author to conform to the style described in this guide.

Manuscripts should be typed with double spacing on one side of $8^1/2$ x 11 inch (22 x 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 x 7 inch (13 x 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.

## Text

Main headings and sub-headings should be used to improve readability, and to break up typographical monotony. The text should *not* be presented as an alphanumeric outline.

The main headings usually should be INTRODUCTION, EXPERIMENTAL, RESULTS AND DISCUSSION, and SUMMARY or CONCLUSIONS. Sub-headings will be specific to the subject.

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 afterwords 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 nonphotographic blue-lined 8$^1/2$ x 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 from original for typesetting.

*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 x 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. For oligomeric or polymeric materials, characteristics such as molecular weight, polydispersity, functional group content, etc. should be provided.

## Equations

Equations 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).

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## In Thanks . . .

As we near the end of November, most of us in the United States turn our thoughts to the upcoming Holidays, the first being Thanksgiving. During this time we give thanks for our blessings, which vary from person to person. Reasons for being thankful run the gamut from family to food to clothing to health, and include many other personal thoughts.

Most of what we are thankful for affects our lives in one way or another, and these things are usually tangible. However, among the Federation family there is a group that functions almost anonymously, doing many of the things necessary to keep the organization going. This group is composed of the members who volunteer their valuable time to serve on the FSCT's many committees.

For example, those of you who attended the 1993 Annual Meeting and Paint Industries' Show in Atlanta have the efforts of the Program Committee, the Host Committee, the Show Committee, and many other committees in unison to thank for the event. These committees operate behind the scenes to ensure a successful event.

The same is true for Spring Week, the awards programs, and any special functions held during the year. In addition, several FSCT Committees assist the leaders of the Constituent Societies by providing training in committee and Society management. The list is almost endless.

In a nutshell, virtually every aspect of the Federation is a result of the work of one or more committees.

This year, when you give thanks for your many blessings, don't forget the FSCT members which give of their time to keep the Federation moving along. The committee members of the organization will appreciate the thought.

Michael G. Bell

Michael G. Bell
Director of Educational Services

# Abstracts of Papers in This Issue

*(Translations provided by: French—Montreal Society Member Alain Brisson, of Hoechst Canada, Inc. and Spanish—Cesar Fuentes, of Instituto Mexicano de Tecnicos en Pinturas y Tintas.)*

### New Monomers for Polyester Powder Coating Resins—L.K. Johnson and W.T. Sade

JCT, 65, No. 826, 19 (Nov. 1993)

An increased emphasis upon the quality of compliant coatings has placed more stringent demands upon formulators of polyester powder coating resins. The ability of formulators to design higher performance resins is limited by the number of useful monomers that are available. When new monomers are considered for powder coating resins, attention is immediately focused upon the effects they impart to the glass transition temperature, $T_g$, and the melt viscosity, as key indicators of powder coating stability and flow during cure. This paper addresses these important variables for a number of glycols and diacids recently introduced for use in polyester powder coating resins.

### Polymetallosiloxane Coatings Derived From Two-Step, Acid-Base Catalyzed Sol Precursors for Corrosion Protection of Aluminum Substrates—T. Sugama, J.R. Fair, and A.P. Reed

JCT, 65, No. 826, 27 (Nov. 1993)

Fractal polymetallosiloxane (PMS) cluster films were fabricated on aluminum (Al) substrates by the hydrolysis-polycondensation-pyrolysis route of two-step, acid-base-catalyzed sol precursors consisting of N-[3-(triethoxysilyl)propyl]-4,5-dihydroimidazole, metal alkoxides, water, and methyl alcohol. Thin PMS coating films prepared by dipping Al into sol solution provided significant protection for Al against NaCl-induced corrosion. The important factors governing a good protective performance of PMS were as follows: (1) an increase in metal-O-Si linkages with increased treatment temperature; (2) a low degree of water wettability and spreadability; and (3) a specific microstructure geometry, in which the cluster units consisting of symmetrically grown fractal polymer branches were linked together. The third factor contributed to a decrease in stress generated by the shrinkage of films, thereby eliminating crazing and peeling of the film.

### Nouveaux Monomères pour les Résines de Polyester de Revêtements en Poudre—L.K. Johnson and W.T. Sade

JCT, 65, No. 826, 19 (Nov. 1993)

Une demande accrue de la qualité des revêtements "vert" a entrainé des demandes plus sévères des formulateurs de résines de polyester pour les revêtements en poudre. La capacité des formulateurs à produire des résines haute performance est limitée par le nombre de monomères disponibles. Lorsque de nouveaux monomères sont étudiés pour les résines de revêtements en poudre, l´attention est immédiatement dirigée vers les effets qu´ils apportent à la température de transition vitreuse, $T_g$, ainsi que la viscosité de fusion, comme indicateurs clés de la stabilité du revêtement en poudre et de la viscosité durant le durcissement. Cette publication discute de ces variables importantes pour un certain nombre de glycols et diacides introduits récemment pour utilisation dans les résines de polyesters de revêtements en poudre.

### Revêtements de Polymétallosiloxane Provenant d´une Réaction en Deux Étapes de Précurseurs Sol Acide-Base Catalysés pour la Protection Contre la Corrosion de Supports d´Aluminium—T. Sugama, J.R. Fair, and A.P. Reed

JCT, 65, No. 826, 27 (Nov. 1993)

Des groupes de pellicules polymétallosiloxane (PMS) ont éte fabriqués sur des supports d´aluminium par un procédé en deux étapes d´hydrolyse-polycondensation-pyrolse, de précurseurs sol acide-base catalysés consistant en N-[3(triéthoxysilyl)propyl]-4,5-dihydroimidazole, alkoxide métalliques, eau et alcool méthylique. De minces pellicules de PMS préparées par immersion des supports d´aluminium dans la solution sol, a donné une protection significative contre la corrosion induite par le NaCl. Les facteurs importants pour une bonne protection du PMS sont les suivants: (1) une augmentation des liens métal-o-si avec une augmentation de température; (2) un bas degré de mouillage et de diffusion de l´eau; et 3) une géométrie microstructurelle spécifique, où les unités des groupes consistent en branches de polymères symétriques liées entre elles. Le troisième facteur a contribué à la diminution du stress généré par la contraction des pellicules, éliminant ainsi le fendillement et l´écaillage de la pellicule.

### Nuevos Monomeros Para Resinas Poliester de Recubrimientos en Polvo—L.K. Johnson and W.T. Sade

JCT, 65, No. 826, 19 (Nov. 1993)

Un aumento en el enfasis sobre la calidad del recubrimiento tiene una demanda mas estricta para los formuladores de resinas poliester para recubrimientos en polvo. La habilidad de los formuladores para diseñar resinas de alto desempeño es limitado por el número de monomeros que estan disponibles. Cuando se consideran nuevos monomeros para las resinas de recubrimientos en polvo, su atención es inmediatamente dirigida a los efectos que imparten a la temperatura de transición vitrea, $T_g$, y su viscosidad al fundirse como un indicador clave de la estabilidad del recubrimiento en polvo y su fluidez durante el curado. Este documento menciona estas importantes variables para un número de glicoles y diácidos recientemente introducidos para su uso en resinas poliéster de pinturas en polvo.

### Recubrimentos Polimetalosiloxano Derivados de Dos Pasos, Precursores Sol Acido-Base Catalizados Para Proteccion de la Corrosion de Sustratos de Aluminio—T. Sugama, J.R. Fair, and A.P. Reed

JCT, 65, No. 826, 27 (Nov. 1993)

Grupos de películas de polimetalosiloxane (PMS) fueron fabricados en sustratos de aluminio (Al) por la ruta de dos pasos hidrólisis-policondensación-pirólisis de dos pasos, precursores “sol” acido-base catalizados consistentes de N-(3-(trietoxisilil) propil)-4,5-dihidromidazol, metales alkóxidos, agua, y metanol. Finas películas de recubrimiento PMS preparados por inmersión de aluminio dentro de la solución sol proveen protección significante para el aluminio contra la corrosión inducida por el NaCl (cloruro de sodio). Factores importantes que gobiernan a un desempeño protector bueno de PMS fueron como siguen: (1) un incremento en el eslabonamiento en metal-O-Si con incremento en la temperatura de tratamiento, (2) un bajo grado de humectabilidad y esparcibilidad de agua; y, (3) un especifica geometría de microestructura, en la cual las unidades agrupadas consisten de crecimientos simétricos fractales de ramificaciones de polímeros que fueron unidas juntas. El tercer factor contribuyó a un decremento en la tensión generada por la contracción de películas, con lo cual se elimina agrietamientos y ralladuras de película.

### Coatings Characterization by Thermally Stimulated Current (TSC) and Relaxation Map Analysis (RMA)—C.M. Neag et al.

JCT, 65, No. 826, 37 (Nov. 1993)

Thermally stimulated current (TSC) and relaxation map analysis (RMA) were used to characterize the molecular behavior of several coating systems. The experiments were designed to test the instruments utility for problem solving, product development, and research in the coatings industry. The results obtained using a TSC/RMA Spectrometer provided information on the glass transition temperature ($T_g$), general morphology, and relative cure levels for container coatings, powder coatings, and a coating for plastic substrates; all of the experiments were completed using coatings over typical substrates. RMA analysis and the resulting degree of disorder (DOD) values were used in quantifying differences in the level of cure for powder coatings and a coating for sheet molding compound (SMC) plastic substrates

### Evolution of the Protective Mechanisms of Sinz-Rich Paints During Atmospheric Exposure—S. Feliu Jr. et al.

JCT, 65, No. 826, 43 (Nov. 1993)

The results of weathering and electrochemical tests were integrated in order to study the evolution of the protective mechanisms of zinc-rich paints (ZRP) over time.

After three years of atmospheric exposure, some of the tested ZRP coatings still maintained a great part of their original capacity for providing cathodic protection. The paints formulated with the ethyl silicate vehicle stand out in this respect. On the other hand, an important improvement of the barrier effect is shown, especially with the paints formulated with the epoxy-polyamide vehicle.

### Caractérisation des Revêtements par Courant Stimulé Thermiquement (TSC) et par Analyse de Relaxation (RMA)—C.M. Neag et al.

JCT, 65, No. 826, 37 (Nov. 1993)

Les techniques de courant stimulé thermiquement (TSC) et d'analyse de relaxation (RMA) ont été utilisées pour la caractérisation du comportement moléculaire de plusieurs systèmes de revêtements. Les analyses ont été mises au point pour tester l'habieté des instruments à régler des problèmes, à développer des produits et la recherche dans le domaine de l'industrie des revêtements. Les résultats obtenus par l'utilisation du spectromètre TSC/RMA ont donné de l'information sur le $T_g$, la morphologie générale et les niveaux relatifs de durcissement pour les revêtements en poudre et un revêtement pour un subjectile en plastique: toutes les analyses ont été complétées en utilisant des revêtements appliqués sur des subjectiles communs. Les valeurs obtenues de l'analyse de relaxation et du degré résultant du désordre (DOD) ont été utilisés pour la quantification des différences de niveaux de durcissement pour les revêtements en poudre et un revêtement appliqué sur un subjectile en plastique.

### Évolution des Mécanismes de Protection de Revêtements Riches en Zinc Durant une Exposition Extérieure—S. Feliu, Jr. et al.

JCT, 65, No. 826, 43 (Nov. 1993)

Une intégration des résultats d'exposition aux intempéries et de données électrochimiques est utilisée pour l'étude de l'évolution avec le temps des mécanismes de protection de revêtements riches en zinc.

Après une période d'exposition extérieure de trois années, quelque uns des revêtements riches en zinc testés ont maintenu une large part de leur capacité originale de protection cathodique. Les revêtements formulés avec du silicate d'éthyle comme liant ont été les plus performants à cet égard. Par contre, une amélioration importante de l'effet de protection est démontrée avec les revêtements formulés avec un liant à base de résine époxydique/polyamide.

### Caracterizacion de Recubrimientos por Analisis de Corriente Estimulada Termicamente (TSC) y por Mapeo de Relajacion (RMA)—C.M. Neag et al.

JCT, 65, No. 826, 37 (Nov. 1993)

Se usaron las tecnicas de análisis de corriente estimulada termicamente (TSC) y de mapeo de relajación (RMA), para caracterizar el comportamiento molecular de varios sistemas de recubrimientos. Los experimentos fueron diseñados para probar la utilidad de los instrumentos para la solución de problemas, el desarrollo de productos y la investigación en la industria de recubrimientos. Los resultados obtenidos usando un espectómetro TSC/RMA suministraron información de la $T_g$, morfologia en general y los niveles de curado relativos para recubrimientos de envases, recubrimientos en polvo y recubrimientos para sustratos plasticos. Todos los experimentos fueron completados usando recubrimientos sobre sustratos típicos. El análisis de mapeo de relajación y los resultantes valores en el grado de desorden (DOD) fueron usados en diferencias cuantificables en el nivel de curado para recubrimientos en polvo y recubrimientos para sustratos plasticos de compuestos de hoja moldeada.

### Evolucion de los Mecanismos de Proteccion de las Pinturas Ricas en Zinc Durante su Exposicion a la Atmosfera—S. Felui, Jr. et al.

JCT, 65, No. 826, 43 (Nov. 1993)

Se usa una integración de los resultados de intemperismo y preubas electroquímicas para el estudio de la evolución con el tiempo del mecanismo de protección de las pinturas ricas en zinc (ZRP).

Después de tres años de exposición atmosférica, algunos de los recubrimientos ZRP probados aun mantienen una gran parte de su capacidad original para proveer protección catódica. Las pinturas formuladas con silicato de etil como vehículo sobresalen en este respecto. Por otro lado, un importante progreso de los efectos de barrera es mostrado especialmente con las pinturas formuladas con vehículos de epoxipoliamida.

# Federation News

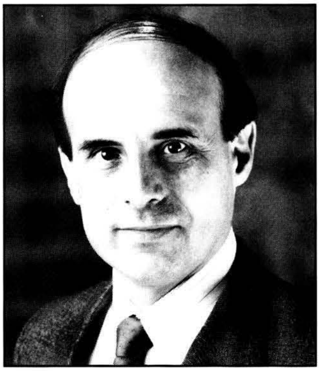
## John P. Lanning, of Louisville Society, Elected 72nd President Of the Federation, at Annual Meeting in Atlanta, GA

John A. Lanning (Louisville Society), Product Quality Manager, Courtaulds Coatings, Inc./Porter Paints Division, Louisville, KY, became the 72nd President of the Federation of Societies for Coatings Technology, on October 29, at the Federation's Annual Meeting, in Atlanta, GA.

Joseph P. Walton (Cleveland Society), Executive Vice President, Jamestown Paint, Jamestown, PA, was named President-Elect, and Darlene Brezinski (Chicago Society), President, Consolidated Research, Mount Prospect, IL, was elected Secretary-Treasurer.

### President Lanning

Mr. Lanning most recently served as President-Elect of the Federation. He served as Secretary-Treasurer of FSCT (1991-92), and is a member of the Executive Committee. Mr. Lanning has been a member of the Board of Directors since 1989, and served on the Board as Member-at-Large from 1985-87. He is a member of the Finance Committee, and has served as Chairman of the Annual Meeting Program, Paint Show Exhibits' Awards, and Society Speaker Awards Committees. In addition, Mr. Lanning was a Trustee of the Coatings Industry Education Fund, an Ex-Officio member of the Professional Development Committee, and a member of the Ad-Hoc Strategic Planning Committee.



J.A. Lanning

A Past-President of the Louisville Society (1983-84), Mr. Lanning received the Society's Outstanding Service Award in 1990. Also, he was a member of the Membership and Technical Committees.

Graduated from the University of Louisville, Mr. Lanning has been a member of the coatings industry for 27 years.

### President-Elect Walton

Mr. Walton previously served as Secretary-Treasurer of FSCT and is a member of the Executive Committee. He is Secretary-Treasurer of the Coatings Industry Education Fund, serves as a member of the Finance Committee, and is an ex-Officio member of the Professional Development Committee. Mr. Walton was a member of the Manufacturing Committee from 1987 to 1992, having served as Chairman from 1987 to 1990. He also was a member of the Annual Meeting Program and the Ad-Hoc Strategic Planning Committees.

Mr. Walton is Chairman of the Cleveland Society's Manufacturing and Directory Committees.

He was graduated from Wheeling Jesuit College and has been active in the coatings industry for 20 years. Mr. Walton served as Chairman of the National Paint & Coatings Association's Manufacturing Committee, and is the President of the Cleveland Paint and Coatings Association (CPCA). He was the recipient of the CPCA's Carl J. Byron Distinguished Service Award and the Western Reserve Chapter of the National Management Association's Manager of the Year Award.

### Secretary-Treasurer Brezinski

Dr. Brezinski is a member of the FSCT Board of Directors, the Publications Committee, the Editorial Review Board of the JOURNAL OF COATINGS TECHNOLOGY, Co-Editor of the *Federation Series on Coatings Technology*, and Editor of *An Infrared Spectroscopy Atlas for the Coatings Industry (4th Edition)*. She also has served as Chairman of the Annual Meeting Program, George Baugh Heckel, Mattiello Lecture, and Roon Committees. In 1983, Dr. Brezinski was the recipient of FSCT's George Baugh Heckel Award, and in 1991, she received the Union Carbide Award. Dr. Brezinski also is a member of the Gallow's Birds.

She has served as Chairman of the Chicago Society's Technical Committee and was instrumental in the development of the FSCT Coatings MSDS System on CD-ROM.

Dr. Brezinski was graduated from Mundelein College, and received the M.S. and Ph.D. Degrees in Chemistry from Iowa State University. She had served as Chairman of the Chemistry Department at Mundelein, prior to becoming a contributor to the coatings industry some 21 years ago.

### Executive Committee

Forest Fleming (Piedmont Society), Technical Director of Wood Coatings Building Products Group, Akzo Coatings, Inc., High Point, NC, has been elected for a three-year term on the Federation Executive Committee. Mr. Fleming is the Piedmont Society Representative to the Board of Directors.

He was President of the Piedmont Society (1989-90) and served on the Society's Publications and Technical Committees. In 1988, while serving as Membership Committee Chairman, Mr. Fleming was presented with a Certificate of Appreciation for the exemplary increase of Society's membership during the 1987-88 year.

He was graduated from Western Carolina University with a degree in Biology, and been affiliated with the coatings industry for 16 years.

### Board of Directors

Milton A. Glaser (Chicago Society), Consultant, a Past-President of FSCT (1956-57), has been elected for a two-year term on the FSCT Board of Directors.

Mr. Glaser is a Society Honorary, Federation Honorary, and 50-Year Member of Federation. He received the FSCT Distinguished Service Award in 1959 and the George Baugh Heckel Award in 1963. In 1974, Mr. Glaser presented the Joseph J. Mattiello Memorial Lecture. Also, he served as President of the Paint Research Institute , and was Chairman of the Educational and Liaison Committees. In addition, Mr. Glaser served as FSCT Delegate to the IUPAC.

A Past-President of the Chicago Society (1945-46), he received the Society's Outstanding Service Award in 1957, and the Merit Award from the Chicago Technical Societies Council in 1968. Also, Mr. Glaser

*(Continued on next page.)*

# 1993-94 Committee Chairmen Appointed by President Lanning

Chairmen of 30 committees of the Federation of Societies for Coatings Technology for 1993-94 have been named by President John A. Lanning. A complete roster of all committees will be published in the 1994 FSCT *Year Book*.

An asterisk (*) indicates re-appointment for 1993-94.

*A.F. Voss/American Paint & Coatings Journal Awards*—Ronald R. Brown, of Rohm and Haas Co., Charlotte, NC.*

*Annual Meeting Host*—Jerry Mattson, of University of Southern Mississippi, Hattiesburg, MS.

*Annual Meeting Program*—Ronda Miles, of Union Carbide Corp., Garland, TX.

*Armin J. Bruning Award*—Robert T. Marcus, Pantone Inc., Carlstadt, NJ.*

*By-Laws*—Fred G. Schwab, of Coatings Research Group, Inc., Cleveland, OH.*

*Corrosion*—Mike Jackson, Louisville, KY.*

*Educational*—Donald Boyd, of PPG Industries, Inc., Allison Park, PA.

*Educational Coordinating*—Donald Boyd, of PPG Industries, Inc., Allison Park, PA.*

*Finance*—Colin D. Penny, of Coatings Technology, Hampton, VA.

*George Baugh Heckel Award*—John J. Oates, Midland Park, NJ.

*Inter-Society Color Council*—Ralph Stanziola, Neshanic Station, NJ.*

*Liaison*—William F. Holmes, National Paint & Chemicals, Inc., Garland, TX.

*Manufacturing*—Don Mazzone, of The O'Brien Corp., S. San Francisco, CA.*

*Joseph J. Mattiello Lecture*—Percy E. Pierce, Consolidated Research, Inc., Monroeville, PA.

*Membership Services*—Brenda Carr, of Coatings Development Co., Painesville, OH.*

*Memorial*—Phil C. Bremenstuhl, of ZENECA Resins, Riverside, CA.

*Nominating*—Colin D. Penny.

*Paint Industries' Show*—Richard E. Max, of Zarco Industries, Chicago, IL.*

*Planning*—Saul Spindel, of D/L Laboratories, Inc., New York, NY.*

*Professional Development*—Roger Woodhull, of California Products Corp., Cambridge, MA.*

*Publications*—Robert F. Brady, Jr., of U.S. Naval Research Lab., Gaithersburg, MD.*

*Roon Awards*—N. Bradford Brakke, of Lilly Industries, Inc., Indianapolis, MN.

*Society Secretaries Awards*—Carolyn L. Tully, of Sun Chemical Corp., Cincinnati, OH.

*Society Speakers Awards*—G.C. Simmons, of Becker Industrial Coatings Ltd., Liverpool, England.

*Technical Advisory*—Gail Pollano, of ZENECA Resins, Wilmington, MA.*

* * * * * *

### Delegates to Other Organizations

*National Association of Corrosion Engineers (NACE)*—Mike Jackson.*

*National Paint & Coatings Association Scientific Committee (NPCA)*—Gail Pollano.*

*NPCA and Governmental Agencies*—Sidney J. Rubin, of Empire State Varnish Co., Brooklyn, NY.*

*IUPAC*—Gerry J. Gough, of Holden Surface Coatings Ltd., Birmingham, England.

*Steel Structures Paint Council (SSPC)*—Mike Jackson.*

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# John Lanning Elected President of FSCT

*Continued from previous page.*

served as Chairman of the Society's Technical Committee.

He graduated Magna Cum Laude from Tufts University with the B.S. Degree in Chemical Engineering, and did graduate work in Chemistry at University of Chicago, and in Polymer Chemistry at Northwestern University.

Prior to his retirement, Mr. Glaser served as Vice President, Research & Development, of the Midland Division, Dexter Corporation. He has been a major contributor to the coatings industry for 55 years.

Elected to serve two-year terms as Members-at-Large on the Board of Directors are Thomas E. Hill (Western New York Society), Corporate Director—Total Quality, Pratt & Lambert, Inc., Buffalo, NY; Rose A. Ryntz (Detroit Society), Technical Specialist/Paint & Materials, Plastic & Trim Products Department, Ford Motor Company, Detroit, MI; and Freidun Anwari (Cleveland Society), Chemist, Coatings Research Group, Inc., Cleveland, OH.

Mr. Hill served on the Executive Committee from 1987 to 1990, and was the Western New York Society Representative to the Board of Directors from 1983 to 1990. Also, he served on the Finance, Paint Show Exhibits Awards, and Annual Meeting Program Committees.

Mr. Hill is a past- member of the Western New York Society Executive Committee.

He received his technical education from West Virginia University and his business education from the State University of New York at Buffalo. Mr. Hill has been active in the coatings industry for 25 years.

Dr. Ryntz is a member of the Editorial Review Board of the JOURNAL OF COATINGS TECHNOLOGY, and the Professional Development and Technical Advisory Committees. Also, she was a member of the Ad-Hoc Strategic Planning Committee.

Dr. Ryntz currently is Chairman of the Detroit Society Technical Committee. In addition, she was presented with the A.F. Voss/*American Paint Journal* Award in 1987, 1988, 1989, and 1991, and the Society Speaker Award in 1986 and 1989.

Dr. Ryntz was graduated from Wayne State University with the B.S. Degree in Chemistry, and from the University of Detroit with the Ph.D. Degree in Polymer Chemistry. Currently, she is an Adjunct Professor at the University of Detroit.

A recipient of a 1992 Women in Coatings Award, Dr. Ryntz has been a member of the coatings community for 10 years.

Mr. Anwari is Past-President of the Cleveland Society and has been a member of the Society's Board of Directors since 1986. He is a member of the Society's Technical Committee, and served as Chairman from 1987-1992. In addition, Mr. Anwari currently is a member of the Society's Awards, By-Laws, and Program Committees. He also has served as a member of the Audit and Finance Committees. In 1991, Mr. Anwari was presented with the Society's Award of Merit.

He was graduated from North Dakota State University with the B.S. Degree in Chemistry, and from Cleveland State University with the M.S. Degree in Chemistry. Mr. Anwari has been a contributor to the coatings industry for eight years.

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# Government and Industry

## NPCA Participates in Wood Furniture Reg-Neg Meeting

The National Paint & Coatings Association and other industry representatives met with federal and state regulators and environmental and public interest groups in the effort to continue consideration of both a national emissions standard for hazardous air pollutants (NESHAP) and control technique guidelines (CTGs) for the wood furniture and kitchen cabinet manufacturing industries.

Industry representatives presented an extensive technical paper intended to function as a basis for subsequent discussions by the reg-neg committee. The paper was prepared by the Industry Caucus after extensive research, technical analysis and consultations, and presented both a potential technical and regulatory framework for the rule.

Because the reg-neg committee has seriously considered an industry proposal to allow emissions averaging across coatings steps, coatings representatives made technical presentations regarding the measurement and reporting of hazardous air pollutants (HAPs) and volatile organic compounds (VOCs) in a representative furniture operation involving such typical coatings applications as stains, sealers, fillers, washcoats, and topcoats. The presentations were designed to explain the great diversity of production operations, facilities, and products within the furniture and cabinet industries, as well as an explanation of how an averaging system would operate.

Following review and discussion of the industry paper, the Environmental/State Caucus provided comments, questions and a response to the industry proposal. Several areas of agreement with the industry were identified, such as a proposed maximum achievable control technology (MACT) category, and a new method for expressing HAPs and VOCs in terms of the pounds of VOCs or HAPs per pound of weight solids. The Environmental/State Caucus also suggested a number of additional areas of discussion and further negotiation, such as solvent substitution by coatings manufacturers, best operating practices, segmentation of the industry for VOC control, and setting of the MACT "floor."

The facilitator for the reg-neg, CDR Associates of Boulder, CO, referred a number of issues to various work groups of the reg-neg committee. The work groups will focus on areas such as data reconciliation, enforcement, work practices, small business, toxicity, and gluing.

The final meeting of the full wood furniture Reg-Neg Committee was scheduled for October 21-22.

### NPCA Restructures Staff

As a result of increased duties in its issue management area, the National Paint & Coatings Association, Washington, D.C., has restructured and renamed its Communications Division into a new Public Affairs Division. The association will continue to address the same industry issues, however, more emphasis will be concentrated on industry outreach as a means of educating the media, legislators, environmentalists, and the public.

As part of the reorganization, NPCA's Kris Cook has been promoted to the position of Division Director, Public Affairs. Ms. Cook has been with NPCA for eight years, starting as a communications manager responsible for writing the association's newsletter, *Coatings*, and editing the *Member Services Directory*. In May of 1992, she was promoted to Director of Publications.

Staff in the new division include Marilyn Ludwig, Director, Communications; Katrina Norfleet, Associate Director, trade press and community affairs; Lisa Warren, Associate Director, publications and public relations; Cathy Young, Manager, desktop publishing; Susan Cottrell, Coordinator, publications and administration; and a new administrative assistant.

In addition, NPCA has hired several employees to fill needs in other divisions. Phillip D. Bour joined NPCA as Accountant/Information Systems Manager and Whitney Long has joined the Health, Safety and Environmental Affairs Division as Manager, Health Affairs. In the State Affairs Division, Heidi McAuliffe will serve as Counsel. Angela Talbot is the new Director, Meetings and Conventions.

## Painting & Decorating Contractors Announce Deadline for Annual Contractor Contest

The Picture It Painted Professionally contractor contest, sponsored by the Painting & Decorating Contractors of America (PDCA), has recognized the creativity and skill of professional painting contractors for the past 12 years. The deadline for entries in the 1994 competition is January 7, 1994.

The competition is open to both PDCA members and nonmembers. Projects must have been completed in 1992 or 1993, and can include interior and exterior, residential and commercial structures. Industrial projects—factories, plants, bridges, towers, tanks, machinery—are also eligible in their own category.

All entries must be accompanied by an official entry form and "before" and "after" 35mm color slides depicting the work that was done. Projects will be judged by an independent panel of industry representatives on the basis of four criteria: use of color, use of special painting treatments, overall creativity, and overall skill and execution. Winners of the contest will be announced during the PDCA Annual Convention in Anaheim, CA, on February 16-19. In addition to being presented with awards, the contest winners will be spotlighted in a fully-narrated slide presentation that will later be converted to videotape for their own personal use.

In addition, all winners will be featured in the contest editions of the *Painting & Wallcovering Contractor (PWC)* and the *American Painting Contractor* magazines.

For an official entry form, or more information on the PIPP contest, write: PIPP Contest, Painting & Decorating Contractors of America, 3913 Old Lee Highway, Suite 33B, Fairfax, VA 22030.

## C.P. Hall Acquires Polyester Resin Line

The C.P. Hall Company, Chicago, IL, has purchased the Paraplex® polyester resin product line from Rohm and Haas, Philadelphia, PA. The acquisition augments the Paraplex polyester plasticizer product line which was originally purchased, together with the tradename, by C.P. Hall in 1981.

The C. P. Hall Company supplies a line of specialty chemicals, polymer additives, and related industrial chemical products to the polymer industry.

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## Relief Efforts for Midwest Flood Victims Assisted by Coatings & Decorating Industries

Victims of the devastating floods that ravaged the Midwest will be offered some relief as a result of the efforts of members of the paint and decorating products industries. The National Decorating Products Association (NDPA) has organized member pledges of nearly $500,000 in cash and product donations to the Salvation Army as part of the Midwest flood relief effort. According to Ernest W. Stewart, Executive Vice President of NDPA, "Our dealer members and affiliated suppliers have pledged over 15,000 gallons of paint and coatings and over 150,000 rolls of wallcoverings, plus over $9000 in cash donations."

The association is coordinating its efforts with the Salvation Army to gather donations of decorating products, tools and cleaning supplies for victims seeking to repair their homes now that flood waters have receded.

In addition, the Sherwin-Williams Company, through the efforts of the Coatings Division paint plant in Coffeyville, KS, is donating 13, 875 gallons of interior paint to the American Red Cross, to be used in repainting the homes of the victims. The in-kind donation is valued at $10 per gallon, or a total of $138,750.

Also, APTUS Environmental Services of Coffeyville is donating transportation for the paint to the St. Louis Red Cross staging area. The equivalent of five truckloads, that donation is valued at approximately $7000.

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### Capacity Doubled at Harcros Organics Ethoxylation Plant

The start-up of a major expansion of Harcros Organics' ethoxylation/propoxylation plant in Kansas City, KS, has been announced. The two-phase upgrade to modernize the plant to state-of-the-art conditions increased the plant's capacity from 30 million to 60 million pounds per year.

Phase one, conducted to meet the company's goals for safety and environmental standards with a modest increase in plant capacity, was completed in late 1990. The second phase, just completed, was devoted to the expansion of reactor capacity and associated support equipment.

Harcros Organics, a division of Harcros Chemicals, Inc., produces ethoxylates and propoxylates for use in a wide range of surfactants, emulsifiers, and other derivatives. These products are sold by the distribution division of Harcros Chemicals' 26 branch operations throughout the U.S.

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### Liquid Carbonic to Build Air Separation Plants

Liquid Carbonic International, Oak Brook, IL, will build two air separation plants in South America. Each of the two cryogenic plants, one of which will be located in Bogota, Columbia, and the other in Jose, Venezuela, will process 60 tons per day of oxygen, nitrogen, and argon for industrial and medical applications.

The new plants are scheduled to begin operations in spring of 1995.

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### PPG and Akzo Cooperate In Automotive Original Coatings

PPG Industries, Pittsburgh, PA has announced its intent to cooperate in the field of automotive original coatings with Akzo America Inc., Akzo nv, Arnhem, the Netherlands.

PPG will take primary responsibility for marketing and customer support for automotive original coatings in Europe. Through this cooperative effort, Akzo's present licensees and European customers will have access to PPG's technology for automotive original coatings.

In addition, the two companies have plans to enlarge their existing cooperation in this area in Brazil and Argentina.

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### Coatings Raw Materials R&D Lab Facilities Expanded by Hüls America

Hüls America, Piscataway, NJ, has expanded its coatings raw materials research & development facilities by building a new laboratory at its plant in Theodore, AL, and expanding its labs in Piscataway.

Hüls America's Alabama laboratory works primarily on powder coatings—one of the fastest-growing segments of the coatings industry. The fully equipped powder coatings R&D lab is part of Hüls America's new isophorone derivatives facility.

The firm's New Jersey labs, which support liquid coatings, have increased space threefold. The expanded labs include more bench area for immediate and future staff additions, six new ventilation hoods, more storage space, a spray booth, a climate-controlled room and extra offices.

# Regulatory UPDATE

## NOVEMBER 1993

This digest of current regulatory activity pertinent to the coatings industry is published to inform readers of actions which could affect them and their firms, and is designed to provide sufficient data to enable those interested to seek additional information. Material is supplied by National Paint and Coatings Association, Washington, D.C.

**Air Quality—Architectural and Industrial Maintenance (AIM) Coatings VOC Regulatory Negotiation**—At the end of July, a so-called tentative understanding was reached with U.S. Environmental Protection Agency (EPA) concerning a preliminary regulatory framework that EPA might use in drafting a detailed proposal. Such a proposal will be presented for consideration at the next AIM reg-neg meeting. With respect to this understanding, it should be noted that there was not a consensus among the reg-neg participates. Two basic features of the tentative understanding concern: (1) the basic concepts that could form the basis of the regulatory framework; and (2) percentage reduction goals for volatile organic compound (VOC) emissions over certain periods.

On the basis of that understanding, the Keystone Center, the facilitator for the negotiation, put together a memorandum framework that incorporated what has been tentatively understood as basis for further refinement. This document will probably form the foundation for an EPA draft that may become the basis for further negotiation.

As to the timing of all this, it is expected that the EPA draft will be out in November.

The VOC reduction goals of the understanding are as follows: a reduction in VOC emissions from AIM coatings of 25% to begin in 1996, followed by an additional 10% reduction in 2000, and another reduction of 10% in 2003. The first reductions would be achieved by specifying VOC limits in a table of standards. The subsequent reductions could be achieved by implementing a company product line VOC averaging methodology. As an alternative to the averaging, a company could meet VOC limits specified in revised and more stringent tables of standards. Reality check provisions would be used to determine whether the additional VOC reductions mandated for 2000 and 2003 would be technically feasible. If these checks determine that the reductions are not feasible, the EPA would adjust the requirements accordingly.

Another important feature of the draft will be provisions to assist small businesses including delaying their compliance dates by at least two years. (As a practical matter, if EPA, as is expected, promulgates a rule in 1994, with a compliance date of 1996, the lead time to small businesses would be four years.)

It must be remembered that the preliminary tentative understanding that is the basis for the EPA's efforts to develop a draft proposal is not binding on any of the negotiators. Those that concurred in the tentative understanding in essence only concurred with EPA's proceeding to develop a detailed proposal using the basic principles or features of the tentative understanding.

Further, the understanding does not yet settle specific VOC limits for specific coatings categories. Additionally, the regulation that is being developed by EPA must be approved by the negotiators before it can become a binding consensus agreement which would then form the basis for a formal proposed rulemaking by EPA. And this formal proposal itself would first have to go through formal public comment before EPA could issue it in final form. Therefore, much activity and decision making lies ahead before anything definite will be settled.

**Federal Clean Air Strategies**—The EPA continues to undertake a number of activities to implement the Federal Clean Air Act Amendments of 1990 that will have major impact upon the paint industry and its customers. While the states remain largely responsible for carrying out the Clean Air Act requirements, EPA is primarily responsible for setting the national standards and requirements for the state programs.

EPA continues to develop programs to carry out the requirement of the Clean Air Act Amendments of 1990. Among the programs that EPA is expected to establish before the end of this year are the following: regulations that will require enhanced monitoring and certification concerning regulated air pollutants that are emitted from facilities; the standards for state economic incentive programs that can be implemented by the states in ozone nonattainment areas which may include pollutant taxes on products; and the schedule by which EPA will issue "maximum achievable control technologies" (MACTs) for sources emitting hazardous air pollutants.

EPA also continues to develop "control technique guidelines" (CTGs) and national regulations for regulating VOC emissions from architectural and industrial maintenance coatings and several surface coating processes (i.e., plastic parts, shipbuilding and repair, automotive refinish, wood furniture, and aerospace). Originally scheduled to be proposed by November 15, 1993, the time frames for most of these efforts

The *Regulatory Update* is made available as a service to FSCT members, to assist them in making independent inquiries about matters of particular interest to them. Although all reasonable steps have been taken to ensure the reliability of the *Regulatory Update*, the FSCT cannot guarantee its completeness or accuracy.

has slipped and many states are considering the adoption of their own regulations for these source categories. Finally, November 15, 1993 is the due date for receipt by EPA of two important state programs: the clean air operating permit programs and the plans of states with moderate and above ozone nonattainment areas to reduce VOC emissions by 15% by 1996.

**RCRA Update**—Although legislative activity to reauthorize the Resource Conservation and Recovery Act (RCRA) has not received strong congressional support this year, the EPA has focused its energies on regulatory actions which affect RCRA compliance and enforcement.

Last October, Congress mandated that EPA finalize a rule to replace the "mixture" and "derived-from" rules by October 1994. The Hazardous Waste Identification Rule (HWIR) Advisory Committee discussions indicate that it will recommend a bright line test for contaminated media—those media with a level of hazardous constituents falling above the bright line will trigger strict handling procedures and contaminated media with a level of hazardous constituents falling under the bright line would be considered lower level hazards and fall outside of the strict Subtitle C regulations. At this time, however, there is no consensus of where the "bright line" should be established. Although the EPA was expected to propose a rule by September of this year, the earliest any action will emerge from EPA will be in November.

EPA's draft Strategy for Combustion of Hazardous Waste in Incinerators and Boilers and Interim Final Guidance for Hazardous Waste Generators on Waste Minimization was published by the agency in May. The guidance placed an 18-month temporary freeze on new hazardous waste incinerators (no new permits) while placing new federal controls on existing incinerators, boilers, and industry furnaces (BIFs). Although EPA initially published this document as "guidance," the agency has retracted somewhat by announcing that the draft strategy and guidance are merely goals and are not to be applied by the EPA Regions on a uniform basis.

**Storm Water Permit Application**—The EPA promulgated final Storm Water Permit Application regulations on November 16, 1990. These regulations require certain manufacturers to obtain a permit to discharge storm water associated with industrial activity. The EPA established three options for obtaining a permit, they are: individual application, group application, or coverage under a general permit.

The group storm water permit applications consist of two parts. Part one requires a list of all participating facilities, basic industry information, and identification of the group representatives. The second part requires quantitative data from the representative facilities. The deadline established for part one was October 1, 1991, and for part two, October 1, 1992.

The EPA recently reported that over 700 groups covering 40,000 facilities remain in the group application process. To effectively permit these facilities, the EPA has categorized the groups into 31 industrial sectors by industrial activity. The EPA is currently developing industry-specific requirements for each sector and plans to incorporate these requirements into a singie, multi-sector permit.

The multi-sector permit will be issued as a general permit for participants in groups located in the 12 states that have not been delegated National Pollutant Discharge Elimination System (NPDES) permitting authority. The remaining states may choose to use EPA's multi-sector permit and may add or delete requirements as necessary.

The multi-sector permit is expected to be published in the Federal Register by the end of October; a 90-day comment period will also be provided. This delay in issuance of the multi-sector permit has resulted in EPA missing the statutory deadline for permit issuance, which was October 1, 1993.

**Toxic Substances Control Act (TSCA)**—The EPA, though its Office of Pollutant Prevention, and Toxic Substances (OPPT), has undertaken a number of initiatives under TSCA, intended to improve the functioning of the program and better protect human health.

—On February 8, 1993, EPA published a number of rulemaking proposals under Section 5 of TSCA which were intended to simplify Premanufacture Notice (PMN) requirements for new chemicals. These proposals included:

(1) An expansion of the Low Volume Exemption (LVE) set forth in 40 C.F.R. §723.50 as well as establishment of a Low Release/Exposure Exemption (LoREX).

(2) Technical amendments to the current PMN Rules set forth in 40 C.F.R. §720, in particular modifying the relationship between bona fide PMN and NOC reporting.

(3) Increased use of Significant New Use Rules (SNURs) for chemicals not now regulated by Section 5(e) in 40 C.F.R. §721.170.

(4) Revisions to the Polymer Exemption Rules in 40 C.F.R. §723.250, touching on the so-called "polymer salts" exemption.

Paint industry comments on these proposals were submitted in July to provide EPA with the industry view of these various regulatory obligations. Essentially, as the comment noted, operations undertaken in the production of paints and coatings are not generally considered to be chemical "manufacturing" as defined under TSCA. Since the formulation and production of paint products has always been considered "processing" of chemicals under TSCA and no reportable new chemicals are formed during processing, it was stressed that despite some inconsistent interpretations of chemical processing in TSCA regulations, premanufacture notifications (PMNs) are not required by the statutory language of TSCA.

—The EPA also published a final rule on July 27, 1993 which required manufacturers and exporters of 10 listed solvents (many of which are used in the industry) to undertake extensive testing for neurotoxicity under Section 4 of TSCA. EPA is permitting the testing to be done by a consortium of chemical manufacturers who enter into an agreement with EPA and has also provided a mechanism for importers of mixtures containing these chemicals to seek an exemption from this rule.

—EPA is conducting a formal re-evaluation of its policy on "substantial risk" to health and the environment under Section 8(e) of TSCA, specifically addressing such issues as reporting deadlines, the circumstances under which reports should be filed, and making claims of confidential business information in connection with such filings. The Agency has solicited comments on this proposal.

**Administration Appointments/Cabinet Status for the Environmental Protection Agency**—Various sponsors introduced a bill, "The Department of the Environment Act of 1993" (S. 171), intended to elevate the EPA to cabinet rank. It passed the Senate on May 4, 1993, and was subsequently sent to the House of Representatives for action. Previous Congresses have failed to pass similar bills, but President Clinton's active support for this measure has greatly increased its chances for success. One important sticking point has been the inclusion of a provision in the Senate bill which requires EPA to conduct environmentally risk assessments in its rulemaking activities. Since Clinton, environmentalists, and their allies in the House have expressed opposition to the inclusion of this requirement in an EPA cabinet bill, it may become a key issue in the conference committee, should this bill pass the House.

**Hazardous Materials Transportation**—On December 21, 1990 (55 *Federal Register* 52402), the Department of Transportation's (DOT) Research and Special Programs Administration (RSPA) issued a final rule which comprehensively revised its Hazardous Material Regulations (HMR) with respect to hazard communication, classification, and packaging requirements based on the United Nations Recommendations on the Transport of Dangerous Goods. Additionally, a document which responded to petitions for reconsideration and containing editorial corrections was published on December 20, 1991 (56 *Federal Register* 66124). Since that time, RSPA has published numerous documents that further amended and provided editorial revisions to the HMRs.

The intended effects of the new regulations are to: (1) simplify and reduce the volume of the HMR; (2) enhance safety through better classification and packaging; (3) promote flexibility and technological innovation in packaging; (4) reduce the need for exemptions from the HMR; and (5) facilitate international commerce.

The most significant change of this rulemaking is the switch in nonbulk packaging, from specification packaging requirements to performance oriented packaging (POP). HM-181 also adopted new classification and hazard communication requirements which are intended to align domestic regulations with international rules.

Immediate industry compliance with the regulation was authorized, but not required. DOT has provided a phase-in period to allow for an orderly transition and to minimize any burdens associated with the new requirements. As of October 1, 1993, shippers will be required to comply with new hazard communication and classification provisions (other than placarding requirements) and new modal segregation requirements.

Other dates of concern are: October 1, 1994, which is the last day a specification package may be manufactured; October 1, 1996, which is the last day specification packages may be shipped; and October 1, 2001, when new placard requirements take effect.

**Lead**—The Secretary of Housing and Urban Development, Henry G. Cisneros, has announced the establishment of a Task Force on Lead-Based Paint Hazard Reduction and Financing. The announcement was published in the *Federal Register* on October 13, 1993 (58 *Federal Register* 52973).

The task force, established pursuant to Section 1015 of the Residential Lead-Based Paint Hazard Reduction Act of 1992, includes individuals representing the following entities: the Department of Housing and Urban Development; the Farmers Home Administration; the Department of Veterans Affairs; the Federal Home Loan Mortgage Corporation; the Federal National Mortgage Association; the Environmental Protection Agency; employee organizations in the building and construction trades industry; landlords; tenants; single-family and multifamily real estate interests; nonprofit housing developers; property liability insurers; public housing agencies; low-income housing advocacy organizations; national, state, and local lead-poisoning prevention advocates and experts; and community-based organizations located in areas with substantial rental housing.

According to the notice, these members were selected on the basis of personal experience and expert knowledge.

The mandate of the Task Force is to make recommendations to the Secretary of HUD and the Administrator of the EPA concerning:

(1) Incorporating the need to finance lead-based paint hazard reduction into underwriting standards;

(2) Developing new loan products and procedures for financing lead-based paint hazard evaluation and reduction activities;

(3) Adjusting appraisal guidelines to address lead safety;

(4) Incorporating risk assessments or inspections for lead-based paint as a routine procedure in the origination of new residential mortgages;

(5) Revising guidelines, regulations, and educational pamphlets issued by the Department of Housing and Urban Development and other federal agencies relating to lead-based paint poisoning prevention;

(6) Reducing the current uncertainties of liability related to lead-based paint in rental housing, by clarifying standards of care for landlords and lenders and by exploring the "safe harbor" concept;

(7) Increasing the availability of liability insurance for owners of rental housing and certified contractors and establishing alternative systems to compensate victims of lead-based paint poisoning; and

(8) Evaluating the utility and appropriateness of requiring both risk assessments or inspections and notifications to prospective lessees of rental housing.

The first meeting of the task force was held on November 8, 1993, at 9 a.m. (EST), at the Holiday Inn Capitol, 550 "C" Street, S.W. (corner of 6th & C Streets), Washington, D.C. 20024. Subsequent meeting dates will be published in the *Federal Register* at least 15 days prior to the meeting.

# States Proposed Legislation and Regulations

## Alaska

*Lead (Regulation)*—The Alaska Department of Labor has proposed rules concerning construction occupational safety and health. The proposal sets a permissible exposure limit for lead at 50 micrograms per cubic meter of air averaged over an eight-hour period; requires employers to make a lead exposure assessment of the construction worksite; and sets forth the safety standards with which employers must comply. For further information, contact the Department of Labor, P.O. Box 21149, Juneau, AK 99802, (907) 465-4855.

## Arizona

*Occupational Safety and Health (Regulation)*—The Arizona Industrial Commissions is proposing to update the Occupational and Safety Health Standards for both construction and general industry purposes by incorporating, by reference, the Federal Occupational and Safety Health Act (OSHA) standards for permit required confined spaces for general industry, air contaminants for general industry, and general industry safety and health standards applicable to construction work. For further information, contact Derek Mullins, Industrial Commission, 800 W. Washington, Rm. 202, Phoenix, AZ 85007-2922, (602) 542-5795.

## California

*Recycled Paint*—CA S. 734 (Rosenthal) requires all state agencies to purchase recycled solvents and recycled paints as defined by this bill. The purchase of the listed materials is required only when they are available of a fitness and quality equal to their nonrecycled counterparts, and are not more costly than five percent of the lowest price quoted by suppliers. The bill was signed by the Governor on October 9.

*Spray Paint*—CA A. 839 (Baca) authorizes the furnishing of aerosol containers of paint or related substances for use in school-related activities that are part of the instructional program when used under controlled and supervised situations within the classroom or on the site of a supervised project, as specified. The bill was signed by the Governor on September 29.

*Lead*—CA S. 617 (Hayden) states legislative intent that the state comply with the training and certification requirements contained in the Federal Residential Lead-Based Paint Reduction Act of 1992. Establishes within the state Department of Health Services a program to meet the requirements of certain federal hazard and safety laws, and requires the Department to adopt regulations thereunder with respect to workers who engage in lead-related construction work. The bill was vetoed by the Governor on October 10.

*Containers*—CA S. 466 (Boatwright) exempts, until January 1, 1996, rigid plastic packaging containers which are manufactured for use in the shipment of hazardous materials from meeting specified requirements, and requires the California Integrated Waste Management Board to make a specified report relating to federal regulations by January 1, 1995. Also exempts containers which are manufactured for use with food or cosmetics from meeting those criteria. The bill was signed by the Governor on September 20.

## Colorado

*Air Quality (Regulation)*—The Colorado Department of Health, Air Quality Control Commission is proposing to make revisions to the regulation regarding new source review requirements for PM10 precursors, as required by Section 189(e) of the Clean Air Act Amendments of 1990. The proposal establishes technical and offset requirements for new and/or modified sources of NOx and $SO_2$ in the Denver PM10 nonattainment area. It also considers pollutant trading to meet offset requirements and includes requirements of minor sources of NOx and $SO_2$. For further information, contact Joseph Palomba, Jr., Technical Secretary, Air Quality Control Commission, Department of Health, APCD-CC-B1, 4300 Cherry Creek Dr., S., Denver, CO 80222-1530, (303) 692-3180, or FAX (303) 782-5493.

*Water Quality (Regulation)*—The Colorado Water Quality Control Commission promulgated a final regulation that implements federal standards for the issuance of storm water permits which are now required at industrial facilities. For further information, contact the commission at (303) 692-3520.

## Florida

*Advanced Disposal Fee for Product Containers*—An emergency rule of Florida's Department of Revenue imposes a one cent advance disposal fee on containers made of metal, glass, plastic coated paper, and plastic. Beginning October 1, 1993, "distributors" of these products are required to register with the department and to charge the fee to "dealers" of the products—anyone who sells the products directly to consumers. Dealers are required to inform their customers that the advanced disposal has been collected. "Distributor" is defined to exclude manufacturers of products and thus manufacturers generally are exempt from charging the fee for its products. However, there are certain exceptions to this, e.g., where the manufacture sells directly to consumers in the state, sells its products through its own retail stores or distribution centers, or uses independent sales representatives in the state. Products sent to a distribution center in the state for sale outside of Florida are not subject to the fee.

Only containers "which are not less than five ounces or greater than one gallon by volume in capacity" are subject to the fee. Also, for the present, steel and aluminum containers are exempt from the fee because their metals have been found to have a recycling rate of at least 50%. Only the material of the container that constitutes the walls of the container are considered in determining whether the container is subject to the fee, e.g., an aluminum paint can that is exempt because of the recycling rate of that metal would not lose its exempt status because it has a handle made of plastic which does not have the required recycling rate.

The rule became effective on September 1, 1993 and distributors subject to the regulation were required to start collecting the fees on October 1, 1993. Because of the emergency nature of the regulation and its rapid implementation, the Department of Revenue has informed us that it presently is not imposing penalties for late registering or collections. Also, the department seems to recognize that the varied manufacturer/distributor/dealer relationships may make it difficult to interpret one's obligations under the regulation in some cases. Nonetheless, if there are any questions concerning one's obligations, the department should be contacted as soon as possible for a clarification. For additional information from the Florida Department of Revenue, contact Jeff Soff at (904) 488-6387. For additional information from NPCA, contact Jim Sell at (202) 462-6272.

## Idaho

*Air Quality (Regulation)*—The Idaho Department of Environmental Quality (DEQ) is proposing regulations to control the emission of toxic air pollutants from facilities through permits that would be issued when the facilities are constructed. The regulations also include the implementation of requirements of the Federal Clean Air Act operating permit program. For further information, contact Tim Teeter at DEQ, (208) 334-0457.

## Kentucky

*Occupational Safety and Health (Regulation)*—The Kentucky Labor Cabinet is proposing to set forth minimum safety and health requirement for those employees who must enter confined spaces for the purposes of performing their duties in the course of their employment. For further information, contact William L. Ralston, Kentucky Labor Cabinet, Office of Standards Development and Interpretation, 1047 U.S. 127 South, Ste. 4, Frankfort, KY 40601, (502) 564-2778.

## Louisiana

*Air Quality (Regulation)*—The Louisiana Department of Environmental Quality intends to submit a plan required by the Federal Clean Air Act which demonstrates a 15% reduction in VOC emissions in ozone nonattainment areas by 1996. For further information, contact Annette Sharp at DEQ, (504) 765-0219.

## Massachusetts

*Superfund (Regulation)*—The Massachusetts Department of Environmental Protection has proposed rules to streamline responses to releases of oil and hazardous materials, including specifying when responses being carried out under federal authorities are sufficiently adequate so as not to be subject to state requirements. For further information, contact the department at (617) 338-2255.

## Michigan

*Process Safety Management (Regulation)*—The Michigan Department of Labor has issued a final regulation that incorporates federal requirements concerning the management of highly hazardous chemicals. For further information, contact Connie Munschy at the Department, (517) 322-1845.

## Minnesota

*Hazardous Waste (Regulation)*—The Minnesota Pollution Agency proposed amendments to existing regulations regarding recycling activities, including provisions that define what constitutes a "recycling facility." For further information, contact Linnea Fredrickson at the agency, (612) 296-8549.

*Lead (Regulation)*—The Minnesota Department of Health has issued amendments establishing training standards for regulations that control the abatement of lead exposures in residences. For further information, contact Douglas Bension at the department, (612) 627-5017.

## Nevada

*Air Quality (Regulation)*—The Nevada Department of Conservation and Natural Resources Environmental Commission is proposing for permanent adoption an emergency rule relating to New Source Performance Standards and National Emission Standards. The rule adds standards for small industrial-commercial-institutional steam generating units, municipal waste combustors, VOC emissions from polymer manufacturing, and VOC emissions from synthetic organic chemical manufacturing. For further information, contact the Environmental Commission, 333 W. Nye Land, Rm. 128, Carson City, NV 89710, (702) 687-4670; FAX (702) 687-5856.

## Ohio

*Lead*—OH H. 447 (Campbell) provides for the licensing and certification of persons performing lead abatement. The bill was introduced in July and referred to the House Committee on Aging and Housing on September 29.

OH S. 162 (Drake) requires lead screening of certain young children; creates a tax credit for lead abatement; provides for licensure of persons performing lead abatement; provides that it is an unlawful discriminatory practice to refuse to sell to or rent housing because it will be occupied by a young child or pregnant woman. The bill is in the Senate Committee on Health and Human Services.

## Rhode Island

*Lead (Regulation)*—The Rhode Island Department of Environment, Health and Natural Resources has proposed amendments that would require reporting of elevated blood lead toxicity in adults. For further information, contact John Barkley at the department, (919) 733-4618.

## Tennessee

*Occupational Safety and Health (Regulation)*—The Tennessee Department of Labor is proposing occupational safety and health standards for general industry concerning air contaminants, cadmium, and lead. For further information, contact the Office of General Counsel, Department of Labor, 501 Union Building, 5th Floor, 501 Union St., Nashville, TN 37243-0655, (615) 741-0851.

## Texas

*Air Quality (Regulation)*—The Texas Air Control Board (TACB) has adopted final regulations that implement the facility operating permit requirements of the Federal Clean Air Act. The rule took effect on September 20, 1993. For further information, contact Gary McArthur at the TACB, (512) 908-1917.

## Vermont

*Air Quality (Regulation)*—The Vermont Agency of Natural Resources has issued final regulations that control the construction or modification of major facilities with the potential to emit 50 tons per year or more of VOCs and establishes control standards for regulating the VOC emissions from the coating of metal parts. For additional information, contact Richard Valentinetti at the agency, (802) 244-8731.

## Virginia

*Occupational Safety and Health (Regulation)*—The Virginia Department of Labor and Industry has adopted an emergency rule updating the Occupational Safety and Health Administrative Regulations Manual. The rule allows employers adequate time to resolve disagreements with citations without incurring the costs of attorneys' fees. It also allows hazards to be abated more promptly through negotiated settlements, rather than experiencing long delays encountered in judicial proceedings. For further information, contact the Department of Labor and Industry, 13 S. 13th St., Richmond, VA 23219, (804) 371-2327.

*Water Quality*—The Virginia Water Control Board has adopted an emergency rule that establishes a general Virginia Pollutant Discharge Elimination System (VPDES) general permit for storm water discharges associated with industrial activity from heavy manufacturing facilities and light manufacturing facilities. The rule requires certain facilities with point source discharges of storm water associated with industrial activity to submit a permit application under the National Pollutant Discharge Elimination System (NPDES) permitting programs. For further information, contact the Water Control Board, Department of Environmental Quality, P.O. Box 11143, Richmond, VA 23230, (804) 786-9100.

## Washington

*Air Quality (Regulation)*—The Washington Department of Ecology is proposing rules relating to operating permits. The rules govern the state's process for determining and assessing fees supporting the air operating permit program as required under the state and federal clean air acts; govern the determination and assessment of operating permit fees; address tracking procedures for program expenditures and establish a system of periodic fiscal and performance audits for the operating permit program. For further information, contact Catherine O'Neill, Air Quality Program, Department of Ecology, P.O. Box 47600, Olympia, WA 98504-7600, (206) 438-8109.

*Lead (Regulation)*—The Washington Department of Labor and Industries is proposing to amend its safety standards for construction work. The proposal would reduce the permitted level of exposure to lead for construction workers from 200 micrograms per cubic meter of air (200 ug/m$^3$) as an 8-hour time-weighted average to 50 ug/m$^3$. The rule also includes requirements addressing exposure assessment, methods of compliance, respiratory protection, protective clothing and equipment, hygiene facilities and practices, medical surveillance, medical removal protection, employee information and training, signs, recordkeeping, and observation of monitoring. It also establishes an action level of 30 ug/m$^3$ as an 8-hour time-weighted average as level at which employers must initiate certain compliance activities. For further information, contact Suzanne L. Mager, Interim Assistant Director, Department of Labor and Industries, P.O. Box 44620, Olympia, WA 98504-4620, (206) 956-5495, or (206) 956-5552.

## Wisconsin

*Toxic Chemical Release (Bill)*—WI A. 732 (Black) requires additional types of facilities to complete toxic chemical release forms and submit them to the Department of Natural Resources. The bill was introduced on September 9 and referred to the Assembly Committee on Natural Resources.

# New Monomers for Polyester Powder Coating Resins

Larry K. Johnson* and William T. Sade†
Eastman Chemical Company

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An increased emphasis upon the quality of compliant coatings has placed more stringent demands upon formulators of polyester powder coating resins. The ability of formulators to design higher performance resins is limited by the number of useful monomers that are available. When new monomers are considered for powder coating resins, attention is immediately focused upon the effects they impart to the glass transition temperature, $T_g$, and the melt viscosity, as key indicators of powder coating stability and flow during cure. This paper addresses these important variables for a number of glycols and diacids recently introduced for use in polyester powder coating resins.

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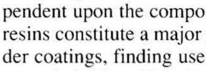
## INTRODUCTION

The requirements for a powder coating composition present the resin designer with a physical dichotomy.[1] In order to form a smooth finish, the powder coating must melt within a particular temperature range to permit timely and ample flow of the polymeric material prior to crosslinking. It is also essential that the formulated coating powders remain in a free-flowing, finely divided state for a reasonable period of time after they are manufactured and packaged. Most polyester resins that are used as binders for powder coatings are amorphous solids. The use of multiple components to produce resins having specialized properties generally reduces their potential for crystallinity. The resulting glass transition temperature ($T_g$) of these resins must exceed the storage temperature to which the formulated powder will be exposed. When storage temperatures reach or exceed the $T_g$, the amorphous resin begins to "cold flow," resulting in agglomeration of the finely divided particles, thereby rendering the powder unsuitable for application. Therefore, the $T_g$ and melt viscosity are important considerations for powder coating resins.[2]

The stability of the finished powder during storage, the flow properties of the enamel during the cure cycle, and the ultimate performance of the crosslinked coating are all dependent upon the composition of the binder resin. Polyester resins constitute a major portion of the binders used in powder coatings, finding use in polyester-isocyanate, polyester-epoxy hybrid, and in polyester-triglycidylisocyanurate (TGIC) systems.[3] These polyester resins are made by well-known polycondensation procedures in which various mixtures of glycols, polyols, diacids, and/or polyacids are fused at elevated temperatures with the elimination of water. An excess of glycol (including polyols) may be used to obtain a polymer that has mostly hydroxyl groups at the terminal positions, or an excess of diacids in combination with polyacids may be used to obtain a polymer that has mostly terminal carboxylic acid groups. Most amorphous polyester powder coating resins use either neopentyl glycol (NG) or ethylene glycol (EG) as the major glycol component in combination with terephthalic acid to provide certain desirable properties to the coating formulation. Smaller amounts of other glycols or polyols and diacids (e.g., isophthalic acid) or polyacids are incorporated to influence performance and physical properties. Such combinations are effective in producing powder coating resins having $T_g$s in the 50-80°C range.

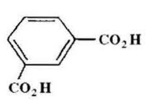

$$HO{-}CH_2{-}\overset{\displaystyle CH_3}{\underset{\displaystyle CH_3}{C}}{-}CH_2{-}OH$$

**Neopentyl Glycol**

$HO{-}CH_2{-}CH_2{-}OH$

**Ethylene Glycol**

**Terephthalic Acid**



**Isophthalic Acid**

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*Chemicals Technical Service and Development Laboratory, P.O. Box 431, Kingsport, TN 37662.

†Resins Market Management, P.O. Box 431, Kingsport, TN 37662.

**Table 1—Effects of Substituting 1,4-CHDA for TPA in Hydroxyl-Functional Powder Coating Resins[a]**

| Acids | Ratio | $T_g$,°C | Mn | Acid Number | ICI Visc. (200°C), P |
|---|---|---|---|---|---|
| TPA | — | 54 | 2426 | 4.0 | 30.0 |
| 1,4-CHDA/TPA | 1/4 | 43 | 2406 | 3.0 | 15.8 |
| 1,4-CHDA/TPA | 2/3 | 50 | 2122 | 3.0 | 4.2 |
| 1,4-CHDA/TPA | 3/2 | 22 | 2363 | 3.5 | 4.4 |
| 1,4-CHDA/TPA | 4/1 | <0 | 2208 | 3.7 | 2.8 |

(a) Basic resin composition: NG/diacids (1.05/0.05/1.0) calculated OH number ˜= 60.

**Table 2—Effects of Substituting 1,4-CHDA for IPA in Carboxyl-Functional Powder Coating Resins[a]**

| Acids | Ratio | $T_g$,°C | Mn | Acid Number | ICI Visc. (200°C), P |
|---|---|---|---|---|---|
| IPA | — | 66 | 1846 | 55 | >100 |
| IPA | — | 68 | 1762 | 61 | >100 |
| 1,4-CHDA/IPA | 1/4 | 56 | 1916 | 53 | 63 |
| 1,4-CHDA/IPA | 2/3 | 48 | 2044 | 87 | 42 |
| 1,4-CHDA | — | 31 | 1790 | 53 | 19 |

(a) Basic resin composition: CHDM/NG/TMP/diacids (4.0/1.0/0.29/6.24).

**Table 3—Use of Ethylene Glycol to Replace Neopentyl Glycol for $T_g$ Enhancement in Hydroxyl-Terminated Polyester Resins[a]**

| EN/NG | $T_g$,°C | Mn | Acid Number | ICI Visc. (200°C), P |
|---|---|---|---|---|
| 0/1 | <0 | 1947 | 5.0 | 1.6 |
| 1/4 | <0 | 2295 | 3.0 | 2.4 |
| 2/3 | <0 | 1898 | 3.2 | 1.6 |
| 3/2 | <0 | 1920 | 1.9 | 1.6 |

(a) Basic resin composition: Glycols/TMP/1,4-CHDA (1.05/0.05/1.0) calculated OH number ˜= 60.

**Table 4—Use of Cyclohexanedimethanol to Replace Neopentyl Glycol for $T_g$ Enhancement in Hydroxyl-Terminated Polyester Resins[a]**

| CHDM/NG | $T_g$,°C | Mn | Acid Number | ICI Visc. (200°C), P |
|---|---|---|---|---|
| 0/1 | <0 | 1947 | 5.0 | 1.6 |
| 1/4 | <0 | 2529 | 3.9 | 3.4 |
| 2/3 | <0 | 2482 | 4.2 | 4.4 |
| 1/1 | <0 | 2473 | 4.9 | 5.6 |
| 3/2 | <0 | 2925 | 3.3 | 7.2 |
| 1/0 | 20 | 2553 | 5.4 | 7.8 |

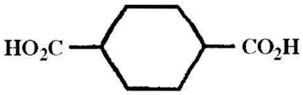
(a) Basic resin composition: Glycols/TMP/1,4-CHDA (1.05/0.05/1.0) calculated OH number ˜= 60.

## RESULTS AND DISCUSSION

In order to fully evaluate the effects of the various monomers for polyester powder coating resins, a series of three resins was selected for modification. The first resin (PC-17-5N) is hydroxyl-terminated (hydroxyl number = 60) and is composed of neopentyl glycol, trimethylolpropane, and terephthalic acid. The second resin (PC-17-4CN) is carboxyl terminated (acid number = 55) while having the same monomer components as PC-17-5N. The third resin (PC-17-4N) is prepared by making a linear hydroxyl-functional polyester that is end-capped with trimellitic anhydride at a lower temperature to provide a resin with a high acid number (95) and with a relatively low polydispersity. This formulation was modified to lower molecular weights to facilitate the low temperature (185°C) end-capping. The result of this modification was a higher acid number (110).

### New Diacids

Although powder coatings are recognized as offering an attractive combination of performance properties, specific performance improvements may be attainable by the incorporation of additional monomers into the polyester resin during the polycondensation reaction. A prime example of a potentially useful monomer is 1,4-cyclohexanedicarboxylic acid (1,4-CHDA). This monomer is commercially available in a ratio of 55/45 *cis-* to *trans-*isomers. Changing this ratio has been shown to have minimal effect on resin properties due to isomerization during resin preparation.[4] In solution coatings and in unsaturated polyester gel coats, the use of 1,4-CHDA in polyester resins provides improved flexibility and resistance to yellowing (as a result of exposure to ultraviolet radiation) compared with aromatic diacids such as isophthalic and terephthalic acids. These benefits are also desirable for powder coatings. Unfortunately, when all the terephthalic acid is replaced by 1,4-CHDA in polyester powder coating resins, the $T_g$ drops from an acceptable level (50-80°C) to a value that is significantly lower (see *Tables* 1 and 2). As a result, such resins have little or no value as the sole crosslinkable moiety in a powder coating formulation.



**1,4-Cyclohexanedicarboxylic Acid**

In order to overcome this detrimental effect on $T_g$, one or more additional monomers must be added in order to compensate for the $T_g$ deficiency. If the purpose of using a monomer such as 1,4-CHDA in a powder coating formulation is to improve resistance to ultraviolet radiation, aromatic diacids or polyacids would not be suitable monomers for increasing the $T_g$ since they have a negative impact on ultraviolet resistance. Because of limited availability of aliphatic diacids, a search for a $T_g$-enhancing glycol was begun.

The first glycol investigated was ethylene glycol (EG). When it was substituted for neopentyl glycol in a basic polyester formulation, no appreciable effect on $T_g$ was de-

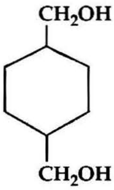
**Table 5—Use of Hydrogenated Bisphenol A to Replace Neopentyl Glycol for $T_g$ Enhancement in Hydroxyl-Terminated Polyester Resins[a]**

| HBPA/NG | $T_g$, °C | Mn | Acid Number | ICI Visc. (200°C), P |
|---|---|---|---|---|
| 0/1 | <0 | 1947 | 5.0 | 1.6 |
| 1/4 | <0 | 2111 | 4.0 | 3.9 |
| 2/3 | 43 | 2051 | 5.7 | 14.0 |
| 3/2 | 62 | 2053 | 7.0 | 36.0 |

(a) Basic resin composition: Glycols/TMP/1,4-CHDA (1.05/0.05/1.0) calculated OH number ≅ 60.

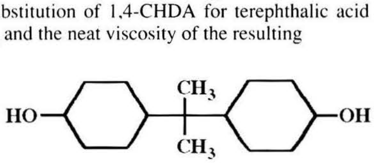
tected (*Table* 3). Note that the resins discussed in this paper follow the trend that $T_g$ increases with melt viscosity for a given resin. Although no specific values of $T_g$ were obtained for this series of resins, there was no trend toward higher melt viscosities with higher EG content.

The next attempt to locate a $T_g$-enhancing glycol focused on 1,4-cyclohexanedimethanol (CHDM). A positive effect was noted when CHDM was substituted for neopentyl glycol (*Table* 4), but the resulting $T_g$ was still not adequate to offset the effect of the 1,4-CHDA. Note that at low $T_g$ values there was a trend toward higher melt viscosity with increasing CHDM content. The effects of the ratio of *trans*- to *cis*-isomer content of the CHDM were not evaluated as this material is supplied commercially as 32% *cis*-CHDM.



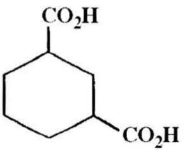
**1,4-Cyclohexanedimethanol**

The investigation then turned to hydrogenated bisphenol A (HBPA). Molecular models reveal that each of the three isomers of HBPA (*cis-cis*, *cis-trans*, and *trans-trans*) are sterically restricted from rotational and conformational changes—factors that would be predicted to enhance $T_g$. When HBPA was incorporated into the basic resin composition in amounts of 50 mole percent or greater (*Tables* 5 and 6), $T_g$s in the normal usable range were obtained. Whereas the substitution of 1,4-CHDA for terephthalic acid lowers the $T_g$ and the neat viscosity of the resulting polyester resin, replacement of neopentyl glycol with HBPA has the opposite effect on these properties. The overall result may be that the flow properties of the powder coating based on this resin may not benefit from the incorporation of the cycloaliphatic diacid. On the other hand, the UV resistance of the coating should be enhanced.



**Hydrogenated Bisphenol A**

A related diacid monomer is 1,3-cyclohexanedicarboxylic acid (1,3-CHDA) which is not commercially available at this time. This monomer appears to be useful for solution coatings in that it offers ultraviolet resistance similar to that of 1,4-CHDA while offering improved solubility and reduced resin viscosity.



**1,3-Cyclohexanedicarboxylic Acid**

In order to investigate its characteristics in a powder coating resin, 1,3-CHDA was substituted for 1,4-CHDA in some of the compositions developed for 1,4-CHDA. Three different types of resins were studied with multiple compositions of each resin type. The results of these comparisons indicate that within experimental error there is little difference in the effects of 1,3-CHDA and 1,4-CHDA on resin $T_g$. *Table* 7 shows the observed effect in hydroxyl-terminated polyester resins, while *Tables* 8 and 9 provide evidence for this phenomenon in carboxyl-terminated polyester resins.

Another important observation can be made from the data presented in *Tables* 7-9. While there was little difference in the $T_g$s they imparted, 1,3-CHDA provided significantly lower melt viscosities than 1,4-CHDA as measured by an ICI cone and plate viscometer. The reduced viscosity was observed in polyester resins having either an excess of hydroxyl groups or an excess of carboxylic acid groups. The effect of this reduction in viscosity translates into improved flow characteristics for the finished powder coating composition.

Because of the relationship observed between 1,4-CHDA and 1,3-CHDA, experiments were conducted to determine if the same behavior occurred between terephthalic acid (1,4-isomer) and isophthalic acid (1,3-isomer). The results are summarized in *Tables* 10-12. In general, the trends are similar to those observed with the cycloaliphatic diacids.

**Table 6—Use of Hydrogenated Bisphenol A to Replace Neopentyl Glycol for $T_g$ Enhancement in Carboxyl-Terminated Polyester Resins[a]**

| HBPA/NG | Acid | $T_g$, °C | Mn | Acid Number | ICI Visc. (200°C) P |
|---|---|---|---|---|---|
| 0/1 | TPA | 58 | 2073 | 103 | 30 |
| 0/1 | 1,4-CHDA | 25 | 1476 | 106 | 10 |
| 3/2 | 1,4-CHDA | 80 | 1254 | 109 | 80 |

(a) Basic resin composition: Glycols/diacids/TMA (2.58/2.25/1.0).

**Table 7—Hydroxyl-Terminated Polyesters Resins Based on 1,4-CHDA and 1,3-CHDA[a]**

| Glycols | Ratio | Acids | $T_g$, °C Onset/Mid | Mn | Mw | Acid Number | ICI Visc. (200°C), P |
|---|---|---|---|---|---|---|---|
| CHDM/HBPA | 4/1 | 1,4-CHDA | 30/33 | 2059 | 6237 | 6.5 | 14.0 |
| CHDM/HBPA | 4/1 | 1,3-CHDA | 36/39 | 2196 | 8162 | 3.0 | 12.0 |
| CHDM/HBPA | 3/2 | 1,4-CHDA | 35/40 | 1433 | 3889 | 5.0 | 8.0 |
| CHDM/HBPA | 3/2 | 1,3-CHDA | 40/42 | 1555 | 3978 | 16.2 | 7.0 |
| NG/HBPA | 1/1 | 1,4-CHDA | 46/51 | 1433 | 5771 | 3.8 | 34.0 |
| NG/HBPA | 1/1 | 1,3-CHDA | 43/49 | 1777 | 4978 | 2.0 | 8.0 |

(a) Basic resin composition: Glycols/TMP/diacid (1.05/0.05/1.0) calculated OH number ≅ 60.

**Table 8—Carboxyl-Terminated Resins Based on 1,4-CHDA and 1,3-CHDA[a]**

| Composition | $T_g$, °C Onset/Mid | Mn | Mw | Acid Number | ICI Visc. (200°C), P |
|---|---|---|---|---|---|
| CHDM/NG/TMP/ 1,4-CHDA | 29/33 | 2367 | 8944 | 60 | 16 |
| CHDM/NG/TMP/ 1,3-CHDA | 28/32 | 2523 | 9397 | 50 | 10 |
| CHDM/HBPA/TMP/ 1,4-CHDA | 44/47 | 2313 | 7759 | 56 | 34 |
| CHDM/HBPA/TMP/ 1,3-CHDA | 42/46 | 2528 | 7688 | 64 | 22 |

(a) Component molar ratio: 4.0/1.0/0.29/6.24.

**Table 9—Carboxyl-Terminated Polyester Resins Based on 1,4-CHDA or 1,3-CHDA and Trimellitic Anhydride**

| Composition | $T_g$, °C Onset/Mid | Mn | Mw | Acid Number | ICI Visc. (200°C), P |
|---|---|---|---|---|---|
| NG/HBPA/1,4-CHDA/ TMA[a] | 75/80 | 1254 | 2459 | 109 | 80 |
| NG/HBPA/1,3-CHDA/ TMA[a] | 74/79 | 1273 | 2503 | 105 | 52 |
| NG/HBPA/1,4-CHDA/ TMA[b] | 54/59 | 1726 | 3779 | 125 | 53 |
| NG/HBPA/1,3-CHDA/ TMA[b] | 54/61 | 1756 | 3483 | 121 | 38 |
| NG/HBPA/1,4-CHDA/ TMA[c] | 49/54 | 1578 | 3018 | 127 | 30 |
| NG/HBPA/1,3-CHDA/ TMA[c] | 51/56 | 1512 | 2781 | 123 | 16 |

Component Molar Ratio:
(a) 1.16/1.74/2.39/1.0.
(b) 1.45/1.45/2.40/1.0.
(c) 1.74/1.16/2.88/1.0.

**Table 10—Hydroxyl-Terminated Polyester Resins[a] Based on Terephthalic and Isophthalic Acids**

| Composition (Ratio) | $T_g$, °C Onset/Mid | Acid Number | ICI Visc. (200°C), P | Mn | Mw |
|---|---|---|---|---|---|
| NG/TMP/TPA (1.05/0.05/1.0) | 51/54 | 4.0 | 30 | 2426 | 8,098 |
| NG/TMP/IPA (1.05/0.05/1.0) | 51/53 | 2.3 | 19 | 2848 | 10,180 |

(a) Calculated OH number ≅ 60.

**Table 11—Carboxyl-Terminated Polyester Resins Based on Trimellitic Anhydride and Terephthalic or Isophthalic Acids[a]**

| Composition (Ratio) | $T_g$, °C Onset/Mid | Acid Number | ICI Visc. (200°C), P | Mn | Mw |
|---|---|---|---|---|---|
| NG/TPA/TMA (2.58/2.25/1.0) | 54/58 | 103 | 30 | 2073 | 5322 |
| NG/IPA/TMA (2.58/2.25/1.0) | 65/69 | 101 | 18 | 1709 | 4515 |

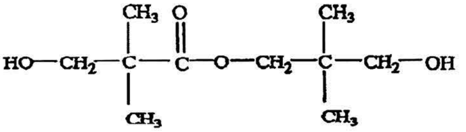
(a) Calculated OH number ≅ 60.

**Table 12—Carboxyl-Terminated Polyester Resins Based on Terephthalic and Isophthalic Acids**

| Composition (Ratio) | $T_g$, °C Onset/Mid | Acid Number | ICI Visc. (200°C), P | Mn | Mw |
|---|---|---|---|---|---|
| CHDM/NG/TMP/TPA (4.0/1.0/0.29/6.22) | Could not make resin—too thick to stir before reaching desired acid number | | | | |
| CHDM/NG/TMP/IPA (4.0/1.0/0.29/6.22) | 68/70 | 65 | >100 | 1864 | 10,860 |

## New Glycols

An example of a monomer that has been shown to provide valued benefits to polyester powder coating resins is hydroxypivalyl hydroxypivalate (HPHP). This glycol is essentially a neopentyl glycol dimer joined by an ester linkage. When substituted for neopentyl glycol in a typical powder coating resin, the higher molecular weight of HPHP reduces the weight percentage of aromatic acid in the polyester. The result is a polymer with more aliphatic and less aromatic character.



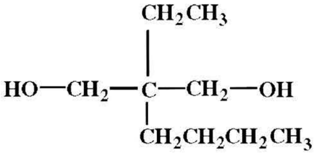
$$HO{-}CH_2{-}\underset{CH_3}{\overset{CH_3}{C}}{-}\overset{O}{\overset{\|}{C}}{-}O{-}CH_2{-}\underset{CH_3}{\overset{CH_3}{C}}{-}CH_2{-}OH$$

**Hydroxypivalyl Hydroxypivalate**

When compared with neopentyl glycol, HPHP provides polyesters with reduced $T_g$s and melt viscosities (*Tables* 13-15). While internally-generated coatings performance data is not yet available for these resins, HPHP reportedly offers slightly better flexibility and weatherability in polyesters than does neopentyl glycol. These characteristics are consistent with expectations from reducing the aromatic content of the polyester.

HPHP contains a highly-shielded internal ester which is resistant to hydrolysis. Because of the high molecular weight of HPHP, polyesters based on this glycol have fewer ester groups that are vulnerable to attack at equal polymer molecular weight than do similar polyester resins based on neopentyl glycol. This characteristic could be responsible for the reported improvement in acid rain resistance of these polyesters compared with those based on neopentyl glycol.

A molecule that is new to most powder resin chemists is 2-butyl-2-ethyl-1,3-propanediol (BEPD), which is now commercially available.



$$HO{-}CH_2{-}\underset{CH_2CH_2CH_2CH_3}{\overset{CH_2CH_3}{C}}{-}CH_2{-}OH$$

**2-Butyl-2-ethyl-1,3-propanediol**

This glycol is structurally related to neopentyl glycol and HPHP in that there are no hydrogen atoms attached to the carbon atom adjacent to the hydroxymethyl (i.e., no beta-hydrogens). The absence of these beta-hydrogens translates into weatherability that is superior to that offered by molecules with beta-hydrogens.[5] This behavior can be explained by a Norrish Type II photochemical cleavage[6] which occurs more readily when R in *Figure* 1 is a hydrogen atom.

However, the aliphatic groups that are attached in the beta-positions differ significantly for BEPD compared with

Table 13—Effects of Substituting HPHP for Neopentyl Glycol in Hydroxyl-Functional Powder Coating Resins[a]

| Glycols | Ratio | Tg,°C | Mn | ICI Visc. (200°C), P |
|---|---|---|---|---|
| NG | — | 54 | 2426 | 30 |
| NG/HPHP | 4/1 | 49 | 2771 | 22 |
| HPHP | — | 26 | 2885 | 4 |

(a) Basic resin composition: Glycols/TMP/TPA (1.05/0.05/1.0), calculated OH number ˜= 60.

Table 14—Effects of Substituting HPHP for Neopentyl Glycol in Carboxyl-Functional Powder Coating Resins[a]

| Glycols | Ratio | Tg,°C | Mn | ICI Visc. (200°C), P |
|---|---|---|---|---|
| NG | — | 59 | 1979 | 12 |
| NG/HPHP | 3/2 | 50 | 1948 | 11 |
| NG/HPHP | 2/3 | 43 | 2005 | 9 |
| HPHP | — | 36 | 1907 | 7 |

(a) Basic resin composition: Glycols/TPA/TMA (3.86/3.36/1.0).

Table 15—Effects of Substituting HPHP for Neopentyl Glycol in Carboxyl-Functional Powder Coating Resins[a]

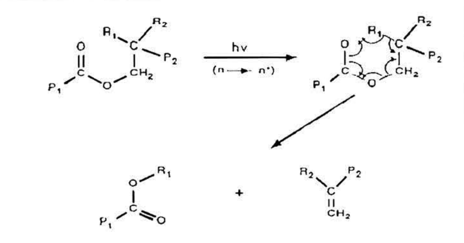
| Glycols | Ratio | Tg,°C | Mn | ICI Visc. (200°C), P |
|---|---|---|---|---|
| CHDM/NG | 4/1 | 68 | 1762 | >100 |
| CHDM/HPHP | 4/1 | 56 | 1599 | 71 |

(a) Basic resin composition: Glycols/TMP/IPA (4.0/1.0/0.29/6.24).

Table 16—Hydroxyl-Terminated Polyesters Based on HPHP and BEPD

| Composition | $T_g$,°C Onset/Mid | Acid Number | ICI Visc. (200°C), P | Mn | Mw |
|---|---|---|---|---|---|
| NG/BEPD/TMP/TPA[a] | 43/47 | 1.4 | 25 | 2683 | 9263 |
| NG/HPHP/TMP/TPA[a] | 43/45 | 3.9 | 17 | 2685 | 8253 |
| BEPD/TMP/TPA[b] | 35/38 | 5.0 | 16 | 2788 | 9883 |
| HPHP/TMP/TPA[b] | 23/26 | 3.0 | 4.3 | 2885 | 7853 |

Resin Molar Ratios:
(a) 0.84/0.21/0.05/1.0, Calculated OH number ˜= 60.
(b) 1.05/0.05/1.0, Calculated OH number ˜= 60.



where

$P_{1,2}$ = polyester polymer

$R_{1,2}$ = H, alkyl

Figure 1—Norrish Type II Photochemical Dissociation Structure

**Table 17—Carboxyl-Terminated Polyester Resins Using HPHP and BEPD as Modifying Glycols[a]**

| Glycols | Ratio | $T_g$, °C Onset/Mid | Acid Number | ICI Visc. (200°C), P | Mn | Mw |
|---|---|---|---|---|---|---|
| NG/BEPD ....... | 4/1 | 55/59 | 109 | 21 | 2031 | 5035 |
| NG/HPHP ....... | 4/1 | 48/54 | 113 | 15 | 2062 | 4347 |
| NG/BEPD ....... | 9/1 | 65/69 | 113 | 36 | 1711 | 4517 |
| NG/HPHP ....... | 9/1 | 57/56 | 101 | 29 | 2223 | 6356 |

(a) Basic resin composition: Glycols/TPA/TMA (3.86/3.36/1.0).

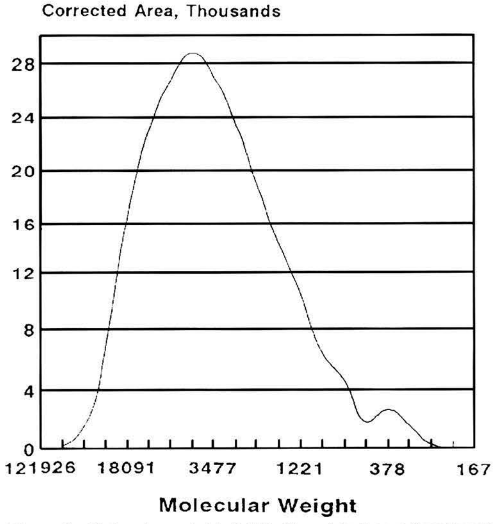


Figure 2—Molecular weight distribution of hydroxyl-functional polyester resin NPG/TMP/TPA/1,4-CHDA (1.05/0.05/0.06/0.4)

**Table 18—Carboxyl-Terminated Polyesters Using HPHP and BEPD as Modifying Glycols**

| Composition | $T_g$, °C Onset/Mid | Acid Number | ICI Visc. (200°C), P | Mn | Mw |
|---|---|---|---|---|---|
| CHDM/BEPD/ TMP/IPA[a] ................ | 59/63 | 61 | 86 | 1684 | 10,688 |
| CHDM/HPHP/ TMP/IPA[a] ................ | 52/56 | 66 | 71 | 1599 | 11,823 |
| CHDM/BEPD TMP/IPA[b] ................ | 59/63 | 69 | 74 | 1805 | 12,573 |
| CHDM/HPHP TMP/IPA[b] ................ | 51/54 | 63 | 58 | 2012 | 11,133 |

Resin Molar Ratios:
(a) 4.0/1.0/0.29/6.25.
(b) 3.0/2.0/0.29/6.25.

neopentyl glycol and HPHP. The latter two molecules contain geminal dimethyl groups, while BEPD contains one ethyl and one butyl group. These longer alkyl chains add hydrophobic character to the polyester and were predicted to reduce the effectiveness with which the polymeric chains pack in the solid resin with a concomitant increase in the free volume of the polymer.

In order to evaluate the effects of BEPD on the $T_g$ and melt viscosity of polyesters, a series of resins was prepared to compare BEPD with HPHP. The results of these comparisons indicate that BEPD provides somewhat higher $T_g$s and melt viscosities than does HPHP. Data in *Table* 16 demonstrates the observed effect in an hydroxyl-terminated polyester powder coating resin, while *Tables* 17 and 18 provide evidence for this conclusion in carboxyl-terminated polyester powder coating resins.

## EXPERIMENTAL

All the resins were prepared by traditional polycondensation techniques.[7] A steam-heated (105°C) partial condenser was used to minimize glycol losses while removing the water formed during the polyesterification reaction.

Progress of the reaction was monitored by dissolving 1-2 g of resin in 125 mL of toluene/methanol (1:1) and titrating with alcoholic potassium hydroxide to provide the acid number values. Melt viscosities were determined with an ICI cone and plate viscometer at 200°C. Approximately 0.5 g resin was placed on the plate, the cone was engaged, and a reading was made after an equilibration period of two minutes.

Glass transition temperatures were determined by differential scanning calorimetry (DSC) with a Mettler TA 3000 Thermal Analysis System according to ASTM Method D 3418.

Molecular weight distribution average determinations were achieved with gel permeation chromatography using polystyrene standards. The method was calibrated using narrow molecular weight standard peak positions covering a molecular weight range of 580 to 1,030,000. Data was presented as molecular weight distribution averages, a time slice report, and a plot of the distribution. The sample was prepared by dissolving 60 milligrams of the resin in 20 milliliters of a solvent which consists of tetrahydrofuran containing 0.3%

Table 19—Elution Data for Molecular Weight Determination of Resin Based on NPG/TMP/TPA/1,4-CHDA (1-05/0-05/0-6/0-4)

| Elution Time | Elution Volume | Molecular Weight | Corrected Area | Cumulative % |
|---|---|---|---|---|
| 19.18 | 18.573 | 121926 | 0 | 0.0000 |
| 19.68 | 19.057 | 72621 | 123 | .0153 |
| 20.18 | 19.542 | 44601 | 1490 | .2997 |
| 20.68 | 20.026 | 28109 | 7222 | 2.1315 |
| 21.18 | 20.510 | 18091 | 16860 | 7.7313 |
| 21.68 | 20.994 | 11833 | 23153 | 17.2071 |
| 22.18 | 21.478 | 7828 | 26947 | 28.7927 |
| 22.68 | 21.962 | 5213 | 28663 | 41.7612 |
| 23.18 | 22.446 | 3477 | 27038 | 54.6866 |
| 23.68 | 22.930 | 2312 | 23333 | 66.3207 |
| 24.18 | 23.414 | 1939 | 18708 | 75.9882 |
| 24.68 | 23.899 | 1569 | 14185 | 83.4989 |
| 25.18 | 24.383 | 1221 | 10312 | 89.0058 |
| 25.68 | 24.867 | 923 | 6429 | 92.8321 |
| 26.18 | 25.351 | 686 | 4655 | 95.6343 |
| 26.68 | 25.835 | 507 | 2794 | 97.0174 |
| 27.18 | 26.319 | 378 | 2640 | 95.4592 |
| 27.68 | 26.803 | 286 | 1603 | 99.5031 |
| 28.18 | 27.287 | 223 | 355 | 99.8693 |
| 28.68 | 27.771 | 181 | 149 | 99.9724 |
| 28.92 | 27.997 | 167 | 113 | 100.000 |
| **Total Area** | **Mn** | **Mw** | **Mz** | **Mo** |
| 6509785 | 2361.8 | 6681.8 | 14546.8 | 5011.2 |

toluene as an internal standard. Both solvents were distilled in glass prior to use. An autosampler (Perkin-Elmer LC-600) was used to inject 20 microliters of the sample solution onto the column (PLGEL mixed bed, 5 micron particles). The sample was eluted at a flow rate of 1.0 milliliter per minute. A Perkin-Elmer LC oven 101 was used to maintain a constant column temperature of 40°C. A Waters 410 Differential Refractometer (sensitivity = 64, scale factor = 20, and internal temperature = 35°C) was used to detect the molecular weight fragments. Typical molecular weight distribution curves are illustrated in *Figures* 2 and 3. The corresponding data is included in *Tables* 19 and 20.

## SUMMARY

All-aliphatic powder coating resins for improved ultraviolet resistance may be prepared using 1,4-cyclohexanedicarboxylic acid (1,4-CHDA) as the sole diacid component

Table 20—Elution Data for Molecular Weight Determination of Resin Based on NPG/TPA/1,4-CHDA/TMA (2-58/1-35/0-9/1-0)

| Elution Time | Elution Volume | Molecular Weight | Corrected Area | Cumulative % |
|---|---|---|---|---|
| 20.35 | 19.373 | 52694 | 0 | 0.0000 |
| 20.85 | 19.849 | 33184 | 136 | .0381 |
| 21.35 | 20.325 | 21361 | 1548 | .7512 |
| 21.85 | 20.801 | 13993 | 5602 | 4.4464 |
| 22.35 | 21.277 | 9285 | 10309 | 13.0424 |
| 22.85 | 21.753 | 6212 | 13969 | 26.2366 |
| 23.35 | 22.229 | 4171 | 14755 | 41.8379 |
| 23.85 | 22.705 | 2798 | 13082 | 56.8006 |
| 24.35 | 23.181 | 2109 | 10371 | 69.2891 |
| 24.85 | 23.657 | 1754 | 7635 | 78.8167 |
| 25.35 | 24.133 | 1396 | 5347 | 85.6692 |
| 25.85 | 24.609 | 1074 | 3666 | 90.4264 |
| 26.35 | 25.085 | 809 | 2504 | 93.5237 |
| 26.85 | 25.561 | 602 | 1533 | 95.6969 |
| 27.35 | 26.037 | 448 | 967 | 96.9701 |
| 27.85 | 26.513 | 337 | 1331 | 98.0395 |
| 28.35 | 26.989 | 259 | 701 | 99.3057 |
| 28.85 | 27.465 | 205 | 282 | 99.7238 |
| 29.32 | 27.909 | 172 | 437 | 99.9999 |
| **Total Area** | **Mn** | **Mw** | **Mz** | **Mo** |
| 2814061 | 2081 | 4765 | 8554 | 3677 |

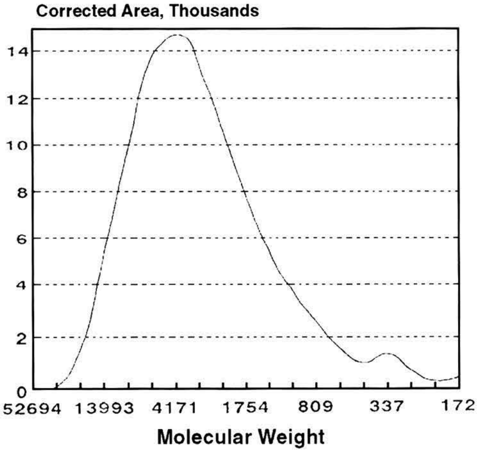


**Figure 3—Molecular weight distribution of carboxyl-functional polyester resin NPG/TPA/1,4-CHDA/TMA (2.58/1.35/0.9/1.0)**

if the formulation contains a $T_g$-enhancing glycol such as hydrogenated bisphenol A (HBPA). The effects of 1,4-CHDA are to reduce both the $T_g$ and the melt viscosity of the resin compared to an analogous resin based on terephthalic acid; whereas, HBPA has the opposite effect.

The melt viscosities of polyester resins for powder coating applications can be further reduced by the incorporation of 1,3-cyclohexanedicarboxylic acid (1,3-CHDA) as the total or partial replacement for 1,4-CHDA without significantly reducing the glass transition temperature of the resins. The resins may be prepared using excess equivalents of either diacids or glycols. Other monomers which enhance the $T_g$, such as HBPA, must also be used in combination with 1,3-CHDA in suitable proportions as to elevate the $T_g$ into a usable range.

Hydroxypivalyl hydroxypivalate (HPHP) reduces both the $T_g$ and melt viscosity of resins into which it is incorporated as a partial replacement for neopentyl glycol. It is most valuable in acid-functional resins due to their inherently higher $T_g$s. Its extended chain length and higher molecular weight as compared with neopentyl glycol has the overall effect of reducing the aromatic content of the resin for improved weatherability and flexibility. The internally hindered ester increases the distance between more vulnerable ester linkages for improved hydrolytic stability.

The use of 2-butyl-2-ethyl-1,3-propanediol (BEPD) as a modifying glycol to replace all or part of the neopentyl glycol or of modifying glycols such as hydroxypivalyl hydroxypivalate has an effect on the $T_g$ that is less than would have been predicted based on its structure. The longer aliphatic side-chains increase the hydrophobic nature of the polyester for improved hydrolytic stability. Weatherability is retained due to the lack of beta-hydrogens.

## Legend

| | |
|---|---|
| BEPD | 2-Butyl-2-ethyl-1,3-propanediol |
| 1,3-CHDA | 1,3-Cyclohexanedicarboxylic acid |
| 1,4-CHDA | 1,4-Cyclohexanedicarboxylic acid |
| CHDM | 1,4-Cyclohexanedimethanol |
| EG | Ethylene glycol |
| HBPA | Hydrogenated bisphenol A |
| HPHP | Hydroxypivalyl hydroxypivalate |
| IPA | Isophthalic acid |
| NG | Neopentyl glycol |
| TMA | Trimellitic acid |
| TPA | Terephthalic acid |
| TMP | Trimethylolpropane |

## ACKNOWLEDGMENTS

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# Polymetallosiloxane Coatings Derived From Two-Step, Acid-Base Catalyzed Sol Precursors for Corrosion Protection Of Aluminum Substrates

T. Sugama
Brookhaven National Laboratory[*]

J.R. Fair
Rice University[†]

A.P. Reed
Las Vegas High School[**]

Fractal polymetallosiloxane (PMS) cluster films were fabricated on aluminum (Al) substrates by the hydrolysis-polycondensation-pyrolysis route of two-step, acid-base-catalyzed sol precursors consisting of N-[3-(triethoxysilyl)propyl]-4,5-dihydroimidazole, metal alkoxides, water, and methyl alcohol. Thin PMS coating films prepared by dipping Al into sol solution provided significant protection for Al against NaCl-induced corrosion. The important factors governing a good protective performance of PMS were as follows: (1) an increase in metal-O-Si linkages with increased treatment temperature; (2) a low degree of water wettability and spreadability; and (3) a specific microstructure geometry, in which the cluster units consisting of symmetrically grown fractal polymer branches were linked together. The third factor contributed to a decrease in stress generated by the shrinkage of films, thereby eliminating crazing and peeling of the film.

## INTRODUCTION

Over the last three years, the characteristics of preceramic polymetallosiloxane (PMS) polymers have been investigated for use as corrosion-protective coatings on aluminum (Al) substrates.[1-3] PMSs were prepared through hydrolysis-polycondensation-pyrolysis synthesis of sol precursor solutions consisting of monomeric organofunctional silane, metal alkoxides, $M(OR)_n$, (where M is Ti, Zr, Ge, or Al; R is $CH_3$, $C_2H_5$, or $C_3H_7$; and n is 2, 3, or 4), methanol, water, and mineral or organic acid as a hydrolysis accelerator. Since a sol is a fluid colloidal solution, the addition of HCl as mineral acid produced a clear sol, thereby helping to form smooth, uniform coatings. The formation of this clear sol was due mainly to HCl-catalyzed hydrolysis of monomeric organosilane and $M(OR)_n$. This hydrolysis transformed the organosilanol compounds containing Cl-substituted end groups, and also the hydroxylated metal compounds. In the second of the reaction stages, dehydrochlorinating and dehydrating polycondensations occurring between these hydrolysates led to the conversion of the sol into a gel. Pyrolytic treatment of the organometallosiloxane gels, as a final stage, induced the formation of PMS network structures, while volatizing the carbonaceous species and eliminating Cl-related by-products from the gel compounds. The removal of these species was recognized from the results of infrared and X-ray photoelectron analyses.

From knowledge of the surface chemistry of Al substrates, we recognize that the outermost surface site of Al is occupied by an Al oxide layer.[4] Such a passivating oxide film is thick (0.4-0.8 nm or more), and contains hydrated polar surface groups, such as -OH and $H_2O$. Considering the pitting corrosion of Al, the following steps[5] will occur: (1) adsorption of halide species onto the Al oxide surface; (2) complexing of Al cations with halide within the oxide lattice to form soluble $AX_4^-$ -(X: Cl, Br, F, or I as the halide) type complexes; (3) diffusion of soluble complexes away from the surface, resulting in a thinning of the passivating oxide layers; and (4) direct reaction of the electrolyte with sufficiently thinned sites Al to produce the pitting corrosion. On the basis of the corrosion mechanisms described, the ideal

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[*] Energy Efficiency and Conservation Div., Dept. of Applied Science, Upton, NY 11973.

[†] Dept. of Chemistry, Houston, TX 77252.

[**] 4505 Maryland Pkwy., Las Vegas, NV 89154.

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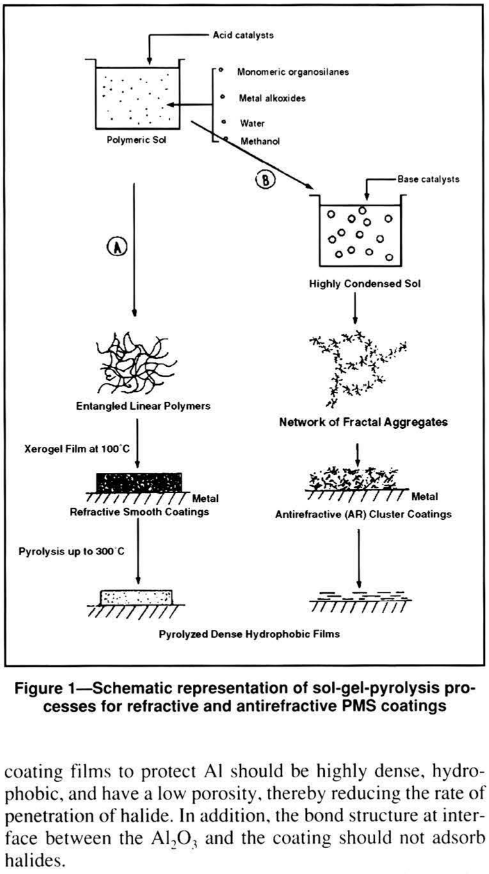

**Figure 1—Schematic representation of sol-gel-pyrolysis processes for refractive and antirefractive PMS coatings**

coating films to protect Al should be highly dense, hydrophobic, and have a low porosity, thereby reducing the rate of penetration of halide. In addition, the bond structure at interface between the $Al_2O_3$ and the coating should not adsorb halides.

From the viewpoint of coating technology, a simple dip- and spin-coating method can be used to deposit the layers of sol precursor solution on Al substrates, and then the polycondensation reaction can be promoted by holding the material for 30 min at 100°C to form the organopolymetallosiloxane xerogel (xero means dry) films. Subsequently, the xerogel films are annealed for 30 min at ≈ 300°C to create the preceramic PMS films.

Ceramic coatings on metallic substrates have not been widely employed for several reasons. First, the coatings must adhere well and have a satisfactory expansion coefficient, especially during temperature cycling. Otherwise, the coating film will separate from the substrates. Second, many ceramic coatings can be applied and processed as coatings only at high temperatures (1000°C) using expensive and sophisticated electron-beam or related methods. Therefore, Al alloys and other low melting point materials are not easily protected. In contrast, the sol-gel technique is very attractive because it is inexpensive and simple; this is particularly true when continuous coating of substrates with a large surface area is required. The preparation of sol precursors at room temperature represents a tremendous technical and economical advantage over other deposition techniques.

Our findings for the PMS coatings derived from acid-catalyzed sol precursors suggested that the following chemical factors play an important role in ensuring that the PMS films afford adequate protection: (1) the selection of monomeric organofunctional silanes which generate smooth, uniform coating layers; (2) the ratio of organosilane to $M(OR)_n$ is critical in achieving spreadability of the sol solution on Al surfaces, and in leaving a minimum amount of organic and crystalline by-products in the organopolymetallosiloxane xerogel; (3) moderate densifications of the Si-O-M linkages in amorphous PMS networks (derived from the 300°C-pyrolytic conversion of the xerogel) are needed to minimize the development of stress cracks in the films; (4) the hydrophobic characteristics of the films; and (5) the formation of the covalent oxane bonds at the interfaces between PMS and Al increase the likelihood of strong adhesion forces.

The microstructure of the film can be altered by varying the rate of polymerization of sol particles; namely, the extent of growth of the polymeric sol in an aqueous medium depends primarily on the pH of the precursor solution. When acid-type catalysts were added to the solution, the clear sol consisted of entangled linear polymers. By contrast, a highly condensed cloudy sol consisting of randomly branched chains was prepared by incorporating base-type catalysts. Xerogel films derived from the acidic sol precursor had a continuous, dense microstructure, while the base-type system showed a specific microstructural geometry in which large fractal clusters were linked together,[6] displaying the properties of an antirefractive surface which means a low rate of the refractive index. The most effective cluster coating for protecting Al against corrosion was that derived from sol in the pH range of 7.0 to 8.0. *Figure* 1 is a schematic representation of

**Table 1—Compositions of Various Sol Precursor Solutions in the pH Range of 7.0 to 8.0**

| TSPI, wt% | $Ti(OR)_4$, wt% | $Zr(OR)_4$, wt% | $B(OR)_3$, wt% | $Al(OR)_3$, wt% | $La(OR)_3$, wt% | $Sn(OR)_2$, wt% | $CH_3OH$, wt% | water, wt% | HCl[a] | NaOH[b] | pH |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 8.3 .................... | 5.5 | — | — | — | — | — | 8.2 | 78.0 | 25.0 | 3.8 | 7.1 |
| 8.3 .................... | — | 5.5 | — | — | — | — | 8.2 | 78.0 | 50.0 | 14.0 | 7.2 |
| 8.3 .................... | — | — | 5.5 | — | — | — | 8.2 | 78.0 | 10.0 | 0.0 | 7.1 |
| 8.3 .................... | — | — | — | 5.5 | — | — | 8.2 | 78.0 | 73.4 | 20.8 | 7.3 |
| 8.3 .................... | — | — | — | — | 5.5 | — | 8.2 | 78.0 | 81.6 | 16.0 | 7.8 |
| 8.3 .................... | — | — | — | — | — | 5.5 | 8.2 | 78.0 | 67.0 | 21.4 | 7.9 |

R: $CH_3$, $C_2H_5$, or $C_3H_7$
(a) Parts per hundred of total mass of TSPI and $M(OR)_n$.

the two different sol-gel-pyrolysis routes for the formation of smooth or fractal coating films.

Based upon this information, the emphasis of our present study was directed toward evaluating various PMS coatings derived from two-step, acid-base-catalyzed sol precursors as protective coatings of Al. The six different PMSs, polytitanosiloxane (PTS), polyzirconosiloxane (PZS), polyborosiloxane (PBS), polyaluminosiloxane (PAS), polylanthanosiloxane (PLS), and polytinsiloxane (PTIS) were synthesized through the B-route (*Figure* 1) of sol precursors consisting of N-[3-(triethoxysily)propyl]-4,5-dihydroimidazole (TSPI) as the monomeric organofunctional silane, $Ti(OC_2H_5)_4$, $Zr(OC_3H_7)_4$, $B(OCH_3)_2$, $Al(OC_3H_7)_3$, $La(OC_3H_7)_3$, and $Sn(OC_2H_5)_2$ as $M(OR)_n$ reactants, methanol, and water over a pH range of 7.1 to 7.9. Our attention was focused on understanding and elucidating the physicochemical factors giving PMS coatings with good or poor protective properties. Our experimental work centered on the microstructural images of the films, changes in the ratios of M-O-Si to Si-O-Si linkages within the PMS structure, and the water wettability of the xerogel (xero means dry) film surfaces as a function of exposure to temperatures up to 300°C. These data were correlated directly with the corrosion-related information, such as electrochemical tests and salt-spray resistances (ASTM B 117) of the coated Al samples. The ultimate goal of our study was to recommend the most effective PMS films as corrosion-protective coatings for Al.

## EXPERIMENTAL METHODS

### Materials

N-[3-(triethoxysilyl)propyl]-4,5-dihydroimidazole (TSPI), supplied by Petrarch Systems Ltd., titanium (IV)-ethoxide $[Ti(OC_2H_5)_4]$, zirconium (IV)-propoxide $[Zr(OC_3H_7)_4]$, trimethyl borate $[B(OCH_3)_3]$, and aluminum isopropoxide $[Al(OC_3H_7)_3]$, obtained from Aldrich, and lanthanum isopropoxide $[La(OC_3H_7)_3]$ and tin (II) methoxide $[Sn(OCH_3)_2]$ from Alfa Products were used as the network-forming monomeric materials. The film-forming mother-liquors, which served as the precursor solutions, were prepared by incorporating the $(TSPI-M(OR)_n$ mixtures into a methyl alcohol/water mixture containing HCl as a hydrolysis accelerator. HCl-catalyzed hydrolysis of the alkoxy groups in the TSPI and $M(OR)_n$ gave a clear precursor solution. To increase the pH of the acidic mother-liquors, NaOH was added as a condensation promoter, except for the TSPI/$B(OCH_3)_3$ system. *Table* 1 shows the compositions of the precursor solutions prepared through such a two-step, acid-base-catalyzed method. The pH of the sol precursors ranged from 7.1 to 7.9.

The metal substrate was 6061 aluminum sheet, containing the following chemicals: 96.3 wt% Al, 0.6 wt% Si, 0.7 wt% Fe, 0.3 wt% Cu, 0.2 wt% Mn, 1.0 wt% Mg, 0.2 wt% Cr, 0.3 wt% Zn, 0.2 wt% Ti, and 0.2 wt% other.

The aluminum surfaces were coated by PMS xerogels in the following sequence. As the first step to remove surface contaminants, the aluminum substrates were immersed for 20 min at 80°C in an alkaline solution consisting of 0.4 wt% NaOH, 2.8 wt% tetrasodium pyrophosphate, 2.8 wt% sodium bicarbonate, and 94.0 wt% water. The alkali-cleaned Al surfaces were washed with deionized water at 25°C for 5 min, and dried for 15 min at 100°C. Then, the substrates were dipped into a soaking bath of precursor solution at room temperature, and withdrawn slowly. The wetted substrates were preheated in an oven for 30 min at 100°C to yield a xerogel coating film which was subsequently pyrolyzed for 30 min in air at temperatures up to 300°C.

### Measurements

The thickness of the PMS film deposited on the alkali-treated Al surfaces was determined using a surface profile measuring system. The changes in surface chemical compositions and states of the alkali-etched Al substrates were measured by X-ray photoelectron spectroscopy (XPS). The contact angle was measured by dropping water onto the surfaces to determine the extent of wettability of the alkali-etched Al and PMS coating film surfaces as a function of etching times and treatment temperatures, respectively. The values of contact angle were measured within the first 20s after dropping the water onto the surface. The surface microstructure of the films formed on Al at 300°C was observed by scanning electron microscopy (SEM). Specular-reflectance Fourier-transform infrared (SR-FTIR) spectroscopy was used to estimate the extent of M-O-Si linkage of the PMS as a function of temperatures. Electrochemical testing for corrosion was performed with an EG&G Princeton Applied Research Model 362-1 Corrosion Measurement System. The electrolyte was a 0.5 M sodium chloride solution, made from distilled water and reagent grade salt. The specimen was mounted in a holder, and then inserted into an EG&G Model K47 electrochemical cell. The tests were con-

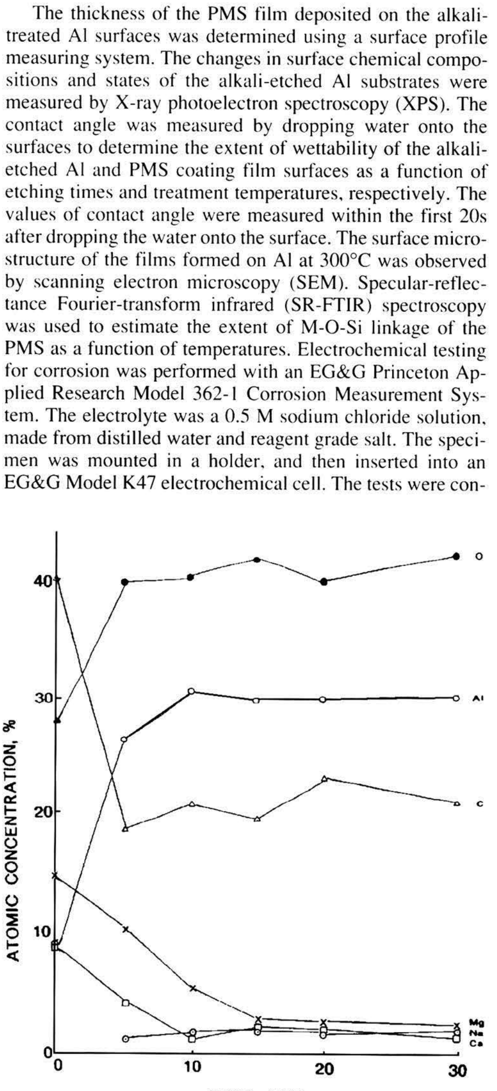

**Figure 2—Changes in XPS atomic compositions of Al substrate surfaces as a function of alkali-etching times**

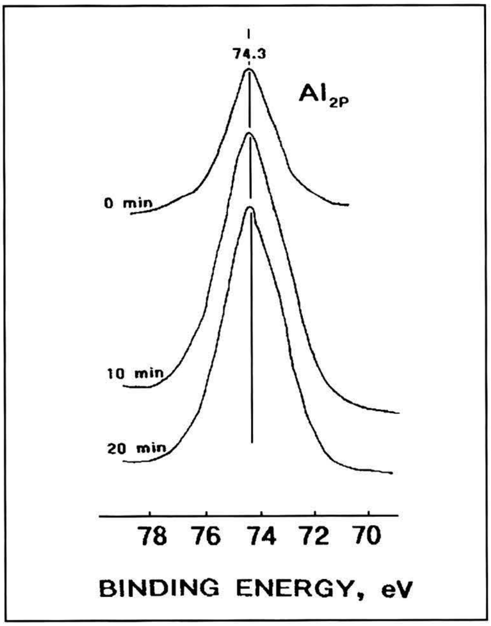

Figure 3—XPS $Al_{2p}$ region for as received, 10 min-, and 20 min-etched Al surfaces

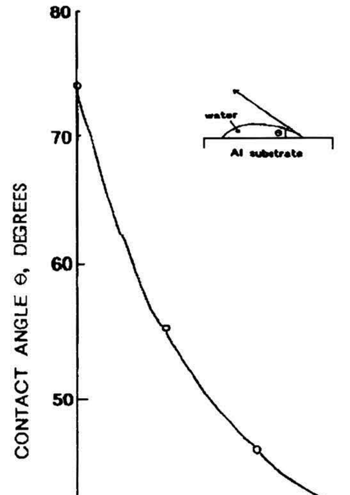

Figure 4—Changes in the contact angle of Al surfaces by water, as function of etching times

ducted in an aerated 0.5 M NaCl solution at 25°C, on an expanded surface area of 1.0 cm$^2$. The polarization curves containing the cathodic and anodic regions were measured at a scan rate of 0.5 mv/sec in the corrosion potential range of -1.0 to -0.4 volts. The salt-spray testing of PMS-coated Al panels (75 mm × 75 mm size) was performed in accordance with ASTM B 117.

## RESULTS AND DISCUSSION

### Surface Preparation of Al Substrates

One important factor contributing to the formation of a continuous, uniform film over the Al is the surface preparation of Al before the sol solutions are deposited. The aim of preparing the surface by oxidizing chemical reagents is not only to eliminate removable chemical contaminants, but also to create a fresh aluminum oxide ($Al_2O_3$) layer which has a strong affinity with the water.[7] Thus, the fresh $Al_2O_3$ surfaces will increase the wetting kinetics of the sol solution.

*Figure* 2 shows the variation of surface elemental compositions as a function of treatment times. These quantitative data for the respective elements were obtained by comparing the XPS $Al_{2p}$, $C_{1s}$, $Mg_{2p}$, $O_{1s}$, $Na_{1s}$, and $Ca_{2p3/2}$ peak areas, which then were converted into the elemental concentrations by using the differential cross sections for core-level excitation. The surface of "as received" Al substrates had ≈ 40 wt% C, ≈ 28 wt% O, ≈ 14 wt% Mg, ≈ 9 wt% Al, and ≈ 9 wt% Ca. Because the Al element belongs to the underlying Al, the as received surfaces are covered with the removable C, as the major contaminant, and Mg and Ca as minor ones. A dramatic change in composition was seen after exposure to the alkali solution for the first 5 min. There was a remarkable decrease in the concentration of C, and a marked increase in Al and O, while the concentrations of Mg and Ca slowly decreased, an additional Na atom migrated from the alkali solution to the top surface of Al. With the extension of alkali-etching time to 10 min, there was a further increase in Al and Na, and a decrease in Mg and Ca; beyond this time, the contents of all elements except Mg seemed to level off. The composition of the surface of 30 min-etched substrates was ≈ 42 wt% O, ≈ 30 wt% Al, ≈ 21 wt% C, ≈ 3 wt% Mg, ≈ 3 wt% Na, and ≈ 2 wt% Ca. The presence of O and Al as the predominant elements revealed that a large amount of $Al_2O_3$ forms at the outermost surface sites of substrates.

To ascertain this information, we investigated the XPS $Al_{2p}$ core-level spectra of the as received, and 10 and 20 min-etched Al surfaces (*Figure* 3). For the scale in the $Al_{2p}$ region, the binding energy (BE) was calibrated with the $C_{1s}$ of the principal hydrocarbon-type C peak fixed at 285.0 eV as an internal reference standard. The $Al_{2p}$ region of the as received substrate surface denoted as 0-min sample had a weak single peak at 74.3 eV, revealing the formation of $Al_2O_3$.[8] The intensity of this peak had significantly increased in surfaces treated by alkali solution for 10 and 20 min. The growth of this peak strongly supported the fact that treatment times of > 10 min create fresh $Al_2O_3$ layers. As we have shown, fresh $Al_2O_3$ surfaces have an extremely high susceptibility to moisture. Hence, the degree of oxidation of Al surfaces can be estimated from the magnitude of wettability of the substrate surface. The most accurate method for esti-

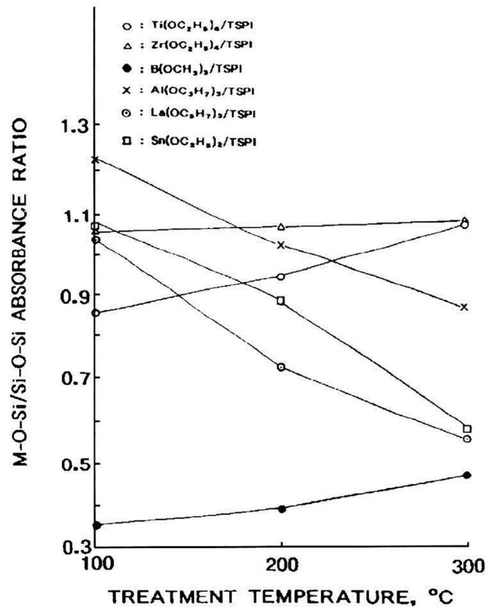

**Figure 5—Absorbance ratios between M-O-Si and Si-O-Si linkages vs treatment temperatures of PMS films**

mating wettability is to measure the advancing water-contact angle, θ (in degrees), on the oxidized Al surfaces. *Figure* 4 plots θ as function of alkali-etching times. Since a low contact angle implies better wetting, the resultant data clearly exhibited that the wetting behavior is improved by a longer etching time. The θ values for the 15 and 20 min-etched surfaces were considerably lower than that for the as received Al surfaces, suggesting that a complete occupation of fresh $Al_2O_3$ layer over the substrate surfaces can be achieved by the etching times > 15 min.

## PMS Coatings

Based upon the previously mentioned information, all the Al substrate surfaces were etched in a hot alkali solution for 20 min, before depositing the sol precursors on the substrates. Although details are not shown, the contact angle of the treated surfaces was too low to be measured. Thus, the sols used in this study have a great affinity for oxide surfaces, and covered them completely. The thickness of various PMS coatings deposited on Al ranged from 0.5 to 0.2 μm.

In our previous study,[1] we found that the extent of densification of M-O-Si linkages formed in the PMS network structures is a key factor in protecting Al against corrosion; PMS coatings which have extremely dense linkages in the xerogel state but show a decrease in the number of linkages with increasing temperature afford poor protection. Because the degree of M-O-Si linkage can be estimated from the difference in absorbance of the IR absorption bands present in the frequency range of 910 and 980 $cm^{-1}$,[10-14] we investigated the absorbance ratios between the M-O-Si and the Si-O-Si bond, ranging from 1000 to 1100 $cm^{-1}$,[15-16] for various PMS coating films exposed to 100, 200, or 300°C. The results are given in *Figure* 5. The data exhibited two different spectral features; one is that the values of the absorbance ratio decreased with an increased treatment temperature, and the other was that the ratio increased as a function of temperature. The PMS systems exhibiting such a decreasing ratio included polyaluminosiloxane (PAS), [$Al(OC_3H_7)_3$/TSPI]; polylanthanosiloxane (PLS), [$Al(OC_3H_7)_3$/TSPI; and polytinsiloxane (PTIS), [$Sn(OC_2H_5)_2$/TSPI]. The PMS systems showing an increasing ratio included polytitanosiloxane (PTS), [$Ti(OC_2H_5)_4$/TSPI]; polyzirconosiloxane (PZS), [$Zr(OC_2H_5)_4$/TSPI]; and polyborosiloxane (PBS), [$B(OCH_3)_3$/TSPI].

In preparing the water-impermeable PMS coating films, the magnitude of wettability of the coatings' surfaces is among the important factors governing good protection. The magnitude of the water-wettability of surface was measured from the average value of the advancing contact angle. *Figure* 6 plots θ as a function of exposure temperatures for various PMS films. At 100°C, the highest θ of ≈ 41° was obtained from the PLS coating, resulting in a low rate of wetting. The PZS coating had the second highest value of θ, while PBS showed the lowest θ, which is the most susceptible to hydrolysis. Of particular interest are the changes in θ value with increased temperature. For instance, the θ of PLS conspicuously drops with increasing temperature. By contrast, a remarkable increase in θ can be seen from the PTS

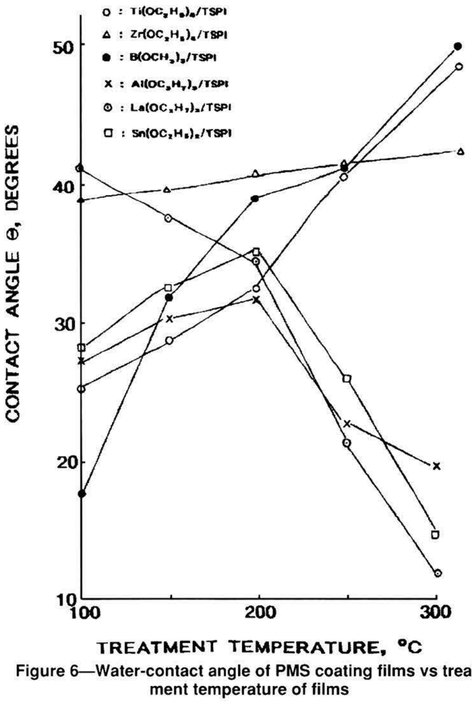

**Figure 6—Water-contact angle of PMS coating films vs treatment temperature of films**

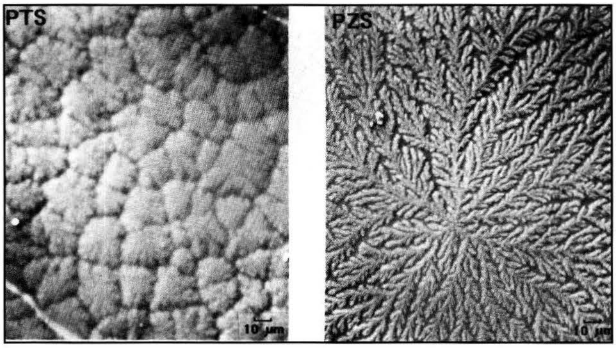

Figure 7—SEM micrographs for fractal PTS (left) and PZS (right) cluster coating surfaces

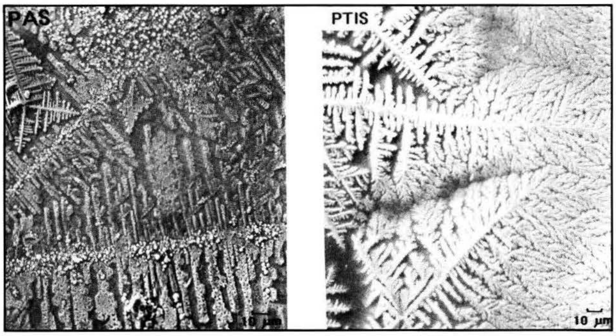

Figure 8—Fractal morphologies of PAS (left) and PTIS (right) coatings

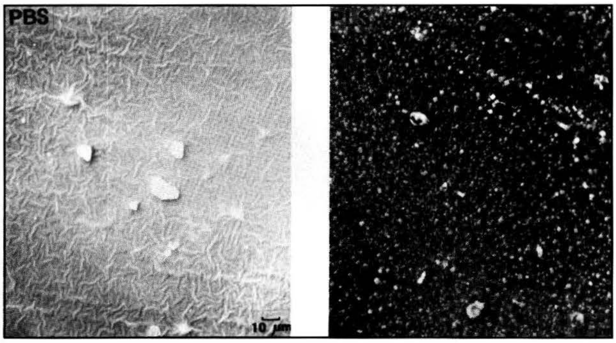

Figure 9—Microstructure of non-fractal PBS (left) and PLS (right) coatings

**Table 2—$I_{corr}$ and Corrosion Rate Obtained from Tafel Calculation for Various PMS-Coated Al Substrates**

| | 100°C | | 200°C | | 300°C | |
|---|---|---|---|---|---|---|
| **System/** | $I_{corr}$ | Corrosion rate | $I_{corr}$ | Corrosion rate | $I_{corr}$ | Corrosion rate |
| **TSPI** | µA/cm² | mpy | µA/cm² | mpy | µA/cm² | mpy |
| 6061-Al | 4.2 | 1.8 | 4.2 | 1.8 | 4.2 | 1.8 |
| $Ti(OR)_4$ | $6.2 \times 10^{-1}$ | $2.6 \times 10^{-1}$ | $1.8 \times 10^{-1}$ | $6.7 \times 10^{-2}$ | $1.6 \times 10^{-2}$ | $1.2 \times 10^{-2}$ |
| $Zr(OR)_4$ | $3.7 \times 10^{-1}$ | $1.6 \times 10^{-1}$ | $9.5 \times 10^{-2}$ | $4.1 \times 10^{-2}$ | $4.7 \times 10^{-2}$ | $2.1 \times 10^{-2}$ |
| $B(OR)_2$ | 2.3 | $9.8 \times 10^{-1}$ | $2.9 \times 10^{-1}$ | $8.3 \times 10^{-1}$ | $5.5 \times 10^{-2}$ | $2.3 \times 10^{-2}$ |
| $Al(OR)_3$ | $5.3 \times 10^{-1}$ | $2.3 \times 10^{-1}$ | $1.8 \times 10^{-1}$ | $7.7 \times 10^{-2}$ | $5.6 \times 10^{-1}$ | $2.4 \times 10^{-1}$ |
| $La(OR)_3$ | $1.5 \times 10^{-1}$ | $6.5 \times 10^{-2}$ | $3.6 \times 10^{-1}$ | $1.6 \times 10^{-1}$ | 1.7 | $7.3 \times 10^{-1}$ |
| $Sn(OR)_2$ | $4.1 \times 10^{-1}$ | $1.7 \times 10^{-1}$ | $2.0 \times 10^{-2}$ | $8.6 \times 10^{-2}$ | 2.6 | 1.1 |

R: $CH_3$, $C_2H_5$, or $C_3H_7$

and PBS coating surfaces. The θ of PZS gradually raises as the temperature increases. The θ values for PAS and PTIS coatings tend to increase from 100 to 200°C; beyond this temperature, θ conspicuously dropped. Such a decline implies that there is an increase in the magnitude of water-wettability and water-spreadability of film. However, there is no evidence whether the enhanced wetting for PAS, PTIS, and PLS coatings treated at high temperatures is due to the conversion into a hydrophilic film or is due to an increase in the surface area of the film caused by the generation of optical microcracks. At 300°C, the PTS, PBS, and PZS coatings having θ of > 40° can be termed hydrophobic films. In fact, after exposure for 30 days to water at room temperature, these coatings did not show any signs of hydrolysis-induced disbandment from the Al. Relating this to the results of M-O-Si to Si-O-Si absorbance ratio, the increase in this ratio as a function of temperature seems to be responsible for the decrease in the θ of the film surfaces, suggesting that an increase in densification of M-O-Si linkages converts hydrophilic films into hydrophobic films.

*Figures* 7-9 show the SEM micrographs of 300°C-pyrolyzed PMS film surfaces. The micrograph of PTS (*Figure* 7, left) showed a dense microstructure of globular clusters (≈ 20 µm size) linked together. The size of cluster for the PZS (*Figure* 7, right) was more than five times that of PTS. The microstructure of the PZS cluster was characterized by the symmetrical growth of multifractal branches. A morphology similar to that of PZS was observed from the SEM examination of single cluster for PTS (not shown). In contrast, the surface structure of PAS and PTIS films showed clusters consisting of the crosslinks of unsymmetrical fractal branches (*Figure* 8); namely, the fractal branches on one side grow abnormally, resulting in highly crosslinked interbranches. But, on the other side, the extent of growth is small, and there are few crosslinking processes. Hence, the difference in microtexture of the growing clusters appears to depend mainly on the $M(OR)_n$ species. No fractal features were found on the surface micrographs of PBS and PLS films (*Figure* 9). The microstructure of PBS films from NaOH-free sol precursor showed a large number of wrinkles, which is due to the internal stresses of film generated by shrinkage. Although the actual data are not presented in any of the figures, the extent of the shrinkage was measured using a hand gauge. The occurrence of shrinking in the cluster-free film clearly verified that the coating films with fractal-based clusters have a low rate of shrinkage, thereby resulting in no crazing or peeling of the films. The impression of PLS film surfaces was one of the incorporation of solid particles (≈ 2 µm size) in the body of the film together with a large number of pits. However, there was no evidence that these particles were associated with Si- and La-related oxide compounds formed at 300°C, nor with any other reaction products.

## Corrosion Resistance

All of the previously mentioned data was correlated with the corrosion protection provided by the PMS coatings on alkali-etched Al substrates. Information on corrosion was obtained from two methods: one is the Tafel extrapolation technique on potentiodynamic polarization diagrams, and the other is the 5% salt spray testing in accordance with ASTM B 117. The former tests were accomplished by exposing PMS-coated Al disks (16 mm diameter × 2 mm thick) to an aerated 0.5 M sodium chloride solution at 25°C. The PMS-coated Al panels (75 mm × 75 mm size) with a X-scribed line which was made through the coating by a sharp instrument, were used for the latter test at 35°C.

*Figure* 10 shows a typical cathodic-anodic polarization curve which plots the polarization voltage (E) versus current (I) (Tafel plot). Based upon this potentiodynamic polarization curve, we attempted to determine the absolute corrosion rates of Al, expressed in the conventional engineering units of milli-inches per year (mpy). Equation (1), proposed by Sterm and Gery,[17] was used in the first step:

$$I_{corr} = \beta_a \cdot \beta_c / 2.303 (\beta_a + \beta_c) R_p \qquad (1)$$

where $I_{corr}$ is the corrosion current density in µA/cm², $\beta_a$ and $\beta_c$ having the units of volts/decade of current refer to the anodic and cathodic Tafel slopes (see *Figure* 10), respectively, which were obtained from the log I vs E plots encompassing both anodic and cathodic regions. $R_p$ is the polarization resistance which was determined from the slope of the polarization curve in the potential range of ± 25 mV from the corrosion potential, $E_{corr}$. When $I_{corr}$ was computed through equation (1), the corrosion rate (mpy) can be obtained from the following equation:

$$\text{Corrosion rate} = 0.13\, I_{corr}\, (EW) / d \qquad (2)$$

where EW is the equivalent weight of the corroding species in g, and d is the density of the corroding species in g/cm³.

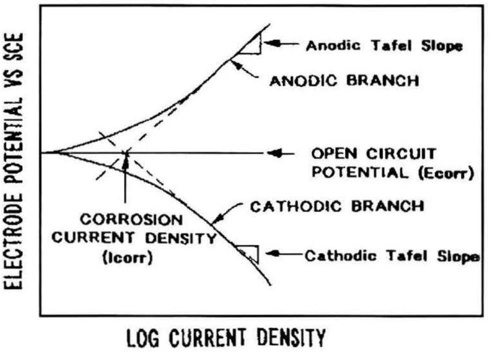

**Figure 10—Typical Tafel plot from a polarization experiment**

*Table* 2 gives the $I_{corr}$ and corrosion rate obtained from this Tafel calculation for various PMS-coated Al specimens. These data are the average of three specimens. The averaged corrosion rate for the 6061-Al substrates, treated at temperatures between 100 to 300°C, was 1.78 mpy, corresponding to the averaged $I_{corr}$ of 4.2 $\mu A/cm^2$. This corrosion rate was significantly reduced by depositing the 100°C-sintered PMS films onto the Al surface (*Table* 2). In particular, the rate of $6.5 \times 10^{-2}$ mpy for the PLS-coated Al was approximately one order of magnitude less than those of other PMS coatings. PZS-coated specimens had the second lowest rate of corrosion, while PBS coatings treated at 100°C exhibited the poorest protection. Relating these findings to the temperature relations for surface wettability described earlier (*Figure* 6), suggests that the ability of 100°C-treated PMS coatings to protect Al against the corrosion depends primarily on whether the film surface is hydrophilic or hydrophobic. PLS with less susceptibility to hydrolysis afforded good protection against corrosion, but PBS, having a high degree of wettability and spreadability, reflecting the low contact angle of surface by water, resulted in poor protection. An increase in treatment temperature to 200°C reduced the corrosion rate of all the PMS coatings, except for PLS that showed an increase in rate. Hence, the effectiveness of PLS as a protective coating declines with increasing temperature. A further reduction in rate, ranging from $1.2 \times 10^{-2}$ to $2.3 \times 10^{-2}$ mpy, was found in 300°C-treated PTS, PZS, and PBS coatings, suggesting that these PMS films contribute significantly to the protection of Al. Although the 200°C-treated PAS and PTIS coatings offer improved protection, treatment at 300°C affects the increase in corrosion rate, thereby changing into a poor protection behavior. Two major reasons for the decline in the protective ability of PLS, PAS, and PTIS at high temperatures are: (1) the decrease in the proportion of M-O-Si to Si-O-Si linkages; and (2) the high degree of wettability of the film's surface by water.

The ability of three PMS coatings, PTS, PZS, and PBS, to protect Al after exposure for up to 30 days in the salt spray chamber was evaluated further. *Figure* 11 shows the changes in $I_{corr}$ for the PTS-, PZS-, and PBS-coated Al specimens as a function of exposure times. During the first seven days, the PTS and PBS specimens showed a slight decrease in $I_{corr}$ value, compared with the unexposed specimens, while the $I_{corr}$ of PZS showed an increase. After seven days, the $I_{corr}$ value increased with the elapsed exposure times up to 30 days for all specimens, although the rate of increase depended on the species of PMS. The lowest rate of increase in $I_{corr}$ was observed in PTS specimens, where the $I_{corr}$ value for 30 days-exposure remained about $10^{-2}$ $\mu A/cm^2$.

When we consider the way in which Al is attacked by chloride, corrosion will occur according to the following mechanisms. The adsorption of $Cl^-$ on the Al oxide and hydroxide surfaces results in the formation of water-soluble Al chloride complex ions, such as $AlCl^{2+}$ and $AlCl_4^-$, thereby promoting the diffusion of $Al^{3+}$ ions and $e^-$ electrons through the oxide film at the anodic reaction sites.[18] Since $OH^-$ ions are generated by cathodic reaction of atmospheric reactants such as $O_2$ and $H_2O$, with the $e^-$ from the anode, $2H_2O + O_2 + 4e^- \rightarrow 4OH^-$,[5] the corrosion reaction between $Al^{3+}$, $Cl^-$, and $OH^-$ ions, $Al^{3+} + 2Cl^- + 2OH^- \rightarrow Al(OH)_2Cl_2^-$, leads to the formation of transitory colloidal complexes which attenuate the oxide film. Once the $Al_2O_3$ layer becomes sufficiently thinned, metallic Al reacts directly with the electrolyte to produce pitting corrosion. The adsorption of $Cl^-$ appears to be a preliminary step to the corrosion pit.

Based upon these concepts of the Al corrosion by $Cl^-$, a low $I_{corr}$ value for PTS specimens corresponds to a low rate of adsorption of $Cl^-$ ions. In other words, PTS coating films display a good barrier which suppresses the rate of penetration and passage of $Cl^-$ reactant through the film. PZS specimens, exposed for 30 days, showed the highest $I_{corr}$ value of $\approx 7.0 \times 10^{-1}$ $\mu A/cm^2$, corresponding to an increase of one order of magnitude over that of the unexposed specimens. Such a high shift in $I_{corr}$ suggests that the protective PZS layer has been degraded. Relating this finding to the SEM examination of film surfaces (*Figure* 7), a possible interpretation for this breakdown is that it is due to the structure linking together large cluster units consisting of extremely well developed fractal branches; this resulting structure of the PZS film may contain numbers of $NaCl^-$ penetrable pores. Thus, the NaCl solution penetrates through the porous structure of PZS coatings during long-term exposure, and can

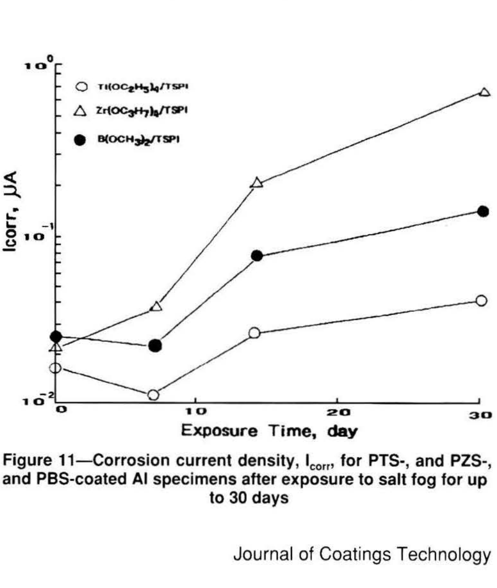

**Figure 11—Corrosion current density, $I_{corr}$, for PTS-, and PZS-, and PBS-coated Al specimens after exposure to salt fog for up to 30 days**

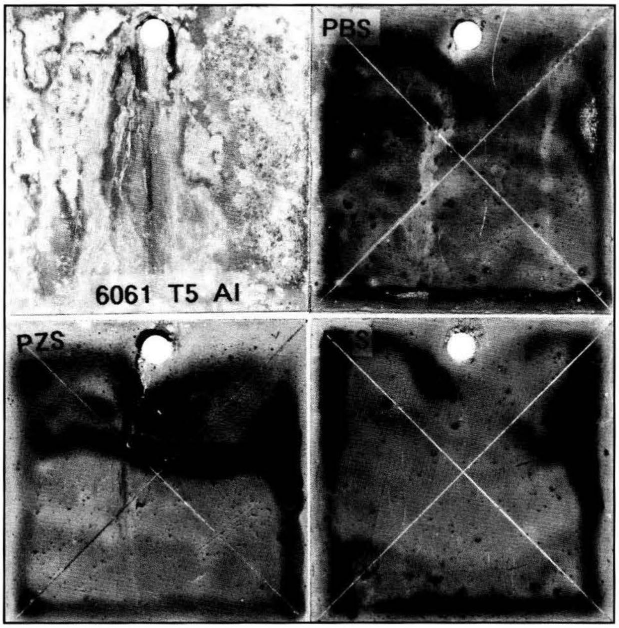

**Figure 12—1200 hr-salt spray resistance of PBS- (right of top), PZS- (left of bottom), and PTS- (right of bottom) coated Al specimens, compared with corroded Al substrate (left of top) after exposure to salt fog for 48 hr**

reach the underlying Al surfaces. The coating still affords protection, as indicated by the $I_{corr}$ of $10^{-1}$ μA/cm$^2$, although the presence of porous structures appears to be undesirable. No large shifts for PTS strongly indicate that the film had not significantly changed during exposure.

*Figure* 12 shows the state of the PMS-coated specimens after salt spray tests for 1200 hours. A scribe of the panels to bare metal is meant to simulate mechanical damage of the coatings, where failure can begin easily. The delamination of coating film from the Al substrate corresponds to the loss of adhesion of the film at the defect, and is the major reason for the scribe. Three major failures were generally looked for in evaluating the results from salt-sprayed specimens: (1) the degree of blistering of the film; (2) traces of white rust stain which represent corrosion of the underlying Al; and (3) the delamination of film from Al. The entire surface of the control specimen (denoted as 6061 T5 Al) without a PMS coating was corroded after exposure to the salt fog for only 48 hours. In contrast, the deposition of PMS coatings to the Al contributed remarkably to protecting Al from salt-induced corrosion during exposures of 1200 hours. Two features considered as failures of the protective coatings were observed in PBS specimens; one was the appearance of some white rust stains surrounding the defect, and the other refers to the partial peeling of film from the substrate, which can be seen at the right and bottom sites of the panel (*Figure* 12, right of top). No signs of peeling and delamination were seen on the PZS- and PTS-coated specimens (*Figure* 12, left and right of bottom). Since both the PZS and PTS films can be categorized as fractal cluster coatings, it is possible to assume that peeling of the non-cluster PBS-coating might be associated with its disbandment brought about by a high internal stress generated by the shrinkage of film. This finding strongly verified that the specific microstructural geometry, in which the fractal clusters were linked together, plays an important role in providing stress-free PMS coatings. Although the PZS specimen showed a trace of rust stain flowing from the defect, there was no blistering of the coating. An excellent condition, in which there was no rust stain, no peeling, and no blistering, was identified on the PTS-coated specimens. Therefore, the size of cluster unit might also be an important factor governing the protection against corrosion by PMS coatings; namely, a PTS coating consisting of a packed structure of cluster units having a moderate size of $\approx 20$ μm displays a better protective performance than that of the PZS coatings in which the clusters come together to have a large overall size of > 100 μm.

## CONCLUSION

Various polymetallosiloxane (PMS) polymers, such as polytitanosiloxane, polyzirconosiloxane, polyborosiloxane, polyaluminosiloxane, polylanthanosiloxane, and polytinsiloxane were synthesized through the hydrolysis-polycondensation-pyrolysis route of two-step, acid-base catalyzed sol precursors consisting of N-[3-)triethoxysilyl)propyl-4,5-dihydroimidazole (TSPI) as monomeric organofunctional silane, metal alkoxides, $M(OR)_n$, [M: Ti, Zr, B, Al, La, and Sn, R: $CH_3$, $C_2H_5$, and $C_3H_7$, n: 2, 3, or 4], methanol, and water, at pHs from 7.1 to 7.9. The synthesized PMS polymers were then evaluated as corrosion-protective coatings of aluminum substrates. The PMS-coated Al specimens were prepared by dipping the alkali-etched Al into the sol precursor solutions, then preheated at 100°C to form xerogel films.

Subsequently, xerogel-coating films were pyrolyzed in air at 300°C for 30 min.

A microstructural geometry of 300°C-treated PMS coating surfaces, in which fractal clusters were linked together, was characteristic of PTS, PZS, PAS, and PTIS coatings, while no fractal features were seen on the PBS and PLS coatings. The surface microstructure for the latter films showed a wrinkled and grained texture caused by thermal shrinkage of film and the formation of oxidized particles in the film layers, respectively.

The following four factors played an essential role in improving the protective ability of PMS coatings on Al substrates: (1) the increase in densification of M-O-Si linkages in the PMS network structure with an increased treatment temperature from 100 to 300°C; (2) the minimum susceptibility of film surfaces to water-wetting behavior; (3) the interlocking morphology between cluster units consisting of symmetrical fractal branches; and (4) the dense microstructure of clusters of ≈ 2 μm entirely covering the metal substrate. The major contribution of factors (3) and (4) was to minimize the stress generated by the shrinkage of films, so that there was no crazing or peeling. Polytitanosiloxane (PTS) was identified as the most effective cluster-coating film which exhibited all of these important factors.

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# Coatings Characterization by Thermally Stimulated Current (TSC) and Relaxation Map Analysis (RMA)

C. Michael Neag
The Glidden Company[*]

J.P. Ibar
Solomat TA Instrumentation[†]

J.R. Saffell and P. Denning
Solomat Mfg. Ltd.[**]

Thermally stimulated current (TSC) and relaxation map analysis (RMA) were used to characterize the molecular behavior of several coating systems. The experiments were designed to test the instruments utility for problem solving, product development, and research in the coatings industry. The results obtained using a TSC/RMA Spectrometer provided information on the glass transition temperature ($T_g$), general morphology, and relative cure levels for container coatings, powder coatings, and a coating for plastic substrates; all of the experiments were completed using coatings over typical substrates. RMA analysis and the resulting degree of disorder (DOD) values were used in quantifying differences in the level of cure for powder coatings and a coating for sheet molding compound (SMC) plastic substrates.

## INTRODUCTION

Dielectric analysis (DEA) is a convenient, nondestructive test that relates molecular motions observed in an electrical field to a variety of polymeric properties. Although the technique was first used in polymer science in 1934, the lack of high quality instrumentation limited the applicability of the technique. Recent advances in instrumentation have rekindled interest in dielectric analysis and the technique is being touted for its ability to produce valuable information about the fundamental physical state of materials. Applications in coatings science include the characterization of new materials and processes, cure studies, film formation studies, formulation optimization, applications development, performance prediction, competitive product evaluation, and QC/QA applications.

Because current engineering limitations prohibit the characterization of the complete continuum of dielectric properties, there are a number of commercial dielectric analyzers available—each offering advantages in their potential range of applications. The operating principle of all of these instruments is the same: a polymeric system is polarized in an electrical field and the time, temperature, or frequency dependent molecular dielectric relaxations are monitored. The changes in dielectric behavior—permittivity, conductance, or polarization current—with changing temperature serve as molecular probes, which correlate with the changing chemical and physical states of the material.

Commercially available instruments may be divided into two broad categories: those polarizing samples with an alternating current (ac) and those polarizing samples using a direct current (dc). Dielectric analyzers employing an ac electrical field measure two fundamental electrical characteristics of a material—capacitance and conductance. Capacitance correlates to a material's ability to store electrical charge and conductance correlates to its ability to transfer electric charge.

Instruments which polarize materials in a dc field represent a relatively new approach and are the focus of this report. In this approach—referred to as thermally stimulated current (TSC)—the dipole movements or low molecular weight compounds are polarized (oriented) in a dc field above the main transition temperatures of the sample. A more detailed description of TSC can be found elsewhere in

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[*] ICI Paints World Group, 16651 Sprague Rd., Research Center, Strongsville, OH 44136.
[†] Glenbrook Industrial Park, 652 Glenbrook Rd., Stamford, CT 06906.
[**] Bristol, United Kingdom.

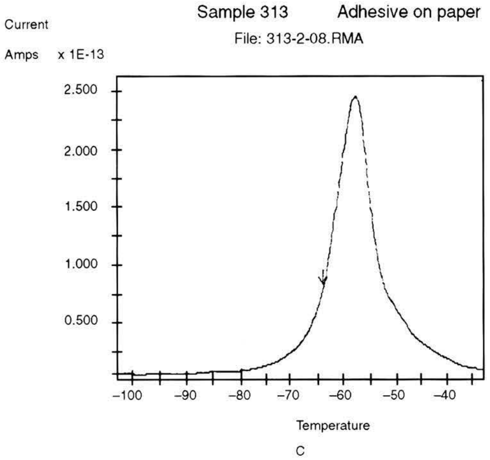

**Figure 1—TSC graph of a pressure sensitive adhesive (PSA) on a paper substrate, in the $T_g$ region**

the book published by Alain Bernes and Colette Lacabanne, entitled *Order in the Amorphous State of Polymers.*[1] In a typical TSC experiment, a high-voltage stabilized dc supply is used for polarizing the sample above its main transition temperature. The sample is heated at a constant rate to the polarization temperature under an electric field of about four million volts per meter of thickness. The sample is kept there for a prescribed time and then cooled down at a controlled rate to -150°C. At this point, the external field is removed and the sample heated at a constant rate through the polarization temperature. An electrometer connected to the sample records the current created as the material depolarizes. This thermally stimulated current marks the molecular motion within the material. The rate of depolarization corresponds to the relaxation times of these motions, providing an opportunity to study the fine morphological characteristics of a material. The current peaks recorded this way correlate well with the transition temperatures measured by mechanical relaxation, DSC, or conventional (ac) dielectric spectroscopy. *Figure* 1 shows the TSC depolarization peak for a pressure sensitive adhesive laid on a paper substrate, showing the $T_g$ of the material.

Relaxation map analysis (RMA) is a powerful extension of TSC technique which involves the repetition of a modified TSC type experiment over a range of polarization temperatures. The depolarization peak observed in a typical TSC experiment appears to consist of a series of overlapping, unresolved, relaxation peaks. By varying the polarization temperature and measuring the depolarization current over a narrow range of small (less than 10°C) temperature "windows," these individual relaxation modes can be isolated. The elementary retardation time $\tau_i$ is given by:

$$\tau_i = P(T) / J(T)$$

where J(T) is the depolarization current at temperature T, P(T) is the polarization of the material at T; J(T) = dP/dT. The smaller the window size used in an experiment, the greater the resolution (or "filtering") and the more likely an individual or "Debye"[9] relaxation will be isolated. The experimental process, known as "thermal windowing," is illustrated in *Figure* 2 and covered in greater detail elsewhere.[1]

The temperature dependence of the resolved elementary relaxation time $\tau_i(T)$ follows either an Arrhenius equation (*Figure* 3), or a free volume equation, depending on whether the relaxation modes relate to dipolar motions or trapped free electrons in the free volume of the material.[10]

A relaxation map (log τ vs 1/T) provides a way to monitor the influence of formulation and processing variables on a material's molecular behavior. A relaxation map formed this way can help deduce the relationship between an experimental variable like molecular weight, chemical structure, or thermodynamic history, and a material's fundamental molecular properties, including the enthalpy of activation ΔH (slope of the Arrhenius line), the entropy of activation ΔS

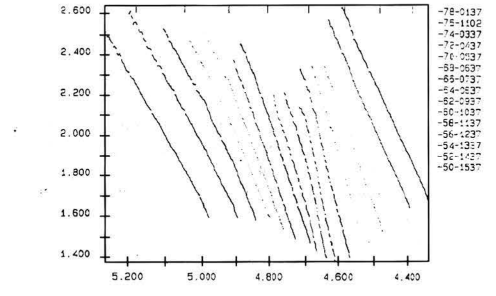

**Figure 2—Principle of "thermal-windowing" to deconvolute the global peak observed at transitions into elementary relaxation components**

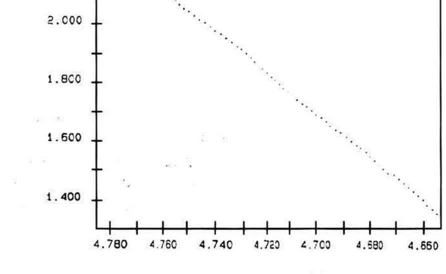

**Figure 3—Arrhenius representation of the relaxation behavior for a single relaxation time**

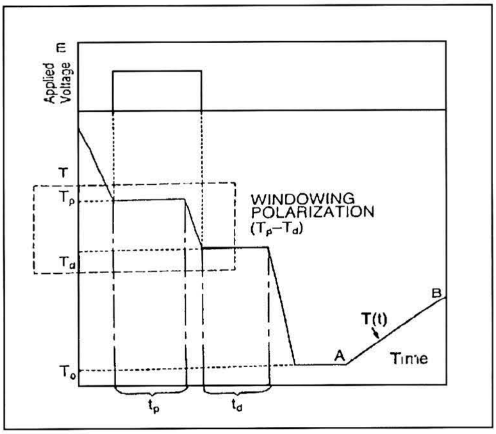

**Figure 4—Relaxation map for a PSA on paper substrate for the $T_g$ transition**

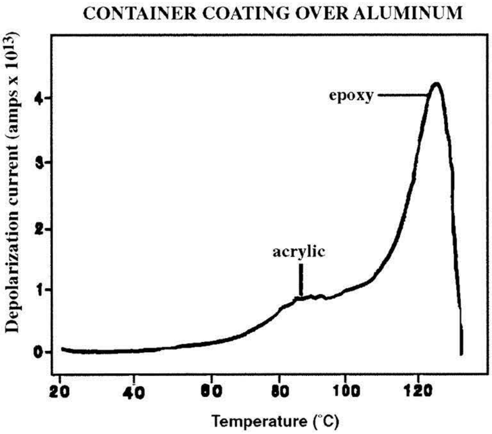

**Figure 5—TSC depolarization current for a container coating over aluminum can stock**

(from the intercept of the Arrhenius line), and (for the free volume relaxations) the coefficient of free volume expansion and the temperature of zero mobility. *Figure* 4 shows the relaxation map for a thin layer of an adhesive on a paper substrate.

The results can be conveniently summarized by the degree of disorder number (DOD), which transcribes the degree of coupling between the various relaxation modes participating in a transition such as $T_g$. The DOD number varies with the degree of cure, the presence of crosslinks, the molecular weight, and the nature of the interactions of the dipoles relaxing at the transition analyzed, providing an interesting opportunity to quantify the properties of the amorphous phase. These concepts are covered in greater depth in several other references.[1-8]

In this study, four sets of experiments were designed to provide a cursory overview of the instrument's capabilities. The coating systems analyzed included two waterborne systems, a solvent-borne system, and an appliance powder coating. The key objectives of this study were to determine the instrument's ability to provide information about coatings directly on typical substrates and to demonstrate the utility of the instrument for cure studies.

## EXPERIMENTAL

TSC experiments were carried out in a Model 41000 TSC/RMA Spectrometer. Temperature ranges and applied voltages varied from sample to sample; polarization temperatures ($T_p$) were taken randomly below, at, or above major transitional regions. Simple TSC experiments were carried out at a heating rate of 7°C/min in a helium atmosphere. In some instances (plastic coatings), incompletely cured materials were heated through the polarization temperature in the instrument, increasing the degree of cure and making conclusions based on RMA results somewhat uncertain. Window polarization experiments for RMA were completed using a window width of 5°C (i.e., $(T_p - T_d) = 5°C$).

**Table 1—Depolarization Current Intensity for Powder Coatings Baked for Different Intervals at 193°C**

| Baked Time (min) | Peak Intensity (Femtoamps) |
|---|---|
| 5 | 1000 |
| 10 | 96 |
| 15 | 77 |
| 20 | 6 |

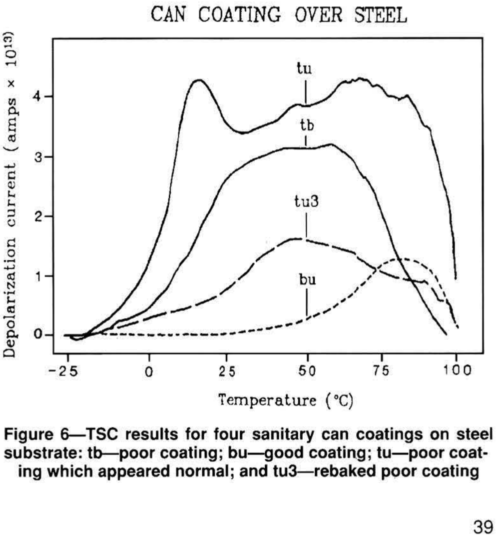

**Figure 6—TSC results for four sanitary can coatings on steel substrate: tb—poor coating; bu—good coating; tu—poor coating which appeared normal; and tu3—rebaked poor coating**

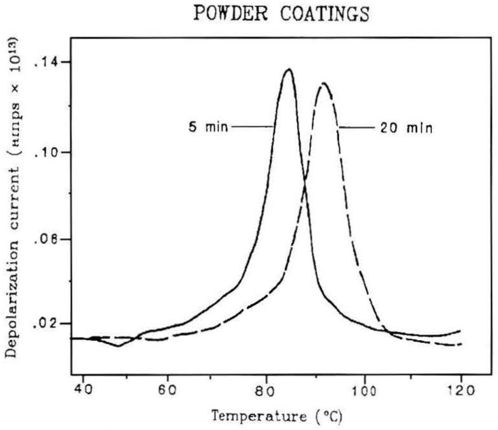

**Figure 7—TSC coplot of $T_g$ region of the powder coatings after 5- and 20-min bakes at 193°C**

**Beverage Container Coatings:** This portion of the study was designed to determine if the instrument could produce satisfactory results for very thin coatings over aluminum and, if so, distinguish between various cure levels. Cans were coated and baked at 204°C. Samples were punched from can bottoms using a 1 cm punch; total film thicknesses were estimated at less than 0.025 mm. The sample was polarized at 125°C.

**Sanitary Can Coatings:** This experiment was designed to distinguish between epoxy based coating samples, which had either passed or failed a production quality control (QC) test. In tests for these coatings, a properly cured coating remains transparent after immersion in warm water. If the coating becomes opaque after immersion, the coating fails the test. TSC experiments were used to identify differences in samples punched from clear (good) and opaque (poor) regions of production cans. Good samples (labeled bu) were removed from a can which passed the QC test (i.e., turned opaque after immersion). The poor samples were removed from cans exhibiting opacity after testing. One sample was removed directly from the opaque region (labeled tb) and another (labeled tu) was removed from an area immediately adjacent to the opaque region on the "bad" can.

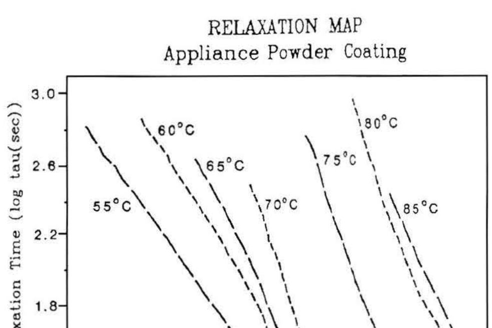

**Figure 8—Relaxation map over the glass transition region of the powder coatings baked 5 min at 193°C**

**Powder Coatings:** This experiment was designed to test the sensitivity of the instrument to different cure levels over a thick metal substrate. An epoxy/polyester appliance coating was sprayed over a Bonderite 1000 steel substrate and cured for 5, 10, 15, and 20 min at 193°C in a forced air oven. The area of these samples was approximately 1 cm$^2$.

**Plastic Coatings:** Two acrylic urethane systems were used in these experiments. The addition of a catalyst distinguished the two systems. One system (labeled H) included a catalyst, while the other (labeled E) did not. Each sample was applied over SMC and cured. These experiments were designed to quantify differences in $T_g$, cure levels, and adhesion levels.

## RESULTS AND DISCUSSION

**Beverage Can Coatings:** Although problems related to film thickness (c. 0.025 mm) and the can's curvature proved quite troublesome, excellent results were obtained for the sample baked at 204°C. In general, simple TSC results produced a DMA-like response for this coating. *Figure* 5 shows TSC peaks at about 85°C and 120°C. Well controlled studies in similar materials using dynamic mechanical analysis and transmission electron microscopy have shown that the first peak corresponds to a low temperature acrylic component and the second peak to an epoxy-acrylic component.

Other experiments revealed a β transition centered around -45°C which probably stems from the motion of side groups pendant to the backbone. The position and relative intensity of these peaks were very similar in the two coatings.

**Sanitary Can Coatings:** TSC analysis indicated that epoxy-based can-end coatings which failed QC tests in production were undercured. *Figure* 6 shows the results for three samples. The large peaks probably coincide with segmental motion at the $T_g$ of each sample. The depolarization curves labeled tb, bu, and tu describe respectively the poor coating, the good sample, and a "good" sample taken from an area immediately adjacent to the poor area. The curve labeled tu3 illustrates the effect of rebake on the poor sample.

Generally, the higher the depolarization peak temperature, the higher the $T_g$, and the greater the extent of cure. By using this crude measure of the extent of cure, the lower relative peak position in the problem sample (tb) indicates that it was undercured in comparison to the good sample (bu). Moreover, the shift in the peak position from roughly 15°C (tu) to about 50°C (tu3) after rebake further corroborates the conclusion that the problem coating was undercured. Note that the polarization temperatures were somewhere within the apparent glass transition region rather than above the $T_g$. Generally, the $T_p$ is selected above the major transi-

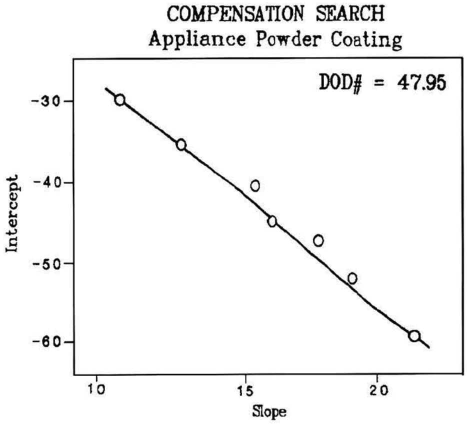

Figure 9—Compensation search (multiple linear regression) for the powder coatings cured 5 min at 193°C

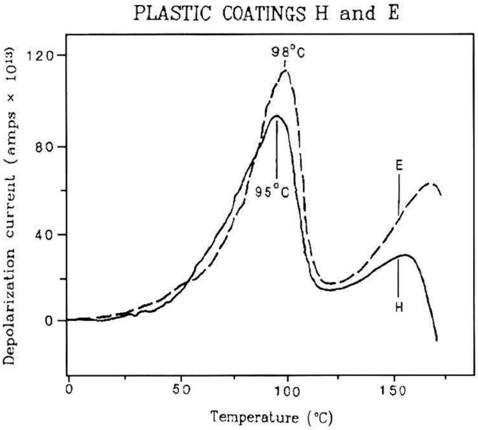

Figure 10—TSC depolarization curves for the standard plastic coatings formulation (E) and the modified (H) formulation

tions (e.g., $T_g$) in order to orient all the dipoles in the transition regions. The effects of choosing $T_p$ within the glass transition region are unclear. The analysis of identical materials by DSC provided no indication of any difference in cure or any other pertinent morphological information.

**Appliance Powder Coating Bake Ladder:** Results obtained by both TSC and RMA provided valuable insight into cure behavior of appliance powder coatings. *Figure* 7 shows depolarization peaks observed in simple TSC experiments for powder sprayed over a steel substrate and baked for 5 and 20 min at 193°C. The peak temperature increased from 83°C after the short bake to 91°C after the longer bake. Since this increase in relative peak position corresponds to an increase in $T_g$, it indicates that the degree of cure is increasing with bake time. Window polarization experiments revealed quantifiable differences in the degree of cure obtained using four bake schedules. The results shown in *Figure* 8 were obtained using window widths of 5°C (i.e., $T_p - T_d = 5$°C) and an 85°C polarization ($T_p$) temperature.

The relaxation peaks—probably correlated with $T_g$—once again increase in temperature as the bake time increases, strongly suggesting that crosslink density had increased with bake time. Any change in the degree of cure with bake time should also produce a change in the peak depolarization current. This occurs because an increase in crosslink density will restrict segmental motion and reduce the number of dipoles which align in the electrical field; thus, if a system has a relatively high crosslink density, the total depolarization current will be low. As *Table* 1 shows, the peak polarization current intensity decreases by some two orders of magnitude as the bake time increases, strongly suggesting an increase in crosslink density.

RMA windowing experiments also yielded a quantifiable difference in the apparent level of cure in two of these samples. RMA analysis for the 5- and 10-min cured samples revealed that the DOD for the sample cured 5 min was 47.95 while the sample cured for 10 min was 44.84. In these experiments, the DOD values are normalized to fit a scale of 1 to 100 + 0.1 with lower values indicating lower system entropy. Thus, the lower DOD values in the 10-min cured sample indicate less entropy, higher crosslink density, and a higher degree of cure. A one unit difference in DOD is considered significant; total error was less than 0.2 units for the seven relaxation curves used in these experiments. A typical compensation search plot is shown in *Figure* 9.

**Coatings for Plastics:** TSC/RMA results strongly suggest that the addition of the catalyst alters the crosslinking chemistry and the total degree of cure reached under standard cure cycles. A comparison of the two systems reveals that the $T_g$ is 3°C lower in the amine-added system. Surprisingly, the total depolarization current was significantly lower in sample H, even though the $T_g$ was lower. Normally, a decrease in $T_g$ would imply less crosslinking, greater dipolar mobility, and a consequent increase in depolarization current. These results show the opposite, suggesting that significant changes in the crosslinking chemistry occurred with the addition of the catalyst—most likely affecting both the nature of the crosslink and the total degree of cure. In particular, the decrease in depolarization current with the addition of the amine indicates a more highly crosslinked network. If these changes in chemistry carry through to the coating-substrate interface, then it is likely that the adhesive bond formed between the coating and substrate has also improved. TSC results for the two samples are compared in *Figure* 10.

RMA windowing experiments for the two relaxation modes observed in simple TSC experiments—one centered about -50°C and another at about 95°C—revealed little difference in the activation energies obtained for either the low or high temperatures process.

## CONCLUSION

The TSC/RMA spectrometer provides a sensitive technique for characterizing the glass transition temperature, fine morphological features, and relative cure levels in a variety of coatings. Excellent results for a variety of coatings were obtained over aluminum, steel, and SMC. The instrument also yielded quantifiable differences in the level of cure for powder coatings and an in-mold plastic coating.

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# Evolution of the Protective Mechanisms Of Zinc-Rich Paints During Atmospheric Exposure

S. Feliu Jr., M. Morcillo, J.M. Bastidas, S. Feliu
Centro Nacional de Investigaciones Metalúrgicas, CSIC*

The results of weathering and electrochemical tests were integrated in order to study the evolution of the protective mechanisms of zinc-rich paints (ZRP) over time.

After three years of atmospheric exposure, some of the tested ZRP coatings still maintained a great part of their original capacity for providing cathodic protection. The paints formulated with the ethyl silicate vehicle stand out in this respect. On the other hand, an important improvement of the barrier effect is shown, especially with the paints formulated with the epoxy-polyamide vehicle.

## INTRODUCTION

It is well known that the protective ability of a zinc-rich paint (ZRP) is mainly based on two mechanisms: at the start of exposure, the ZRP coating provides cathodic protection to the base steel, but its effect decays with time and is gradually displaced by a barrier effect.[1,2] While the former mechanism depends on the galvanic action of zinc particles in electrical contact with each other and with the base steel, the latter is enhanced by the accumulation of zinc corrosion products within the coating. It is obviously desirable for the cathodic protection stage to be extended as long as possible and for the barrier effect to consolidate as soon as possible.

Saline solutions are commonly used in the laboratory for immersion tests of ZRP coatings.[1,3,4] The specimens usually exhibit a fast drop in electrochemical potential, and, after a few months, at most, are no longer able to provide sufficient cathodic protection to the steel. Experience with saline solutions, however, may not necessarily be extrapolated to atmospheric exposure; it must be kept in mind that wetness time and degradation of the coating in the atmosphere will probably be much lower than in saline solutions. A longer period of cathodic protection in the atmosphere is thus foreseeable, although there is a lack of quantitative information on this matter. Due to the slower formation of zinc corrosion products, the barrier effect probably develops much more slowly in the atmosphere than in a saline solution. In both cases, to extend the information already obtained in the laboratory with saline solutions to the atmospheric behavior is a suitable goal. Therefore, in the present work an effort has been made to know the effect of atmospheric exposure on the protective mechanisms of ZRP coatings.

In general, the atmospheric exposure tests are a reliable predictor of the paint life in this medium. However, field performance evaluation in itself provides limited information on the individual processes that develop in the ZRP coatings. Electrochemical laboratory tests, on the other hand, can provide insight into the evolution of the protective mechanisms in a rational and quantifiable manner. Unfortunately, experimental conditions in the laboratory may be remote from those that prevail in practice. For these reasons, an approach integrating both types of tests may be advisable. The experiments in this paper are based on a combination of weathering tests and electrochemical measurements in a 3% NaCl solution.

Rest potential and electrochemical impedance measurements on ZRP coatings have been used in previous studies to obtain information on the extent of cathodic protection and the magnitude of the barrier effect developed in them.[1,2] On the one hand, the rest potential ($E_{corr}$) of specimens submerged in a 3% NaCl solution measured against a reference electrode permits monitoring of the possibilities of cathodic

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*Avenida de Gregorio del Amo, 8, 28040 Madrid, Spain.

protection of the base steel. On the other hand, the apparent polarization resistance ($R'_p$) gives information on the evolution of the barrier effect.[5] A definition of $R'_p$ is given in the following paragraphs.

In general, the impedance diagrams for ZRPs (Nyquist complex plane plots) are made up of a high (or intermediate) frequency semicircle and a low frequency arc or tail. At the beginning of its exposure to the corrosive medium, the response of the ZRP coating to the ac electrical signal is mostly due to the corrosion reaction of the zinc particles.[2,5] Assuming that an analogous relation to Stern-Geary equation[6] is applicable, the rate of the metallic attack ($I_{corr}$) will be inversely related to $R'_p$. The increase in diameter of the highest frequency semicircle with time has been interpreted as a result of the partial electrical disconnection of the zinc particles from each other and from the base steel, and from the construction of an insulating barrier of corrosion products around them.[2,7] After a few days of specimen immersion in the saline solution, the semicircle occurring at the higher frequencies tends to depend more and more on the ohmic resistance of the ZRP coating.[2,8] In this phase of the test, the semicircle diameter corresponds to an apparent polarization resistance ($R'_p$), whose value bears no definite relation to $I_{corr}$.

It is to be expected that the evolution of the parameters ($E_{corr}$ and $R'_p$) with immersion time in the 3% NaCl solution will be a function of both the original intrinsic properties of the paint and of the effect caused in the coating by the previous period of atmospheric exposure. Thus, it seems reasonable to use those parameters to assess the protective ability of a ZRP coating at the moment of its immersion in the saline medium, and also to follow the changes that occurred in the ZRP coatings due to the atmospheric exposure.

## EXPERIMENTAL

The zinc-rich paints (compositions shown in *Table* 1) were formulated with two vehicles: ethyl silicate and epoxy-polyamide. They include paints with diverse contents of zinc and conductive extender ($Fe_2P$) particles. It was considered convenient to use them as a basis for the present study because of the information already obtained on the same series of paints.[8,9]

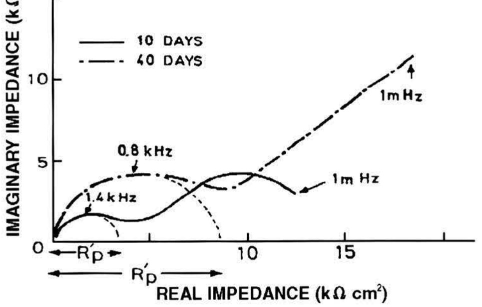

**Figure 1—Effect of the exposure time in a 3% NaCl solution on the Nyquist impedance diagrams for a ZRP coating. The value of $R'_p$ is deduced from extrapolation to the real axis of the semicircle of the high (or intermediate) frequencies**

The paints were air-sprayed on grit-blasted carbon steel specimens. From the series of specimens prepared, those with an average dry film thickness within the range 60 ± 5 μm were selected for the experiments. After a curing time of one week in the laboratory atmosphere, the painted specimens (10 cm × 15 cm) were kept in desiccators until used in the experiments. Electrochemical testing of the unexposed specimens started in about six months. At the same time, sets of specimens were exposed for one and three years outdoors, on a rack at 45° to the horizontal, facing south. Then, they were electrochemically tested. The exposure site was the roof terrace of the CENIM Corrosion Laboratories in Madrid, in an atmosphere classified as urban, with the following characteristics (annual data): RH, 62%; rainy day, 75; wetness time (fraction), 0.26; temperature, 13.4°C; sulphur dioxide, 0.51 $mgSO_2 \bullet dm^{-2} \bullet d^{-1}$.

For the electrochemical tests, methacrylate tubes 5 cm high and 2 cm interior diameter were attached to the painted specimens following an established technique.[1] The interior of the tubes housed the electrodes and the electrolyte constituted by a 3% in-weight solution of NaCl. The reference electrode was a saturated calomel electrode (SCE) and the counterelectrode was a sheet of platinized titanium.

The faradic impedance measurements were obtained at the rest potential using a SOLARTRON 1250 Frequency Response Analyzer coupled to a SOLARTRON 1286 Potentiostat Interface. A 10 mV amplitude sinusoidal signal was applied to the system, with a logarithmic frequency scan of five sweeps per decade, in the range from 55kHz to 1 mHz. The value of $R'_p$ was obtained from the arc drawn by the impedance diagram at the highest frequencies, extrapolating this arc up to its intersection with the real axis and assimilating it to a semicircle with diameter $R'_p$ (*Figure* 1). The potential of the painted steel was measured using the same cell as in the impedance measurements. The specimen was electronically connected via a high impedance voltmeter to the SCE.

Electrochemical measurements for testing the corrosion protective quality of coatings depend greatly upon the homogeneity and integrity of the coating (e.g., presence of pores). For this reason the reproducibility is often poor. Impedance measurements taken from several areas of the same painted surface, or identically prepared coated specimens, usually show significant scatter, especially after lengthy exposure to a saline solution.[10-13] In this work, measurements have been made in duplicate and repeated at time intervals throughout the testing period. As anticipated, an important scattering has been found between the data of the two replicates for the same test periods in the saline solution. Even so, the general trend of the change in the $E_{corr}$ and $R'_p$ curves with time was rather similar between the two replicates. *Figures* 2 and 3 refer to only one of them.

## RESULTS AND DISCUSSION

*Figure* 2 shows the values of the rest potential ($E_{corr}$) of the ZRP coatings in the 3% NaCl solution for unweathered

**Table 1—Characteristics of ZRP Coatings**

| | Pigment, % by weight in dry film | | | |
|---|---|---|---|---|
| **Binder** | **Zinc dust[d,e]** | **$Fe_2P$[f]** | **Paint designation** | **Other** |
| Ethyl silicate | 84 | 0 | 84/0 ethyl silicate | For all variants: |
| Silester ZNS[a], organic silicate | 72 | 12 | 72/12 ethyl silicate | Solid content, 83.5 |
| with a 40% of $SiO_2$ content | 60 | 24 | 60/24 ethyl silicate | PVC, 68.5 |
| Density, 1.058 g/cm$^3$ | | | | |
| Epoxy-polyamide | 91 | 0 | 91/0 epoxy-polyamide | For all variants: |
| Epikote 1001[b] (96.2, % by weight), | 80 | 11 | 80/11 epoxy-polyamide | Solid content, 85.0 |
| bisphenol epichlorohydrin | 70 | 21 | 70/21 epoxy-polyamide | PVC, 70.5 |
| Epoxy equivalent, 450-525 | | | | |
| Molecular weight, 900 | | | | |
| Equivalent weight, 130 | | | | |
| Density, 1.206 g/cm$^3$ | | | | |
| Versamid 115[c] (3.8, % by weight), | | | | |
| high viscosity polyaminoamides | | | | |
| Amide value, 240-260 | | | | |
| Solids content, 100% | | | | |
| Density, 0.98 g/cm$^3$ | | | | |

Suppliers: (a) Monsanto España; (b) Shell; (c) Schering España; (d) for silicate binder, Societé de la Vielle Montagne; (e) for epoxy binder, Refinados Metálicos; (f) Hooker Chemicals and Plastics.

coatings and after one and three years of atmospheric exposure. *Figure* 3 shows the corresponding values of $R'_p$ determined from the impedance diagrams according to the established procedure.

In the introduction, we raised the question of the time during which the ZRP coatings are able to provide cathodic protection during atmospheric exposure. According to the results of this study, all coatings in the atmospheric exposure maintained part of their original ability for cathodic protection even after three years of exposure. This behavior agrees with the results obtained by Simancas[14] with an ethyl silicate zinc-rich primer over a period of eight years in the atmosphere of Madrid, and contrasts with the results in the 3% NaCl solution, in which cathodic protection does not last very often more than 100 days.[1,8,15,16] In this paper, the use of zinc-rich paints with different contents of $Fe_2P$ conductive extender also shows the possibility of replacing 11-12% of the zinc with the same percentages of $Fe_2P$ without excessively impairing the action of the cathodic protection mechanism in atmospheric exposure (*Figure* 2).

A widely applicable empirical criterion for steel cathodic protection is to ensure a minimal negative value of –850 mV versus a copper/copper sulphate electrode (–780 mV/SCE).[17] In the present study, the number of days during which the painted steel exhibited more negative potentials than this value in the 3% NaCl immersion test has been used for quantifying the ability of ZRP coatings to provide cathodic protection. Examination of *Figure* 2 indicates that the ethyl silicate coatings with the highest metallic zinc contents (84/0 and 72/12) show the best cathodic protection capability. After three years of atmospheric exposure, this capability continues to be high for the 84/0 ethyl silicate coating; more negative potentials than –780 mV/SCE are measured even after 60 days of immersion in the saline solution. In the case of the 72/12 ethyl silicate coating, protective potentials are measured all through the 60 days of testing in the unweathered condition or after one year of atmospheric exposure. However, after three years of atmospheric exposure protective potentials do not last longer than 12 days in the immersion test. For one or three years atmosphere-exposed 60/24 ethyl silicate coatings, more negative potentials than –780 mV/SCE are only measured during a few days in the 3% NaCl immersion test.

On the other hand, none of the tested epoxy-polyamide coatings exposed to the atmosphere for one or three years have been able to maintain the protective potential for more than ten days in the immersion test.

In relation to the barrier effect, there was a significant increase in the value of $R'_p$ during atmospheric exposure (*Figure* 3). The evolution of the barrier effect runs in the opposite direction to the ability for providing cathodic protection. The 84/0 ethyl silicate coating shows the greater ability for cathodic protection, as well as the least development of the barrier effect.

The degree of development of the barrier effect should also be quantified in function of atmospheric exposure time. As a rough guide, the degree of development of this effect has been considered to be inversely proportional to the time needed in the saline immersion test to develop a given value of $R'_p$. In the absence of an accepted index, we have arbitrarily taken a value of $R'_p = 6$ kΩcm$^2$. This reference level is 10-20 times higher than the $R'_p$ values found for the unweathered epoxy-polyamide coatings at the start of the saline immersion and 50-100 times higher than for the ethyl silicate coatings. Whereas the 6 kΩcm$^2$ level was surpassed quickly by some of the coatings, with others (mostly with the ethyl silicate coatings) it was scarcely reached, even after 60 days of immersion.

According to the experimental results, the optimum behavior with regard to the barrier effect corresponds to the 70/21 epoxy-polyamide and the 60/24 ethyl silicate coatings after three years of atmospheric exposure. Both reached a $R'_p$

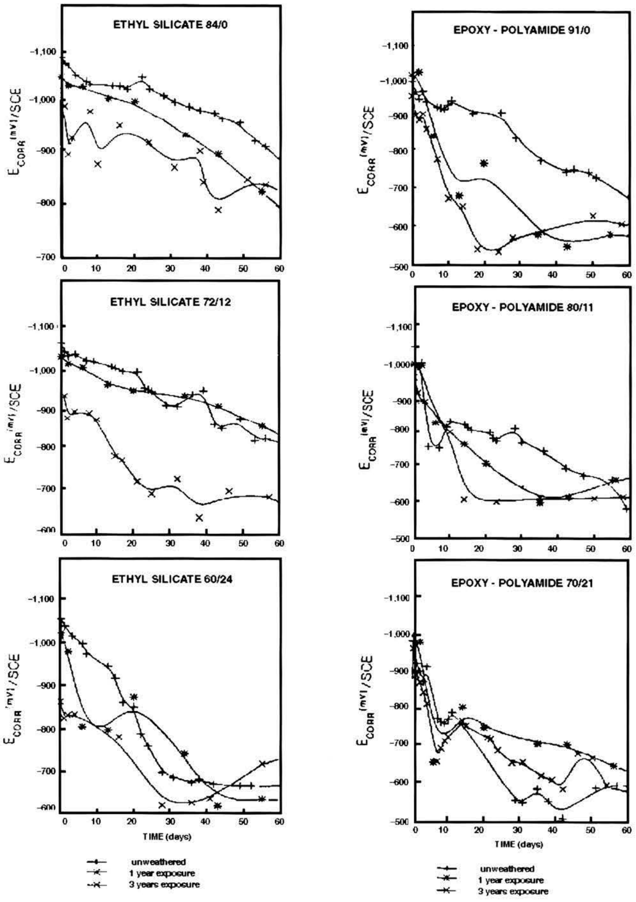

**Figure 2—Variation of rest potential with the immersion time in a 3% NaCl solution. ZRP coatings unweathered and weathered during one and three years in the atmosphere**

of 6 kΩcm$^2$ after approximately one day of immersion in the saline solution. As shown in *Figure* 3, the barrier effect is rather low in the ethyl silicate coating with the highest zinc content (84/0), even after three years of atmospheric exposure, but grows measurably when part of the zinc content is replaced by particles of the $Fe_2P$ extender; especially in the case of the 60/24 composition.

In the epoxy-polyamide coatings, a significant barrier effect is found from almost the beginning of the immersion test. After one and three years of atmospheric exposure, this effect increases significantly with the 91/0 and 80/11 compositions, and quite noticeably in the paint with the highest content in extender particles (70/21 composition).

## COMPARISON OF PERFORMANCE BETWEEN THE TWO BINDERS USED

In previous papers,[1,15] it was found that ethyl silicate ZRP coatings give much longer cathodic protective action under immersion conditions than epoxy-polyamide based ones do. Also, in paints with various zinc contents, the galvanic activity decreases with decreased zinc content. A satisfactory corrosion resistance of systems in which part of the zinc has been replaced by $Fe_2P$ has also been reported.[3,8] With reference to the cathodic protection mechanism, this type of replacement appears to be somewhat more critical in the epoxy-polyamide coatings than in the ethyl silicate ones.[8]

In the present work we had the opportunity to examine the effect of the two binders mentioned on the cathodic action of ZRP coatings exposed to the atmosphere. After one year of atmospheric exposure, the behavior of the ethyl silicate and epoxy-polyamide coatings in the subsequent immersion test showed a certain parallelism with that of the unweathered coatings (*Figure* 2); the longer cathodic protective action generally corresponds to the silicate coatings. After three years of atmospheric exposure, the cathodic protection capability of the 84/0 ethyl silicate coating con-

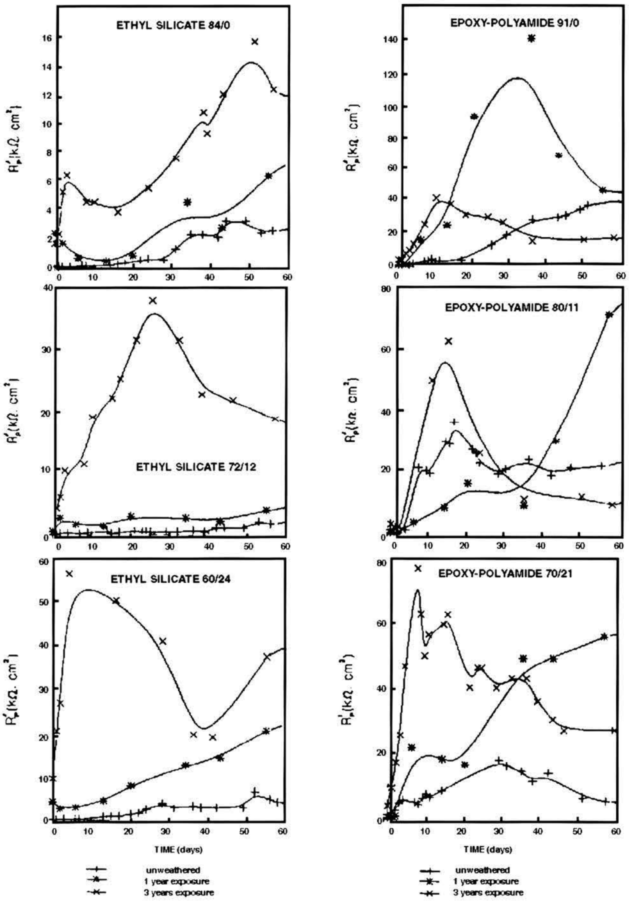

**Figure 3—Variation of $R'_p$ with immersion time in a 3% NaCl solution. ZRP coatings unweathered and weathered during one and three years in the atmosphere**

tinued to be high and much greater than for any of the tested epoxy-polyamide coatings. For the remaining coatings this capability was reduced considerably after three years of atmospheric exposure, and the differences between the coatings of any one of the two binders used are less evident.

It is known that the protection given by ZRPs lasts longer than the initial stage of protective cathodic ability due to the barrier coating properties (final stage of protection), which are promoted by the accumulation of zinc corrosion products within the coating.[2,4,16,18] The barrier effect, expressed by the value of $R'_p$, increased more noticeably for epoxy-based ZRP coatings than the ethyl silicate coatings during immersion in a 3% NaCl solution.[2,8] In the present work, the same general trend was verified after one year of atmospheric exposure. After three years of atmospheric exposure, a rapid and very marked rise of the value of $R'_p$ during the subsequent immersion test should be noted (*Figure* 3). This effect is observed both with the silicate and epoxy type coatings, suggesting an important effect of the atmospheric exposure period on the sealing and blocking of the coating layer by zinc corrosion products.

## SUMMARY

The behavior of zinc-rich paints in saline solutions may not necessarily be applicable to atmospheric exposure. In this paper an attempt has been made to determine the direct influence of a prolonged period of atmospheric exposure on the evolution of the protective characteristics of the zinc-rich paint (ZRP) coatings, in particular, the cathodic protection and the barrier effect mechanisms.

Weathering tests combined with electrochemical tests in a 3% NaCl solution allow quantitative information to be obtained about: (a) the residual ability of the ZRP coating to provide cathodic protection after varying periods of atmospheric exposure; and (b) the degree of development attained by the barrier effect in this type of coating functioning with atmospheric exposure time.

This study has demonstrated that after three years of atmospheric exposure, some of the tested ZRP coatings maintain considerable ability to provide cathodic protection; especially those of the ethyl silicate type. On the other hand, the barrier effect, in general, tends to increase with time during atmospheric exposure; especially with those ZRP coatings of the epoxy-polyamide type.

## ACKNOWLEDGEMENTS

The authors thank Comision Interministerial de Ciencia y Tecnología for financial support, BASF Pinturas y Tintas, S.A. for the manufacturing of ZRPs, and Hooker Chemicals and Plastics Corporation for providing the conductive extender ($Fe_2P$).

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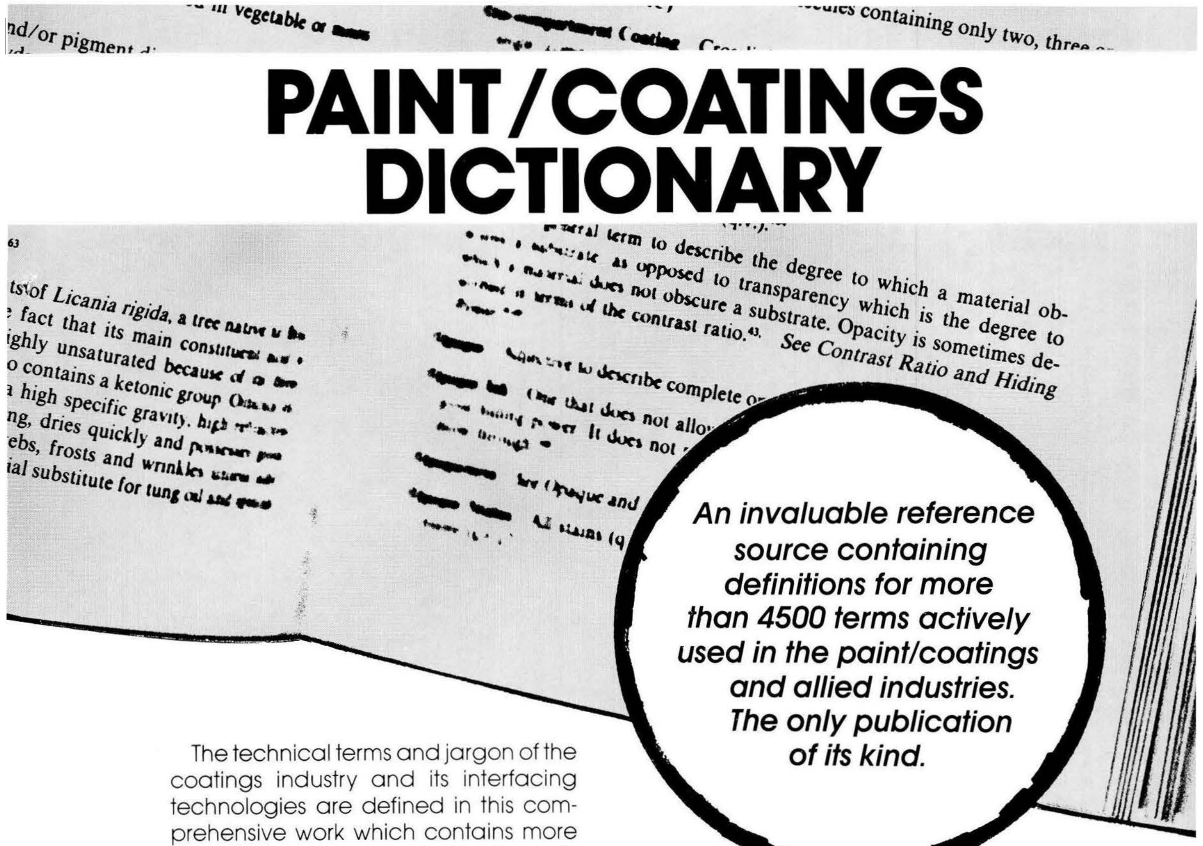

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# June 1993 Subcommittee Reports of ASTM Committee D01

The June meeting of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications was held June 27-30, 1993 at the Franklin Wyndham, Philadelphia, PA. In the three and one-half days preceding the final session and general meeting of Committee D01, 181 members and guests met in about 190 scheduled meetings of D01 and working task groups. The current membership of Committee D01 is 600.

## Highlights

D01.18—A mini-symposium on slip resistance will be held on Tuesday afternoon during the January 1994 meeting.

D01.21—In response to the increasing focus of the U.S. Environmental Protection Agency on the reduction of hazardous air pollutants highlights, D01.21.52, chaired by J. Benja, will focus on developing test methods to measure hazardous air pollutant materials in coatings.

D01.37—The task group, Standard Ink Oil, in cooperation with oil suppliers, has arranged for a supply of a reference oil, Standard Magiesol 47, for use in their test methods. It can be obtained by calling 1-800-Magie 47. With the continued evolution of aqueous and high-solids flexo and gravure inks, D01.37 is seeking new members so that the subcommittee can effectively pursue the development of liquid ink vehicle test methods.

D01.51—A planning session to discuss a technique for measuring uncured powder film thickness is being planned for the January 1994 meeting. Interested people are invited to attend.

D01.53—An accelerated weathering testing program is being planned. Currently, 20 sets of panels, comprising all of the common coating chemistries, have been received.

D01.61—The subcommittee will propose an expansion of their scope to include components of paint applicators in addition to paint applicators.

## Awards

The following awards were presented during the D01 meeting.

Award of Merit—Joseph M. Behrle

D01 Honorary Members—Mary McKnight and James Berry

Henry A. Gardner Award—Paul R. Guevin, Jr.

Task Group Chairman Award—Warren Vanderslice

Certificates of Appreciation—Fred Marschall, Maynard Euverard, Richard Osterman, and Gerard Litvan

## Memorials

John C. Weaver spoke in remembrance of Walter A. Gloger, and Paul R. Guevin, Jr. spoke in remembrance of Maynard Euverard. T. Sliva announced that the Paul N. Gardner Co. had presented a monetary contribution to D01 for an award in honor of Maynard Euverard. The award will be known as the Gardco Award and it will be presented yearly to a member of D01 who has made an outstanding contribution to test methodology.

## Future Meetings

January 23-26, 1994—Crown Sterling, Fort Lauderdale, FL.
June 26-29, 1994—Raleigh, NC.
January 22-25, 1995—Hyatt Regency, San Antonio, TX.
June 25-28, 1995—Atlanta, GA.

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## DIVISION 1 ADMINISTRATION

### SUBCOMMITTEE D01.08 ENVIRONMENTAL CONCERNS

### J.J. Brezinski, Chairman

Included in the topics discussed at the meeting were: the status of negotiated rulemaking relating to Volume Organic Compound (VOC) control in architectural/industrial maintenance coatings and related rulemaking discussions for the control of VOC emissions from aerospace and shipbuilding/repair coatings; test-related actions and the need for new test methods at EPA; and the continuing misdirected focus in state legislatures on previously applied lead-containing paint as the major contributor to lead in household dust and soil.

*VOC Control: Rulemaking Status*—Time limits have been set for the reg-neg meetings relating to efforts to achieve a consensus on a rule to control VOC from architectural coatings and VOC and hazardous air pollutant (HAP) emissions from wood furniture coatings. The control of both VOC and HAPS is also being addressed in discussions pertaining to aerospace and shipbuilding/repair coating operations. The growing emphasis at EPA on the limit of the emission of HAPS has highlighted a need for new test methods to define the amounts of HAPS, and specifically of designated solvents, in paints and coatings. The agency suggested that ASTM D01 consider developing suitable test procedures.

*VOC-Related Standards*—The EPA also urgently needs a new consensus standard for volume-nonvolatile content of paints, a requisite parameter for calculating VOC content based on the volume of applied paint solids. High priority effort is urged for the study of the use of the helium pycnometer to define the density of coating solids.

*Lead Related Testing*—Test procedures capable of identifying the source of lead pollutants in household dust and soil are still needed to help redirect the continuing focus in state legislatures that still consider lead-containing paint as either the sole or major contributor.

## SUBCOMMITTEE D01.15 LECTURES AND SYMPOSIUM

### T. Sliva, Chairman

*D01.15—Lectures and Symposium*—T. Sliva, Chairman, reported that a mini-symposium on slip resistance will be held at the January meeting. The speakers will include Paul Guevin, Jr. and William English. Topics to be discussed will include both instrumentation and test methodology related to slip resistance. T. Sliva will serve as the moderator.

The chairman reported that questionnaires had been submitted to all subcommittee chairpersons to solicit information and ideas on future topics to be presented. Currently, speakers are available for symposiums on "Volatiles Released During Application of Organic Coatings" and "Application Tools."

# DIVISION 2 RESEARCH

## SUBCOMMITTEE D01.22 HEALTH AND SAFETY

### J.J. Brezinski, Chairman

*D01.22.01—Flammability and Combustibility*—R. Osterman, Chairman, reported that the limited number of available Setaflash open-cup testers has slowed the progress of the round-robin study of the suitability of D 4206, "Sustained Burning of Liquid Mixtures," as an alternate for ISO 9038. The latter is being considered for adoption as an international standard to characterize liquid materials that do not support combustion, though they yield a finite flash point. ERDCO Engineering will loan an open-cup tester for the study. Additional round-robin participants are needed.

*D01.22.02—Chromates, Health and Safety Standard*—J.J. Brezinski, Chairman, recommended several revisions to E 848, "Practice for Safety and Health Requirements Relating to Occupational Exposure to Water-Insoluble Chromates." The changes, to be circulated in a D01.22 ballot, include the addition to the Introduction Section of a statement on the need for compliance with all applicable governmental regulations.

*Administrative Change*—J. Benga is the new Chairman of D01.22.

## SUBCOMMITTEE D01.21 ANALYSIS OF WHOLE PAINTS AND PAINT MATERIALS

### K.H. Fujimoto, Chairman

*D01.21.10—Lead Paint Policies*—J.C. Weaver, Chairman, reviewed the actions of several groups. D01.57 is revising its Standard Test Method for Bioavailability of Metals (including lead in art materials) based on negative votes and comments received in a recent D01 ballot. This method is adapted from a European analytical practice on partial solubility in simulated stomach acids. E06.23 on Abatement of Lead in Buildings and Structures is holding quarterly meetings. Of the 13 task groups, there are special needs for participation by paint industry specialists in: E06.23.10, XRF Measurement of Leaded Paint in Buildings; E06.23.11, Performance Criteria for Portable XRF Instruments for Paint Measurements; E06.23.16, Laboratory Test Methods; and E06.23.30, Encapsulants. The University of Rochester, NY was awarded a contract to study the relationship between blood lead levels in children and lead dust by the National Center for Lead-Safe Housing of Columbia, MD. Speciation of the lead compounds to identify the paint versus gasoline sources of lead should be a critically important part of this research. The final report of the EPA's three-city (Boston, Baltimore, and Cincinnati) study of lead in soil versus lead in blood is scheduled for release in the summer of 1993.

The ASTM 1993 Boulder, Colorado Conference on Lead in Paint, Soil and Dust, on July 25-29, is jointly sponsored by D01, D18 on Soil and Rock, D22 on Atmospheres, and E06 on Structures. Among 35 papers to be given, J. Weaver is presenting "Control of Lead Hazards Through Source Identification: The Case for Lead in Gasoline as The Principal Source."

*D01.21.13—Coordination of VOC Standards & Information*—J.J. Brezinski, Chairman, presented an update on the status of the reg-neg meetings which relate to the development of national regulations affecting the VOC content of architectural/industrial maintenance coatings (AIM) and wood furniture coatings, as well as CTG's for aerospace and shipbuilding. The cutoff deadlines for the reg-neg meetings for AIM and wood furniture are the end of September and October, respectively.

Recent developments pertaining to the state operating-permit program, VOC related regulations, and emission trading programs at the state level were reviewed. The California South Coast Air Quality Management District will decide early in the fall whether to initiate an emission trading program which would include reactive organic compounds. If enacted, it will be the first regional air pollution trading program in the United States.

Amendments in the San Francisco Bay Area to limit the VOC emissions from metal, aerospace, and plastic-parts coatings and aerospace stripping operations will require the use of high transfer efficiency spray operations to be in place by July 1, 1994.

*D01.21.14—VOC New Publication & Workshop*—K.H. Fujimoto, Chairman. J. Brezinski, editor of "ASTM MNL 4, Manual on Determination of Volatile Organic Compounds in Paints, Inks, and Related Coating Products," reported that the new revised edition is complete and should be published in the latter part of July.

K.H. Fujimoto announced that the next ASTM Paint VOC Measurement Workshop will be held November 3-4, 1993, at the Sheraton Industry Hills Resort, City of Industry, CA. The host laboratory is the South Coast Air Quality Management District under the guidance of Cori Choa. On May 5-6, 1994, a workshop will be held in Chicago near the O'Hare Airport. The host laboratory will be the Rust-Oleum Corporation with host Richard Osterman.

*D01.21.24—Revision of D 2369, Volatile Content of Coatings*—M.E. Sites, Chairwoman, reported that D 2369 was reballoted as a result of a negative received from K.H. Fujimoto concerning the wording of Note 10, Section 7.3, which referenced the allowance by U.S. EPA of an induction period of up to 24 hours on multicomponent paints. The negative was found to be persuasive since induction periods longer than one hour had not been included in the round-robin studies used to generate the precision statements. The task group agreed to reword Note 10 to read "Other induction periods are used. See U.S. EPA Reference Method 24."

The Precision and Bias Section was also revised to include multicomponent paint systems. The precision obtained in the round-robin was as good as those obtained for solvent and water-reducible coatings reported in the original D 2369 round-robin studies. Other considerations discussed included the lack of a bias statement, the terminology of solvent-based and water-reducible coatings and wording in Note 3. All negatives concerning these items were withdrawn or found not persuasive.

*D01.21.24A—Ion Chromatography, Electrocoat Bath Sample*—J. Furar, Chairman. With the resignation of M. Mahon, J.

Furar volunteered to chair this task group. A revised guide, "Determination of Anions in Cathodic Electrocoat Permeates by Ion Chromatography," was distributed to the group for review. Four editorial changes were made at which point the task group agreed to submit the document to D01.21 ballot.

For future work, the task group decided to concentrate on the determination of phosphate. In the past round-robin work, the determination of this elusive anion has been disappointing. A library search will be conducted as a starting point for this new endeavor. K.H. Fujimoto requested that the chairman determine from M. Mahon the status of work on the practice for the analysis of electrocoat bath samples and recommended that the "ion method" be incorporated.

*D01.21.26—Review of D 2697, Volume Nonvolatile Matter in Clear Or Pigmented Coatings*—B. Neff, Chairman, lead discussions on the scope of the group's new endeavor to replace the disk method in D 2697 for the determination of the density of the baked paint films with a helium gas pycnometer method. The pycnometer takes less time and is more accurate. The biggest problem has been preparing the dried or baked films. The group decided to submit the draft method to D01.21 ballot before another round-robin was completed. Incomplete results of the current round-robin were discussed. No trends could be identified. In this round-robin, three samples were sent to five collaborators: (1) a latex house paint, (2) a high PVC latex, and (3) an alkyd. Although Tedlar™ is the recommended release substrate upon which to cast wet films in the proposed method, two other release materials will be included in the next round-robin, Lenata Corporation release paper and glass slides.

The task group extends its appreciation to H. Stanley, of Micromeritics, who has volunteered to donate loaner instruments to those laboratories without a pycnometer. This generous response is appreciated since it makes it easier for the collaborators to generate data for the round-robin.

In the following discussion, K.H. Fujimoto reminded the task group to control the thickness of the dried or baked film close to one mil. Otherwise, solvents will be trapped in the film which gives fleeting and erroneous readings on the gas pycnometer. W. Zimmt suggested that thin uniform wet films could be made with a spin coating apparatus. D. McCunn recommended casting the films on a metal counter top to reduce the generation of static electricity.

*D01.21.27B—New Approach to VOC*—R. Jayanti, Chairman. In the absence of the chairman, K.H. Fujimoto chaired the meeting. He reviewed the activities of the task group to date and the proposed method to measure the VOC directly. In the proposed method, a sealed glass apparatus, in which the paint specimen has been placed and through which dry nitrogen is passed, is placed in an oven and heated. The effluents are collected in two charcoal filled tubes, in tandem. A third tube containing indicating drierite is used to collect the moisture/water. By directly weighing the gain in weight of the tubes, the amount of VOC and water present in the paint can be determined. It was hoped this direct method would eliminate or minimize the inherent calculation errors present in reporting VOC as specified in U.S. EPA Reference Method 24.

Based on preliminary round-robin results, R. Jayanti transposed the method into ASTM format and submitted it to D01.21 ballot in 1992. Nine negatives and three comments were received. However, no additional work has been done on the balloted draft. The task group agreed that a direct method for determining the VOC in paints is needed to circumvent the errors intrinsic in the calculations. While the present method under study for the direct determination VOC is undergoing revision, two additional methods were suggested for investigation; direct injection (gas chromatograph (GC)/mass detector) and trap/collect and identify by GC. The task group requested that other ideas for a direct method to determine VOC in paints be sent to K.H. Fujimoto, 5171 Rock Run, West Bloomfield, MI 48322.

*D01.21.49—Sample Preparation for Trace Metal Analysis*—B. Neff, Chairman, reviewed the proposed sample preparation method, an enclosed wet digestion scheme at atmospheric pressure, which uses commercially available equipment. However, this method cannot be used to prepare samples for lead analysis due to the formation of a lead sulfate precipitation. The chairman agreed to revise the method and sent it to the task group for comments. Once a method is agreed to, another round-robin will be initiated. Alternative digestion methods were also discussed, such as the microwave techniques and wet and dry oxidation.

*D01.21.51—Determination of Formaldehyde in Paints*—D. McCunn, Chairman, reviewed past discussions that have narrowed the search for a method for measuring evolved free formaldehyde by GC using OSHA approved Method #52 or possibly by liquid chromatography. D. McCunn reported on the results obtained when he used Method #52 and industrial hygiene sampling tubes supplied by S.A. Kail. Although he obtained a nonlinear calibration curve, this appears to be less of a limitation that the low recovery of formaldehyde in the sample. Further, paint samples spiked with formaldehyde may not be stable or may not be a valid standard material due to reaction with the water in the sample. A suggestion that future experiments include a latex sample instead of a completed paint were discussed. Also, there was a discussion about current methods which are used to determine "free" formaldehyde in latex samples. They involve direct measurement by GC, liquid chromatography (LC), or colorimetric analysis after centrifugation or extraction. However, these techniques have been tried in the past without success.

The remaining discussion centered around future work to be done and the best approach for preparing samples for round-robin evaluation and method validation. The chairman agreed to finish the current experiments and then prepare a method using either the GC or LC methodologies and distribute it to the task group for comments. An initial benchmark round-robin is to be conducted with a latex and a paint sample to be spiked at the participating laboratories. The task group agreed that although it would be desirable to have a method with very low bias and good precision, a method having poorer precision may be acceptable as a starting point.

*D01.21.52—Paint Solvent Analysis by Gas Chromatography*—J. Benga, Chairman. K.H. Fujimoto announced that J. Benga had volunteered to be the new chairman for this task group. The primary focus of this task group is to develop a standard for determining volatile organics in liquid paint and coatings, with emphasis on analysis for those substances on the EPA's Hazardous Air Pollutants (HAP) list. As a follow-up, the task group will also investigate the need for determining HAPs that may be generated as the cure products of coatings.

Methods to be investigated include GC with conventional detectors such as flame ionization, GC/mass spectrometry (MS) such as EPA 8170A and any other appropriate procedures. For HAPs generated as cure products, a thermal extraction combined with GC/MS will be investigated. Methods for the determination of solvent content of paints and coatings by GC are being solicited. Candidate methods and ideas should be sent to J. Benga, PPG Industries, Rosanna Drive, Allison Park, PA 15101. Once a representative set of procedures is available, a draft method will be circulated to the task group members for comments. Following the draft review and appropriate revisions, a mini round-robin will be conducted with the results available for discussion at the January 1994 meeting.

*D01.21.54—Revision of D 4017 Standard Test Method for Water in Paints and Paint Materials by Karl Fischer Titration*—R. Osterman, Chairman, distributed a revision of D 4017 which included a modified methanol extraction procedure for paint systems exhibiting variable results. The variations in results may be due to the inability of the pyridine reagent, which is used to extract the water from the paint, to remove the water tightly held in hydrophilic latex emulsions. But after a lengthy discussion, the task group members decided to modify this revision and eliminate a separate procedure for the methanal extraction, but to recommend its use if variable results were obtained using the direct titration method. W. Golton agreed to help rewrite the scope, and he offered to review the final draft of the revised method. A round-robin will be conducted before the next meeting to evaluate the proposed method. It was suggested that both techniques be used on the test samples so that a comparison of results could be made.

*D01.21.56—Revision of D 3960 Practice for Determining Volatile Organic Compounds (VOC) of Paint and Related Coatings*—M.E. Sites, Chairwoman, distributed a copy of the latest revision of D 3960-93 which contains a reference of D 5095-90 for use in the determination of VOC content of silane and siloxane coatings. D 5095 has been approved by the U.S. EPA. Another change is the replacement of Section X3 (regulatory definition of VOC) with Appendix X3, the revised definition of VOC published by the EPA under 40 CFR, Part 51, *Federal Register*, Volume 57, No. 22, February 22, 1992, pp 3941-3946. This latest revision of D 3960-93 will be available as a "separate" shortly and it will appear in the *1994 ASTM Book of Standards*, Volume 6.01.

*D01.21.80—Exploratory Analytical Chemistry*—K.H. Fujimoto, Chairman, reviewed the purpose of this task group which is to: (1) review new or needed test methods; (2) recommend changes and/or revisions to existing D01.21 test methods; (3) ascertain whether proposals made to D01.21 have merit for consideration; and (4) recommend the formation of new task groups.

The Plassma flier submitted by J.C. Weaver at the last meeting was discussed. This is a portable laser spark surface mass analyzer, which is used to obtain rapid measurements of surface contaminants such as lead at the 0.06% concentration level. Other hazardous heavy metals can also be determined. A laser is used to form a microplasma or spark to atomize and electrically excite the elements. B. Neff agreed to investigate the instrument to see if it can be used in analyzing trace heavy metals.

ASTM D 2369 for determining percent volatile in paints, which was developed by D01.21 and which is listed in EPA's Reference Method 24 for the determination of VOC in paints, has been criticized as inadequate and subject to erroneous results. However, no data has ever been submitted to substantiate the criticism. After much discussion, the task group decided that it has been at least six years since D 2369 has been evaluated to determine the percent volatiles in the new modern paint technologies, and it was time to initiate a round-robin.

The chairman will request that individuals who are having difficulty in running D 2369 on their paint or coating products send their data and descriptions of their problems to him. After investigating the complaints and comments received, the task group will decide whether to reactivate the D 2369 task group. (If you have comments please send them to K.H. Fujimoto—see D01.21.27B for address.)

J. Berry, U.S. EPA, has asked D01 to address the problem of analyzing wet paint and the baking volatiles for the presence of any of the compounds listed in EPA's list of 189 HAPs. These compounds include xylene, toluene, styrene, 2-nitropropane, methyl isobutyl ketone, methyl ethyl ketone, methanol, formaldehyde, ethylene glycol, and diethanoiamine.

D01.21.52 is developing a method for the analysis of solvents in wet paint by use of direct injection GC or GC/MS or both. This activity can be tied into the analysis of some of the listed HAP solvents. However, unless the experimental parameters under which baking volatiles are to be measured are known, the task group cannot address the problem. K.H. Fujimoto will contact J. Berry for this information.

## SUBCOMMITTEE D01.23 PHYSICAL PROPERTIES OF APPLIED PAINT FILMS

### P.R. Guevin, Jr., Chairman

*D01.23.10—Adhesion*—G.E. Nelson, Chairman, reported the proposed Appendix to D 3359-90, "Test Methods for Measuring Adhesion by Tape Test," citing various issues relating to the tapes currently specified in the method, had completed D01 ballot. One negative was received from W. Ketola, but the voter has agreed to withdraw the negative. The revision will proceed to society ballot. Comments from W. Ketola and other voters will form the basis for a new ballot for revision. The chairman read a letter from the Swedish Institute of Wood Technology Research inquiring about the use of D 3359 for coatings on wood. The task group has agreed to solicit suggestions for developing a tape test for both wood and plastic substrates and the findings will be presented at the January meeting.

*D01.23.12—Dry Film Thickness*—S.K. Boocock, Chairman, reported the revisions to D 1186-87, "Test Methods for Non-Destructive Measurement of Dry-Film Thickness of Non-Magnetic Coatings Applied to Ferrous Base," and D 1400-87, "Test Methods for Non-Destructive Measurement of Dry Film Thickness of Non-Conductive Coatings Applied to a Non-Ferrous Base," had successfully completed D01 ballot. Editorial comments received were reviewed and will be incorporated into the revision before it is submitted to society ballot.

It was the decision of the task group to set up a round-robin to update the precision and bias statements of both methods.

*D01.23.14—Hardness, Abrasion and Mar Resistance*—P.R. Guevin, Jr., Chairman, reported that the revisions to D 968, "Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive," and D 2134, "Test Method for Determining the Hardness of Organic Coatings with a Sward-Type Rocker," had successfully completed D01 ballot. B. Gregorovich reported on the results of the second round-robin evaluating the proposed "Test Method for Mar Resistance of High Gloss Coatings." The method uses a Taber Abraser and measures gloss retention after abrasion. The data from four of the five collaborators showed good reproducibility. The data will be subjected to statistical analysis before the next meeting. The proposed test method was submitted to D01.23/D01 ballot and received three negative votes. The negative from P. Guevin was found persuasive and the method was withdrawn from ballot.

N. Bednar reported on his evaluation of Sward Hardness on three representative organic coatings using four different instruments with differing weights and dimensions. The chairman distributed data plotting the hardness of the same coatings using a dynamic hardness tester, a modified Pfund indentor, an electronic Sward instrument and pencils.

*D01.23.15—Slip Resistance*—P.R. Guevin, Jr., Chairman, read a report from M. Morse on round-robin activity to develop a precision statement for D 4518-91, "Test Methods for Measuring Static Friction of Coating Surfaces," using a "drop sled" inclined plane test procedure and calibrated spring horizontal pull test method under wet conditions. The method will be revised to incorporate these findings.

The chairman discussed the Ergodyne Slip-Resistance Tester and reported that he will determine slip resistance under wet and dry conditions on various porch and deck enamels using this instrument. The results will be reported at the mini-symposium to be held in conjunction with the January meeting. The chairman also reported on activities related to slip resistance within other ASTM Committees.

*D01.23.16—Water Vapor Transmission*—T.J. Sliva, Chairman, reported that the proposed revision to D 1653-85, "Test Method for Water Vapor Transmission of Organic Coating Films," to include a statement in the Appendix outlining differences expected between methods and the applicability and limitations of both methods under typical use, had completed concurrent D01.23/D01 balloting with one negative. The negative from W. Elfring, who objected to a guidance statement, was found not persuasive by the task group. Editorial comments received during balloting will be incorporated into the method before it proceeds to society ballot.

*D01.23.19—Drying Time*—T.J. Sliva, Chairman, reviewed the scope of the task group. Subcommittee D01.23 has assumed stewardship and responsibility for D 1640, "Standard Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature." The method will be reviewed for editorial and technical revisions and submitted to D01.23/D01 ballot before the next meeting. The chairman distributed Draft No. 1 of the proposed "Test Method for Determination of Drying Characteristics of Organic Coatings using Mechanical Drying Time Recorders" for review. The task group will look to incorporate both straight line and circular drying time recorders into the method. A round-robin was initiated at this time using a straight line drying time recorder. Four cooperators have agreed to evaluate four coatings. In addition, cooperators will determine the drying time characteristics of the four coatings following D 1640. The results of the round-robin will be reported at the next meeting.

*D01.23.20—Exploratory Research*—P.R. Guevin, Jr., Chairman, introduced D. Klepac, of Fischer Technology, who demonstrated the uses of their dynamic indentor hardness instrument. Data was obtained on three powder coatings applied to steel panels. R. Bearhalter, of New Age Industries, distributed data generated using a similar instrument with the same panels. While the results were not similar as different indentors were used, the rank order of results was similar.

The chairman reported that data measuring the film thickness of organic coatings applied to cement substrates nondestructively using ultrasonics were not available, but will be ready for the January meeting. The chairman reviewed documents submitted by Dr. Mann regarding the "Constrained Blister Test" to measure adhesion.

## SUBCOMMITTEE D01.24 PHYSICAL PROPERTIES OF LIQUID PAINTS

### C.K. Schoff, Chairman

*D01.24.18—Dispersion Phenomena*—R.K. Morrison, Chairman, discussed the revision of D 1210, "Fineness of Dispersion of Pigment-Vehicle Systems" and the inclusion of a new step gauge for cleanliness and provision for a lighting box to better standardize all the tests in the method. The revision has been prepared, and R.K. Morrison is waiting for clearance from his legal department to publish the drawing of the lighting box in the method.

*D01.24.19—Viscosity by Efflux Cups*—C.K. Schoff, Chairman, discussed D 4212, "Viscosity of Dip-Type Viscosity Cups," and D 1200, "Viscosity by Ford Cup." In the most recent D01 ballot, D 4212 had one negative, but this has been withdrawn. There were a few comments which are being incorporated. The method needs bias statements (one each for the Zahn and Shell cups), but these will be added in the next revision. The method will go for society ballot. Regarding D 1200, there was a suggestion that the Fisher cup also be included in the method. This fits with a decision at a previous meeting that D 1200 should be made into a general non-dip efflux cup method.

*D01.24.21—Stormer Viscometer*—M. Morse, Chairman, discussed D 562, "Viscosity by Stormer Viscometer," and the changes needed because of the introduction of two new digital "Stormers," the Brookfield Engineering KU-1 and the Cannon Instruments/Thomas Scientific Electronic Stormer™. The task group will add a Method B to take care of the digital instruments. A draft of the additional instruments. A draft of the additional section was presented and discussed. The revised method will be balloted in D01.24, preferably after first doing interlaboratory testing for precision with the two new instruments (using results from the older model for comparison). The revisions will include the correction of Thomas Scientific's name and address and the noting that Stormer™ is a registered trademark of Thomas Scientific.

*D01.24.26—Electrical Properties of Liquid Paints*—This task group met informally to discuss the revised version of the proposed method for electrical resistivity of liquid paint and related materials. R. Diem, of DuPont, will review the method and see if it needs any more work. We hope to submit the method for concurrent ballot by the end of the year.

*D01.24.33—Odor Evaluation*—D.E. Darr, Chairman, discussed the proposed "Guide to the Detection, Identification, and Characterization of the Odors of Paints, Inks, and Related Materials." A. Rutkiewic had reviewed the ASTM literature for applicable methods and found more than 20 . He then examined the methods to determine which ones were the most appropriate. He found one dominant method, E 544, "Practice for Referencing Suprathreshold Odor Intensity," and five secondary methods. E 544 deals with how strong the smell is (the suprathreshold is where people can just detect something). The methods and practices that have been identified can handle whether the material has a detectable odor or not. The quality of the odor is another story. The discussion led to a suggestion that the proposed guide to broken down into several sections:

(1) Threshold—can you smell something?

(2) Intensity—how strong is the smell?

(3) Characteristics—what does it smell like?

(4) Other—could include means for identifying odor source.

Work on the guide will continue, and it is hoped that outlines or very rough drafts of one or more sections can be ready for the next D01 meeting.

*D01.24.34—Viscosity by Falling Needle Viscometer*—J. Hartnett, Chairman, discussed the proposed method, "Viscosity by Falling Needle Viscometer." The two negatives from the most recent ballot of this method had been withdrawn based on the understanding that proper precision and bias statements (there are four of them) will be balloted and then added to the method. The method will go to society ballot without the statements, but the latter will be balloted at D01.24 and D01 levels this year, then incorporated into the method.

There was no meeting of D01.24.20, Rotational Viscometers, But D 4287, "High-Shear Viscosity Using the ICI Cone/Plate Viscometers," was discussed during the meeting of D01.24. The method had been balloted for reapproval, but had received several negatives. In addition, a new viscometer similar to the ICI was being introduced by Brookfield. D01.24 recommended that D 4287 be made more generic, but that major changes be left for the next revision as the method already is five years old and action is needed in 1993.

## SUBCOMMITTEE D01.28 BIODETERIORATION

### M.C. McLaurin, Chairman

*D01.28.01—Package Stability*—M.C. McLaurin, Chairman. The proposed method revision was balloted and received only one negative vote from the D01 ballot, along with some affirmatives with comments. Although the negative was withdrawn, several of the affirmative voters' comments were indicated by S. Orthey to be substantive, and would require a further ballot. It was decided that the balloted document, with editorial changes to address the negative vote, should be allowed to proceed to society ballot since it is greatly improved over the old method. Concurrently, the standard would be revised and submitted to D01.28 ballot. We would like to thank all those who contributed to the success of this task group's efforts!

*D01.28.02—Rapid Determination of Enzymes*—J. Hinkle, acting Chairman for C.W. Vanderslice. A conflict in schedule prevented C.W. Vanderslice from attending, but he forwarded an assurance that he would draft the method into ASTM format and pursue the round-robin. L. Sadasivan, of Hüls America, briefly discussed an agar plate, colorimetric spot test method for detection of cellulosic enzymes, and some of the work that she has done with this method. Interest was expressed for her to draft this procedure for circulation to the D01.28 for consideration.

*D01.28.03—Microbial Quality of Raw Materials*—J. Hinkle, Chairman. The proposed new method received only one negative vote from a D01 ballot, along with some affirmatives that included editorial suggestions. Part of the negative was editorial and changes were made. The other part referred to requesting a change of 5.5 from:

> "To sample unopened, dry raw materials in bags, wipe a large area of the outside of the bag clean with a clean rag or paper towel. Using a clean knife, cut open the bag within the cleaned area."
>
> to:
>
> ". . . or paper towel and isopropyl alcohol. Using a knife which has been sterilized with isopropyl alcohol, cut . . ."

This objection was made in a 1992 ballot, was discussed, and the proposed change was rejected at our June 1992 meeting. Unfortunately, the voter was not willing to compromise on the issue, so the negative was found not persuasive by unanimous 6-0-0 vote at D01.28.03 (and again 7-0-0 at the D01.28 meeting). The reasons were that: isopropanol will not completely sterilize the area or knife (although it may help sanitize it); inclusion of isopropanol in the method adds handling of a hazardous material where it is not required; residual isopropanol could affect the accuracy of findings for sterility by killing viable contaminating cells; and complications from the solvency of the isopropanol through the bag could contaminate the raw material, or carry chemicals into the users' skin. In addition, numerous members have sampled dry powders without using isopropanol with no negative effects. (However, the negative vote was upheld at the D01 meeting.) Since many requests for this method have been made, we will work with ASTM and the negative voter to resolve this issue.

*D01.28.04—Resistance of Paint Films to Algae Attack*—J. Hinkle, Chairman. The proposed new method was balloted and received only one negative vote from a D01 ballot, along with some affirmatives that included editorial suggestions. Although the negative voter agreed to withdraw with the editorial changes made, we were informed by S. Orthey that the changes were substantive and would require reballoting. As a result, all changes suggested will be incorporated into the method prior to reballoting.

*D01.28.05—Revision of ASTM D 3273 (Mildew Chamber)*—J. Hinkle, Chairman, apologized that with the ballot actions of the other task groups, he had neglected to get this item completed. A draft of the method as previously agreed will be prepared and balloted. Full specifications for the updated component, and/or details on how to obtain chambers meeting the new specifications from one source, are now available upon request from the chairman.

*D01.28.06—Resistance of Paint Films to Fungal Attack*—J. Hinkle, Chairman. The same situation as reported for D01.28.04 occurred for this method with one of the two negative votes. The other, submitted by L. Wienert, provided some excellent suggestions which will be incorporated to improve the method and clarify some potentially misleading points. We appreciate his input and will request that he review a draft before reballoting.

*D01.28.07—Revision of D 3274 (Defacement Ratings)*—B. Matta, Chairman. The task group commenced with a discussion as to the necessity of better textual explanation of whether fungal morphology, frequency of defacement, or color depth of organism constitutes the severity of ratings. Cases exist whereby total, square defacement coverage is considered more aesthetically acceptable than isolated bands of fungus containing deep-colored fruiting bodies. The following textual revisions were agreed upon: update fungal genus name; expand mycology definitions; and clarify seemingly contradictory statements in 5.1, 6.1, and 7.1.

The task group agreed that current photographic plates contained within D 3274 do not accurately represent the type of mildew defacement typically observed on exterior test panels. Consequently, revision of photographic plates will be attempted.

B. Matta presented a 1962 FSPT standards Manual depicting mildew defacement photographs which realistically represent degrees of failure observed in field exposures. Unfortunately, negatives of those photos are no longer available according to B. Hopkins.

M. Crewdson presented data indicating the current rating scale, based on total coverage, to be geometric as opposed to linear. He also provided photographs of exposure panels displaying both spore and hyphae defacement. R. Walter suggested use of computer imaging thereby digitizing degrees of failure. L. Gutman, of ASTM, will be contacted as to feasibility of this approach.

*D01.28—Biodeterioration*—M.C. McLaurin, Chairman. It was noted that we have made considerable progress on many of our methods. The negative vote from D01.28.03 was discussed and found not persuasive by unanimous vote 7-0-0.

The new ASTM rules forced D 4610 to be balloted for withdrawal. The negatives for withdrawal were found persuasive. The previously proposed revision, which was not balloted before in D01, will be balloted.

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# DIVISION 30
# PAINT MATERIALS

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## SUBCOMMITTEE D01.33
## POLYMERS AND RESINS

### J.G. Lamberton, Chairman

*D01.33*—J.G. Lamberton, Chairman, reported results of the D01 ballot. The majority of the items submitted for D01 ballot were approved and will be sent to society ballot. Negatives concerning D 0563, "Standard Test Method for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions," were found persuasive. A round-robin to establish precision for the method is planned before the January 1994 meeting. Discussion concerning the use of toluene as a replacement for benzene in several methods was indicative of the concern for the use of benzene in the methods. Volunteers for round-robins using toluene in these methods are to be sought at the next meeting. Due to the absence of task group chairmen, D01.33.12 and D01.33.14 met jointly with D01.33.

## SUBCOMMITTEE D01.34
## NAVAL STORES

### J. Russell, Chairman

(Minutes of May 14, 1993 Meeting)

*D01.34.01—Capillary Gas Chromatography of Rosin and Fatty Acids*—W. Trainor, Chairman, had distributed a revised draft of the method after incorporating suggestions made at the January meeting. To test the method, it was agreed that all interested parties would analyze a sample of distilled tall oil using one or more of the three methylation procedures described in the method and their own preferred GC operating conditions. The gas chromatographic traces will be discussed at the next task group meeting in order to establish the most generally satisfactory chromatographic conditions.

*D01.34.02—Softening Point of Rosin and Rosin Derivatives by the Automatic Mettler Apparatus*—P. Zawislak, Chairman, explained that the aim of this method was to identify the operating conditions of the Mettler method that would give essentially the same result as the current manual Ring and Ball method, E 28. W. Trainor had identified and distributed a new standard stainless steel ball since the last meeting and this had been used by several participants to measure the softening point of rosin. The preliminary results showed poor reproducibility and so the round-robin was postponed until the method could be discussed at this meeting.

It was concluded that the method of sample preparation could be affecting the results. It was agreed that another round-robin would be run with all test samples being prepared by W. Trainor. The results of this round-robin would be discussed at the next task group meeting.

*D01.34.05—Softening Point of Rosin and Rosin Derivatives by Automatic Ring and Ball Apparatus*—P. Zawislak, Chairman, explained that the purpose of this study was to determine why an automatic Ring and Ball method gave a higher softening point and less precision than the standard manual softening point method (E 38). To help resolve this problem, a representative of Walter Herzog attended the task group meeting to discuss his company's instrument. He speculated that the glycerol and the thermometer stem correction could be associated with the discrepancy. It was agreed that a silicone fluid would be evaluated in place of glycerol prior to running another round-robin.

*D01.34.04—Iodine Value of Tall Oil Fatty Acids Using Solvents Other than Carbon*—J. Bowers, Chairman, explained that the purpose of this method was to find a solvent to replace the current hazardous solvent, carbon tetrachloride. Previous experimental work had confirmed that isooctane was a satisfactory replacement and so the chairman agreed to draft a new method for discussion and preliminary approval at the next meeting.

*D01.34*—J. Russell reported that the final nine current standard methods for which D01.34 has responsibility were approved in a society ballot. All of D01.34's current methods are now fully approved and will be published as editing is completed.

## SUBCOMMITTEE D01.35 SOLVENTS, PLASTICIZERS, AND CHEMICAL INTERMEDIATES

### R.G. Montemayor, Chairperson

*D01.35.10—Hydrocarbon Solvents and Ketones*—S.A. Yuhas and R.L. Hinrichs, Co-Chairpersons. A new method, "Boiling Point Distribution of Hydrocarbon Solvents by GC," has passed the society ballot and has been given the designation of D 5399-93. It will appear in Vol. 6.04 of the *1994 ASTM Book of Standards*. This method provides an alternate volatility-measurement technique to D 086 or D 1078 for narrower boiling range hydrocarbon solvents, which are outside of the scope of D 2887. It is easily adapted for on-line analysis and allows close and timely monitoring of solvent boiling point distribution during manufacture.

The round-robin to evaluate a capillary GC method for the determination of total aromatics in mineral spirits as part of D 3257 is in progress. Some problems were encountered in this round-robin such as different column lengths were sent to participating laboratories. R.G. Montemayor is assuming responsibility for this round-robin. Two negatives on D 3257 on a D01 ballot were successfully resolved. D. McCunn withdrew his negative contingent with an editorial change. R. Janusz withdrew his negative with the understanding that a round-robin is being run for the capillary method. F. Tate withdrew his negative on D 1363 at the D01.35 ballot. These documents will be balloted at the next level.

There is continued comment from industry representatives whether to continue testing the odor of solvents by D 1296, "Method of Testing Odor of Volatile Solvents," in view of the health hazard associated in performing the test. The opinion of J. Brezinski and D01.22 has been solicited. The consensus opinion of D01.35.10 is that the standard should be kept, although odor testing is being dropped or made optional in all D01.35 solvent specifications as they come up for review and reapproval.

*D01.35.20—Reactive Monomers*—J.E. Frugé, Chairperson. Eight standards passed society ballot and seven standards passed the D01 ballot. An attempt will be made to come up with a repeatability value for D 4415, "Test for Dimer in Glacial Acrylic Acid," from various laboratories which run the test. It is not possible to run a round-robin for this method because of the unstable nature of the acrylic acid. A capillary GC method for D 3362, "Test Method, Purity of Acrylate Esters by GC," was forwarded to D01.35.60 for evaluation.

*D01.35.30—Chemical Intermediates*—J. Morrison, Chairperson. Nine standards were balloted at the D01 level. One received a negative related to the bias statement. The bias statement will be changed to correspond to the latest revision (August 1992) of ASTM Form and Style Manual. A GC method for the determination of "Acetone in Diacetone" was submitted to D01.35.60 for consideration.

*D01.35.40—Plasticizers and Ester Solvents*—J.E. Lawniczak, Chairperson. A negative was received on D 1296, "Standard Test Method for Odor of Volatile Solvents." J.C. Stanley requested that D 1296 be discontinued because of the hazards associated with smelling solvents and the existence of alternate methods to identify solvents. As in D01.35.10, the consensus was that D 1296 is an important industry standard although odor testing is being dropped or made optional in a number of solvent specifications. The negative was found to be not persuasive. A late D01 negative by W. Zimmt was discussed. He believes that a statement in Section 1.2 of D 3728, "2-Ethoxy Ethyl Acetate," explaining its use in urethanes is an endorsement. The task group found this negative not persuasive on the basis that the statement in question was not an endorsement but a technically valid statement.

*D01.35.50—Coordination*—L. Forrest, Chairperson. A number of standards from D02, D 16, and E15 were discussed. Among these was the round-robin to determine improved precision on D86 using a wider-necked flask and thermometer centering device. There is also a round-robin being planned by D02 on the effect of viscosity of liquids on their flash points when determined by D93 Procedure A or Procedure B. This round-robin activity was relayed to D01.22.01 and a request was made to indicate any interest of participation by organizations which are D01 members to R.G. Montemayor. There was no response received from D01 members. R.G. Montemayor reported that his findings showed that when Procedure A is used, the observed flash point is depressed as the viscosity of the sample increases. Revisions of D 848, "Acid Wash Colour of Industrial Aromatic Hydrocarbons," D 849, "Copper Corrosion of Industrial Aromatic Hydrocarbons," and D 850, "Distillation of Industrial Aromatic Hydrocarbons," all under D16 jurisdiction passed, society ballots.

*D01.35.60—Method Development*—R.G. Montemayor, Chairperson, assumed the responsibility of the task group. Three new methods are under development. (1) Acetone in Diacetone Alcohol—GC methods for this analysis were received from J. Morrison, J. LeBeau, and J. Frugé. They will be reviewed, and a method for a possible round-robin will be recommended. (2) Capillary GC Method for Acrylate Esters—GC methods for this analysis were received from J. Morrison and J. Frugé. They will be reviewed and a recommendation will be made. (3) Capillary GC Method for Total Aromatics in Mineral Spirits—a round-robin to establish precision data for the capillary GC method is in progress. Not all of the data have been received.

Two new methods will be considered for development. First, a capillary GC method for MAK/MIAK purity, cf. D 3893. Data were received from S. Yuhas on works done by T. Rendl and R.E. Shirley. Second, a capillary GC method for D 3545, "Alcohol Content and Purity of Acetate Esters."

*D01.35*—J. D. Frugé, of Hoechst Celanese, has volunteered to be the Vice-Chairperson of D01.35. S.A. Kail, formerly of Supelco Inc., has relinquished her position as Secretary of D01.35 for personal reasons. The position of Secretary is now vacant and D01.35 is requesting for a volunteer to fill the position.

Bias statements were discussed during both task group meetings and the D01.35 meeting. D01.35 probably has the largest number of standard methods that produce quantitative results. Recognizing the enormous time, effort, and resources required to develop a valid bias statement on existing standards, D01.35 is taking the position that as standards come up for review, revision, or reapproval, a definitive bias statement will be included in the standard. For those standards whose bias cannot be determined, the bias statement will be in the form of examples given in the latest revision (August 1992) of the Form and Style Manual for ASTM standards, e.g.: (1) bias cannot be determined because there is no available material having an accepted reference value; and (2) the test method has no bias because the value of the test result is defined only in terms of the test method. The bias of new standards or existing methods that are currently involved in round-robins whose bias can be determined, will be determined using gravimetrically prepared calibration blends using NIST-traceable materials.

## SUBCOMMITTEE D01.36 CELLULOSE AND CELLULOSE DERIVATES

### G.Y. Moore, III, Chairman

*D01.35*—In old business, Method D 4085, "Standard Test Method for Metals in Cellulose by Atomic Absorption Spectrophotometry," was successfully reballoted after a negative vote had been addressed. The method will appear on the August society ballot. A new standard, D 5400, "Test Method for Hydroxypropyl Cellulose," was approved in a society ballot.

In new business, two methods require action this year, D 4794, "Determination of Ethoxyl Substitution in Cellulose Ether Products by Gas Chromatography," and D 4795, "Nitrogen Content of Soluble Nitrocellulose-Alternative Method." Both will be submitted for D01.36 ballot after editorial and technical changes have been made by G.Y. Moore.

Seventeen additional methods must be balloted prior to 1995. Five of these methods will be on the next D01.36 ballot. J.S. de Wit will prepare methods D 1348 (Moisture in Cellulose), D 1787 (Pentosans in Cellulose), D 1926 (Carboxyl Content of Cellulose), and D 2438 (Silica in Cellulose). G.Y. Moore will prepare D 1795 (Intrinsic Viscosity of Cellulose).

D01.36 will continue recruiting efforts. The steps will be to identify some of the most important standards maintained by D01.36 and to produce a publicity piece. We also will identify trade associations which have direct interests in these methods, and target those associations for publicity.

## SUBCOMMITTEE D01.37 INK VEHICLES

### A.N. Scarlatti, Chairman

*D01.37.01—Resin Solutions*—J.T. Daust, Chairman. The proposed "Standard Practice for the Preparation of Oil-Based Resin Dispersions by the Hot Plate Stirrer" has been resurrected and will be reballoted. A. Scarlatti distributed three proposed test methods for review by D01.37. He asked that members comment on the methods for (1) "The Preparation of Resin Solutions Using a Waring Blender," (2) "The Preparation of Resin Solutions Using a Hot Air Gun," and (3) "The Preparation of Resin Solutions Using a Hot Oil Bath." D. DeLegge offered to present a proposed method using a combination of blender and hot oil bath dissolution at the January 1994 meeting.

*D01.37.03—Gelability of Resin Solutions*—A.N. Scarlatti, Chairman, reported that the proposed practice D 5165 for "The Laboratory Preparation of Gelled Vehicles Using a Resin Kettle" has passed society ballot. J. Fetsko mentioned that the Shepherd Flow Plat can be used with an inclined plane to measure vehicle gelation. Such a method can be considered in the future.

Method D 4040, using the Laray Viscometer, is the current ASTM method for determining ink rheology. H. Brooks is reviewing this method to determine whether it needs to be modified for the evaluation of ink vehicle gelability. He will also request that W. Duke write a method for the new Duke Viscometer.

*D01.37.04—Standard Ink Oil*—J.T. Daust, Chairman. Pennzoil has put 3,000 gallons of mid-specification Magiesol 47 aside for use as a "Standard Oil" for industry users desiring a reference solvent. This Standard Magiesol 47 can be obtained by calling 1-800-Magie 47. P. McCabe agreed to have Exxon set aside some volume of mid-specification Exxprint 283D (Exxon's heat-set ink oil) for those wanting a Standard Exxprint 283D. This solvent should be available soon. Solvent properties for these standard oils will be available upon request. A.N. Scarlatti will write an announcement for the *American Ink Maker* to publicize the availability of the two standard ink oils made possible by D01.37. We are still in search of some linseed oil producer who will make a standard alkali refined linseed oil (ARLO) available to the industry.

*D01.37.05—Quality Control of Ink Oils*—J.B. Sardisco, Chairman, reported that the proposed PKP method for "The Quality Control of Ink Oils" has passed D01.37 ballot and will appear on the D01 ballot. D01.37.05 is interested in other methods suitable for the quality control of ink oils. Recommendations will be considered.

*D01.37.06—Pigment Dispersibility of Paste Ink Vehicles*—A. Mercado, Chairman. Nothing was reported regarding a new test for analyzing pigment dispersibility. A.N. Scarlatti will contact pigment flush producers to ask for a recommended laboratory flushing procedure using the Baker Perkins Mixer. H. Brooks will contact several producers to obtain a proposed method to evaluate dry pigment dispersion.

*D01.37.07—Resin/Solvent Compatibility*—D.P. Frisch, Chairman. A.N. Scarlatti distributed a proposed method for "Determining the Compatibility of Resin in a Given Ink Solvent by Precipitation Temperature," and requested D01.37 members to review it. The method will be discussed in January before being submitted for D01.37 ballot. J. Fetsko mentioned that method D 5010 on "Clarity and Cleanliness of Paint and Ink Liquids" may have applicability for determining resin/solvent compatibility. No further investigation of this method is planned at this time. P. McCabe suggested that this task group solicit input on resin compatibility from ink makers and will suggest this interaction to some of his contacts.

*D01.37.08—New Membership*—D.P. Frisch, Chairman, welcomed three new members to D01.37: Dan DeLegge (Lawter), Edward Casserly (Pennzoil), and Priscilla Zawislak (Hercules). We encourage other industry specialists to consider membership in ASTM and D01.37.

*D01.37—Ink Vehicles*—A.N. Scarlatti, Chairman, reported that no new test task groups were initiated at this session. Other avenues of investigation regarding the preparation, use, and/or evaluation of ink vehicles are being sought. No liquid ink vehicle procedures are currently under investigation. With the continued evolution of aqueous and high-solids flexo and gravure inks, D10.37 would like to invite representatives of liquid ink vehicle suppliers and ink producers to join our group so that we can effectively pursue the development of liquid vehicle test methods. Committee members were encouraged to continue the solicitation of new members, particularly those in the vehicle production, ink, and printing fields.

# DIVISION 40 PAINT PRODUCTS APPLIED ON SITE

## SUBCOMMITTEE D01.42 ARCHITECTURAL FINISHES

### L. Schaeffer, Chairman

*D01.42.03—Porosity of Paint Films*—C. Tatman, Chairman, reviewed the group's scope and updated attendees on the progress of developing mineral oil saturant and fluorescent dye techniques to measure porosity. Both techniques will be used in the next round-robin if preliminary tests with the fluorescent dye technique in the chairman's laboratory show promise. Three test paints will be prepared, one with low oil-absorption $TiO_2$; a second with high oil-absorption $TiO_2$; and a third with low oil-absorption TiO2 plus opaque polymer. Each cooperator will receive test paints and uncoated panels for individual test applications, and also a set of panels, pre-coated with the same paints, for parallel tests to eliminate interlaboratory variables related to panel preparation.

*D01.42.04—Wet Adhesion of Latex Paints*—W. Vanderslice, Chairman, reviewed the status of the proposed new test method using a Gardner scrub machine rather than a "weight pull-off" technique to measure wet adhesion. Data were distributed from the chairman's laboratory comparing the wet adhesion of two latex semigloss control paints representative of good and of poor wet adhesion. These were applied over an air-dried gloss alkyd primer versus special treated and untreated polyester panels. Scrub tests using either water or a liquid abrasive cleaner readily distinguished between the control paints applied over the alkyd primer. Neither type of polyester panel showed discernible difference between control paints because both paints failed immediately over the untreated and adhered perfectly to the treated panels. As a next step toward the development of a suitable standard test surface, black plastic panels will be coated with a medium-oil alkyd varnish of defined composition. These panels will be tested by the chairman periodically to determine if the fully cured surface will distinguish between the two control paints.

As a refinement of the test procedure it was decided to make mandatory the use of the 7 mil clearance side of the Dow Latex Applicator for applying test paint films. Variables to be investigated will include aging of test paints for one-half, one, and seven days; shortening the water exposure time to 30 minutes; increasing the size of the crosshatch cuts prior to soaking; and using only water during scrubbing. These variables will be evaluated prior to running a round-robin.

*D01.42.05—Adhesion of Latex Paints to Chalky Surfaces*—A. Leman, Chairman, reviewed the group's previous scope and definition, which was to develop an "artificial/synthetic" chalky substrate in order to evaluate the degree of chalk adhesion of test paints. A consensus was reached to change the scope to develop a "naturally weathered" painted panel as a standard chalky substrate. Some felt that such a substrate could be produced and sold to the coatings industry by commercial exposure companies. It was agreed to use Method D 4214 to rate the degree of chalk, and to perform chalk adhesion testing on panels that had weathered to a "5" chalk rating. The chairman will send the cooperators several weathered chalky panels for the dual purpose of rating the degree of chalking and evaluating the interlaboratory reproducibility of the rating method. The objective of the next meeting will be to review candidate paint formulations that are known to chalk rapidly on exterior exposure, and to decide on several to be compared by actual weathering. One formulation will ultimately be chosen for producing standard weathered chalky panels to be used in round robin adhesion testing by this group.

*D01.42.09—Color Development in Tinted Latex Paints*—L. Schaeffer, Chairman, passed around copies of recently developed Method D 5326, along with a list of proposed editorial and technical revisions. In the ensuing discussion, additional useful suggestions and comments were offered. The consensus was to submit the document with these additional changes for subcommittee ballot.

*D01.42.21—Project Planning*—S. LeSota, Chairman, solicited ideas for developing new test methods. Early water-resistance, heat stability, alkali-resistance, and wet-edge time were properties suggested for evaluation. Of these "wet-edge" appeared to be of most interest. The British Standards Institute defines wet-edge as "length of time that the edge of a wet, painted area remains workable." (At the D01.42 meeting the question was raised as to whether the correct term was "wet-edge time" or simple "wet-edge.") Members will send the wet-edge methods they presently use in their laboratories to the D01.42 chairman. These will be reviewed at the next meeting and a task group chairman appointed if feasible.

*D01.42.22—Guides for Testing Architectural Coatings*—H. Ashton, Chairman, briefly reviewed the status of the waterborne (D 5324) and solvent-borne (D 5146) guides. He reported that on D01 ballot, standards D 2932, D 3323, D 3383, and D 3425, all now incorporated into D 5146, were voted to be withdrawn. These are now being submitted to society ballot. A revision of D 3730, "Guide to Testing High-Performance Interior Architectural Coatings," to make it agree more closely with D 5146 and D 5324, was balloted successfully at the D01 level. A late comment will be reviewed for inclusion in the next revision of D 3730. Revisions applying to both D 3730 and D 5146 were circulated and should be completed by November for balloting. J. Weaver requested and was granted time to discuss the possibility of using HIPAC (D 3730) materials in the lead abatement program in place of more conventional coatings being sold at extremely high prices. He suggested that those present keep aware of the activities of E06.23 on this issue.

*D01.42.23—Gloss Definitions*—S. LeSota, Chairman, stated that an acceptable qualitative definition for gloss has been sought for over a decade. *The Compilation of ASTM Standards Definitions* lists five different qualitative definitions of gloss, not including one from D01.16. Even more controversial is the attempt to define gloss levels quantitatively. A recently balloted attempt to do this received a strong negative response. It appears that a number of paint companies do not want ASTM to establish measured numerical gloss values for defining popular gloss terms like flat, semigloss, and high gloss, because these values might be interpreted as specifications by their customers. A suggestion was made to revive a previous proposal, that gloss level categories be identified by numerals instead of names. A preliminary vote by the group members was favorable to this suggestion, which will be pursued further at the next meeting. (At the D01.42 meeting, comments were made as to the need for 20° gloss readings in addition to 60° readings for glossy latex paints. The task group chairman cited the previously determined adequacy of 60° readings for defining gloss levels in architectural coatings, and asked for specific examples of contrary experience.)

*D01.42.24—Application Properties of Architectural Paints*—J. Price, Chairman, invited discussion of the term "natural spreading rate." A valid definition of that term is needed, along with a statement relative to its usefulness and limitations. From the previous round-robin, it was shown that it is difficult and perhaps impossible to determine natural spreading rates with a reasonable degree of precision, due to the many subjective elements involved. Definitions from various sources are being sought, and the chairman will present a candidate definition and other relevant statements at the next meeting, for further discussion.

*D01.42.25—Scrub Resistance of Latex Paints*—D. Licata, Chairwoman, led a discussion to resolve several issues about variables in the test method. Recent work showed film thickness to vary by as much as 25%, depending on the width of the applicator doctoring edge. Variations in shim types and possible need to secure the shim to the glass plate were also discussed. The decision was made to specify the use of the Dow Latex Film Applicator and a brass shim in the upcoming round-robin. Other variables will be minimized by the use of a standard scrub brush, scrub medium, and detergent solution.

*D01.42.26—Burnish Resistance of Latex Paints*—W. Vanderslice, Chairman, reviewed his laboratory's dry burnishing data that compared the effect of "moleskin" vs "cheesecloth" as abrasive surfaces at cycle ranges from 5-200, using the Gardner scrub machine and a special Gardner abrasive-surface attachment. Three latex paints (interior eggshell, exterior flat, interior high-PVC) were evaluated. Results showed the moleskin to be markedly less aggressive than cheesecloth, and unable to distinguish between the exterior flat and interior high-PVC paints at any number of cycles, whereas cheesecloth produced discernible differences at as low as five cycles. It was agreed that the first round-robin would be run with cheesecloth, the test paints to be an interior eggshell, a moderate-PVC interior flat, and a high PVC flat. Burnishing (i.e., the change in 85° gloss) would be measured after 50 cycles under both dry and wet conditions. Commercial paints of low, medium, and high PVC will serve as references to determine acceptable degrees of burnishing in this or in a subsequent round-robin.

*D01.42.28—Paint and Coating Removers*—T. Sliva, acting Chairman for V. Scarborough, reviewed the group's scope, which is to develop a method for evaluating the efficiency of removers for organic coatings on horizontal metal or wood surfaces. Draft No. 1 of the proposed test method was reviewed and editorial and technical comments noted for incorporation into the next draft. T. Sliva exhibited wood and metal panels to be used in the first round-robin. These were coated with: (1) three vari-colored coats of a latex semi-gloss enamel; (2) three vari-colored coats of an alkyd gloss enamel; (3) two coats of an alkyd varnish; and (4) two coats of a polyurethane varnish. For the first round-robin the four cooperators will use one remover. Results will be reported at the next meeting.

*D01.42.29—Testing of Exterior Stains*—A. Leathers, Chairperson. At this initial meeting, it was agreed that the group's first objective would be to develop a "Standard Guide to Testing Exterior Stains." Definitions are to be established and ASTM methods and tests applicable to stains determined. Possible attributes for testing include wet-edge, lapping, penetration, water repellency, water-beading, UV protection, water sensitivity, recoatability, color retention, tanning resistance, adhesion, and various physical properties. The chairperson will gather information from stain producers, raw material suppliers, and related wood industries organizations, and will also solicit methods through ASTM-related publications.

*D01.42—Subcommittee Meeting*—Standards D 1848, D 4062, D 4400, D 4946, and D 5007 will be submitted to D01 ballot for reapproval. It was agreed to submit the following standards to D01.42 ballot for withdrawal: D 2931 and D 3358 because they have been incorporated into D 5324, "Guide to Testing Water-Borne Architectural Coatings"; D 2833 because it is duplicated in D 5146 and D 5324; and D 1736, "Efflorescence of Interior Wall Paints," because the problem it deals with is essentially obsolete. Editorial and technical changes in the new standard D 5326, "Color Development in Tinted Latex Paints," were approved for submission to D01.42 ballot. Standard D 1849, which is overdue for reapproval, will be modified to include ASTM rating scales in response to a previous negative vote, and the method will then be resubmitted for D01.42 ballot. Comments from the floor on the reports presented by task groups 21 and 23 have been incorporated parenthetically into the minutes of those task groups.

## SUBCOMMITTEE D01.46 INDUSTRIAL PROTECTIVE COATINGS

### G.W. Gardner, Chairman

*D01.46.02—Surface Preparation*—K.A. Trimber, Chairman, reported that D 4417, "Standard Test Methods for Field Measurement of Surface Profile of Blast Cleaned Steel," passed the April society ballot and was approved by the Committee on Standards to be published in Vol. 6.02 in 1994. K.A. Trimber added that this task group will begin immediately working on revising this standard to use Dixon's test for discarding outliers, adding comments to Notes 3 and 5 on other suitable comparators and gages, and add information on the type of equipment used to generate precision.

D 2092, "Standard Practice for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting," passed D01/D01.46 ballot for reapproval with no negatives and proceeded to society ballot. D01.46 has been unsuccessful in attempts to move this standard to another subcommittee. S. Boocock has previously agreed to revise D 2092 with a scope limited to zinc-coated (galvanized) structural-type steel. S. Boocock reports that he has begun gathering information from experts that should enable him to also address treatments used on electroplating-type galvanized surfaces. These experts say that treatments listed in D 2092 are still being used, as well as new techniques utilizing silicates and zirconates. The Department of Defense adopted this standard on July 18, 1991, to replace MIL-T-12879A(MR). S. Boocock will submit a revised D 2092 for fall D01.46 ballot.

*D01.46.03—Repainting*—G.W. Gardner, Chairman, reported that the new standard D 5402, "Standard Practice for Assessing the Solvent Resistance of Organic Coatings Using Solvent Rubs," passed the April 1993 society ballot and will be published in Vol. 6.02 in 1994. Draft No. 1 of this new standard was submitted to the task group May 11, 1990. With publication in 1994, it will have taken approximately four years to move this standard from concept to completion. G.W. Gardner thanked the task group and D01.46 members for their diligent efforts to keep this standard moving, and for the fine practice they had produced.

The next phase on this standard is to develop a precision statement. D. Chance will do an intralaboratory evaluation using a minimum of three materials with two replicates. In addition, G.W. Gardner will contact DJH Designs to determine if their new MEK Rub Test Machine should be evaluated at the same time as the D 5402 Standard Practice.

D 1014, "Standard Test Method for Conducting Exterior Exposure Test of Paints on Steel," is due for reapproval. G.W. Gardner contacted D. Grossman, Chairman of D01.27, who agreed to take responsibility for this standard since it fits better in the scope of D01.27 than in D01.46. The D01 Executive Committee approved this change in responsibility in their June 27, 1993, meeting.

D 610, "Standard Test Method for Evaluating Degree of Rusting on Painted Steel Surfaces," will de due for reapproval in 1994. J.K. Rule, Supervisor General Engineer by Direction of the Commanding Officer of the Department of the Navy, has requested that additional photographic standards of 3.0 and 0.3% rust be made available in conjunction with D 610. Present photographs depict 0.03, 0.1, 1.0, and 10.0% (9, 8, 6, and 4 ratings, respectively). Adding photographs of the two additional rust grades would correspond to ratings 7 and 5, and thereby D 610 would cover essentially all rust levels needed for field evaluation. The task group unanimously agreed that it would be worthwhile to have photographs of all six rust grades from 9 through 4. However, adding only two photographs to the present four photos would not provide consistency in appearance of the new photos with the old photos. The task group will proceed with producing new photographs for all six rust grades. Since SSPC and ASTM jointly approved D 610, then it will be necessary to coordinate any changes with SSPC. G.W. Gardner will work with S. Boocock of SSPC in this regard. Since new photographic standards will be expensive and may require considerable time in which to obtain approval by ASTM and SSPC. D 610 will be sent out to D01.46 for reapproval with a cover letter stating our intent to produce new photographs representing the six rust grades, 9, 8, 7, 6, 5, and 4.

D 4752, "Standard Test Method for Measuring MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub," is past due for revision. This standard was last balloted in 1992. The negative and comments from this ballot have been incorporated into a new draft. With completion of D 5402, D 4752 will be modified to include latest improvements in D 5402 and sent out for D01.46 ballot. In addition, precision information will be generated using three materials and two replicates.

*D01.46.04—Pull-Off Adhesion*—M.E. McKnight, Chairman, voted negative on D 4541, "Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers," on April 1993 society ballot. This negative pointed out an incorrect formula in Footnote 7 and received unanimous consent to be persuasive. Two items correcting D 4541 were sent to D01.46 ballot. S. Boocock originally voted negative on these ballot items because he did not receive attachments. S. Boocock withdrew his negative when attachments were received. D 4541 then passed D01 ballot with only an affirmative comment. However, this draft of D 4541 did not have both the items balloted in D01.46. M.E. McKnight will attempt to get this corrected prior to going to society ballot.

*D01.46.07—Inspection*—G.W. Gardner reported that D 3276, "Standard Guide for Painting Inspectors (Metal Substrates)," received two negatives on a D01 ballot to withdraw. These negatives received unanimous consent to be persuasive. D 3276 is being revised and many improvements have been incorporated. Several new improvements were added during the task group meeting and the new draft of D 3276 will be sent out for D01.46 ballot.

Draft No. 5 of the new "Standard Guide for Painting Inspectors (Concrete and Masonry Substrates)," will be sent out for D01.46 ballot.

*D01.46.12—Sampling*—K.A. Trimber, Chairman, reported that Draft No. 2 of the new proposed "Standard Practice for Field Sampling of Coating Films for Analysis of Heavy Metals," received one negative and four comments, in a D01.46 ballot. J.C. Weaver's negative and J. Raska and B. Wittenbaum's comments were discussed at the January 1993 meeting. S. Boocock and J.J. Brezinski's comments, along with the mentioned negatives and comments, have been incorporated in Draft No. 3 of the new standard. Several additional improvements were suggested by task group members. K.A. Trimber will incorporate these changes and send them out as a Draft No. 3 for D01.46 ballot.

*D01.46*—G.W. Gardner, Chairman, welcomed four new members to the D01.46 roster: Vikki Young, of Tennant Company, Minneapolis, MN; John Zarzeckie, of Soils & Materials Engineers, Plymouth, MI; David Kuniega, of Penndot Materials & Testing, Harrisburg, PA; and Robert Olen, of Cooper Powder Systems, Franksville, WI. New business discussion centered around developing an Index or Guide for Industrial Protective Coatings that would be similar to other ASTM guides such as those for Architectural Paints and Waterborne Paints. Further consideration will be given to this subject at the January 1994 meeting.

## SUBCOMMITTEE D01.47 MASONRY TREATMENTS

### F. Gale, Chairman

*D01.47.01—Water Repellency of Treated Masonry*—V. Scarborough, Chairperson. The meeting was chaired by F. Gale in the absence of the chairperson. F. Gale distributed Draft No. 3 of the "Proposed Test Method for the Determination of the Water Repellency of Treated Masonry," and data developed during an initial screening of the revised method by V. Scarborough.

The task group decided to do the following: evaluate the effect of capillary absorption as opposed to immersion of the test specimens, review the data related to the absorption of untreated specimens, and look into a standardized substrate and coat the test substrates at a specified spreading rate. Five cooperators have agreed to participate in a round-robin to be conducted before the next meeting to evaluate these suggested changes to the method. The chairwoman will report the results of this study at the January meeting.

*D01.47.05—Water Vapor Transmission of Treated Masonry Substrates*—L.V. Mertz, Chairman. The meeting was chaired by F. Gale in the absence of the chairman. F. Gale distributed Draft No. 7 of the proposed test method and the data from the recently completed round-robin. The round-robin consisted of evaluating the WVT of brick coated with a urethane, and two silane/siloxane blends, one of 8% solids content and one of 20% solids content.

A discussion of the results and comments from task group attendees resulted in several recommendations for revisions to the method before the next round-robin. Six cooperators have agreed to participate using the same materials from the previous study. The chairman will report the results at the January meeting.

*D01.47.06—Freeze/Thaw Resistance of Treated Masonry*—L. Stark-Kasley, Chairman, led a discussion of the results of the second round-robin. Two-inch mortar cubes were treated with three different waterproofing materials and subjected to 50 freeze/thaw cycles. Weight change and changes in the condition of the test substrate were recorded weekly.

Because correlation between labs has been good with both round-robins, the chairman will prepare the method for submission to D01.47 letter ballot. She will revise the method to improve the descriptions of physical deterioration noted for the test specimens after freeze/thaw and expand the section describing the test substrate preparation. The method will be reviewed by task group members and a precision statement developed.

*D01.47.10—Evaluation of Field Applied Treatments*—E. McGettigan, Chairman, reviewed Draft No. 1 of the proposed "Test Method to Determine the Water Absorption of Cured Concrete Treated with Water Repellent." The method was reviewed by the task group and the chairman will revise the method to include all editorial and technical comments received.

The chairman made a presentation showing the method for sealing the untreated sides of the test cores and suggested weighing procedures. It was the decision of the task group to initiate a round-robin. Five cooperators have agreed to participate. The chairman will present the data at the next meeting.

*D01.47.11—Research and Planning*—F. Gale, Chairman, discussed a letter from A. Wendt suggesting formation of a task group to establish a guide for surface preparation of brick substrates. F. Gale has agreed to reactivate Task Group 04 and will work with T. Subasic, of the Brick Institute of America, to develop such a guide. Other outstanding work the subcommittee is looking into includes the development of a standard for textured coatings and setting up a task group on bond breakers.

*D01.47.12—Water Beading*—P. Harvey, Chairman, led a discussion on the scope and purpose of the task group. While the chair agrees that water beading is an important physical property used in the field to assess the effectiveness of applied treatments, it was the opinion of several attendees that the task group be discontinued. It was decided that members of D01.47 should be polled for a decision about the future of the task group.

*D01.47*—T. Sliva reported that the revision to D 1734 had successfully completed D01.47/D01 ballot without receiving any negatives. The revision will now proceed to society ballot. F. Gale reported on the reactivation of Task Group 04 to establish a guide for the preparation of brick substrates.

# DIVISION 50 PAINTS FOR FACTORY APPLICATION

## SUBCOMMITTEE D01.51 POWDER COATINGS

### N.D. Emily, Chairman

*D01.51.02—Hiding Power of Powder Coatings*—N.D. Emily, Acting Chairman. Round-robin test results on film thickness determination were returned by four collaborators. Data analysis and task group discussion revealed that the types of measurement devices and calibration procedures used have a significant impact on the accuracy of the determination of contrast ratio hiding powder. Film thickness test methodology is to be rewritten to include refinements identified to date and another round-robin test series is to be conducted. The alternate weight method is to be revised and submitted for task group review prior to the January 1994 session.

*D01.51.03—Spray Characteristics of Powder*—C. Merritt, Chairman. The second draft of the method for determining powder transfer efficiency was reviewed. Both flat panel and penetration, recessed surface, and efficiency techniques have been tested. Repetitive tests using the same powder have produced laboratory results with a variation of ± 2%. The draft will be revised and circulated for subcommittee evaluation in a timely manner so that the second draft will be available for discussion at the next committee session.

*D01.51.04—Optical Properties of Powder*—N.D. Emily, Acting Chairman. D 5382, "Guide to the Evaluation of Optical Properties of Powder Coatings," has been approved by society ballot and will appear in the 1994 publication of volume 6.01.

*D01.51.05—Specific Gravity of Powder*—D. Schneider, Chairman. The results of round-robin testing from five collaborators were reviewed. For the wet/weight method, test data repeatability improves as the operator familiarity increases. Discussion of the gas pycnometer results of two collaborators revealed that procedures of testing were somewhat different in individual sample selection. Three types of powder were measured using each method. They were epoxy, urethane, and metallic. Additional testing will be conducted to refine the methodology.

*D01.51.06—Review of Particle Size Measuring Techniques*—D. Pont, Chairman. The results of the round-robin where thirteen collaborators measured the particle size of one powder sample were discussed. Test equipment included laser transmission and back scatter; liquid; sedimentation; and sieve types. Indications are that particle size distribution values are equipment dependent and are relative rather than absolute measurements. Reporting will require the inclusion of the specific equipment and test parameters used to measure the particle size and distribution. A first generation draft of a guide is scheduled to be distributed for discussion at the January 1994 session.

*D01.51*—A technique for measuring uncured powder film thickness is being investigated for feasibility with the assistance of an instrumentation supplier. A planning session will be scheduled to discuss this and other new projects at the January 1994 D01.51 meeting.

## SUBCOMMITTEE D01.53 COIL COATED METAL

### R.J. Tucker, Chairman

*D01.53.01—Pretreatment of Metal Substrate*—H.E.G. Rommal, Chairman, distributed the third draft of the proposed practice, "Determination of Chromium in Metal Surface Treatments by X-ray Fluorescence," prepared by D.C. Steele. Minor changes will be made and the standard will be submitted for D01.53 ballot.

*D01.53.03—Accelerated Weathering*—D.A. Cocuzzi, Chairman, discussed the Testing Outline for the D01.53.03 Testing Program. To date, 20 sets of panels comprising all of the common coating chemistries for the construction marketplace (acrylic emulsion, fluorocarbon, plastisol, polyester, and silicone polyester), in a variety of colors, have been received. A few key issues were discussed and agreed upon:

(1) Only one South Florida exterior real-time weathering site will be utilized, since evidence indicates little variation exists between different sites for long-term exposure work.

(2) Only one start time will be established (probably fall 1993) for the same reason previously stated.

(3) It is important to get the real-time weathering started as soon as possible since it will be the rate-determining step in the generation of data for this study.

(4) Real-time weathering will de done in triplicate for each sample tested. This will obviate the need to repeat any of this long-term weathering testing by providing a sound statistical basis upon which to base our comparisons and conclusions.

Regarding the accelerated weathering devices to be considered, the list includes UV-Condensation Devices (with UV-A340 bulbs), Xenon Arc, EMMA-NTW, and the Dew Cycle Weatherometer. Length exposure in each is to be determined at the next meeting.

The type of measurements to be taken will be color change (using Hunter ΔE 0°/45° geometry, in addition to that recommended by SAE J1960), gloss retention (@ 60°), and chalk (utilizing the tape chalk method).

*D01.53.04—Dry Film Thickness of Coil Coatings*—G.R. Pilcher, Chairman, began a general discussion about the evaluation of and draft practice for the DJH Designs Dry Film Thickness (DFT) Measurement System. This device bores a conical hole into the coating, and the DFT is measured by measuring the lateral distance across the "cut," and converting this reading to the DFT by dividing by 10 (as a result of the cutting angle being precisely 5° 42'22", a 10:1 slope). While existing magnetic and eddy current equipment exists to measure DFT on cold-rolled steel and aluminum, respectively, no such device offers the apparent accuracy and ease of use for HDG as the one marketed by DJH Designs. We felt that the approach which D01.53.04 should take would be to draft a general practice for using the device, and then establish a round-robin test to measure interlaboratory correlation. R.M. Olszewski, J.C. Larsen, and M.R. Lagowski will create the draft document by November 1, 1993. D.A. Cocuzzi has the responsibility to develop a film thickness standard to use (in the future) by groups involved in the round-robin testing. Such a standard will need to be a coated material, deposited onto a base material of a different color, such that the DFT can be precisely determined and that the DFT is uniform across the surface of the panel (standard). These standards will also need to be available by November 1. J.J. Peters has agreed to contact DJH Designs about attending the Ft. Lauderdale ASTM meeting, brining a unit to demonstrate the method and perhaps helping in the creation and/or revision of the draft document. J.J. Peters will also attempt to bring a Sabourg Drill, which may function in the same manner as that of the DJH device. The round-robin testing should include at least six laboratories, measuring at least three different samples, with each sample measured at least in triplicate. Samples may include a plastisol, conventional coil coatings in white and colors, and a three-coat system where the topcoat is actually a clear coat.

*D01.53*—R.S. Tucker reviewed recent subcommittee ballots:

•D 4147, "Applying Coil Coatings Using the Wire-Wound Drawdown Bar," and D 3794, "Standard Guide for Testing Coil Coatings," will move on for D01 ballot.

•D 3003, "Pressure Mottling and Blocking Resistance of Organic Coatings on Metal Substrates," received one negative vote, which was declared persuasive. It will be revised by D.A. Cocuzzi and resubmitted for balloting.

## SUBCOMMITTEE D01.55 FACTORY APPLIED COATINGS ON PRE-FORMED PRODUCTS

### E.A. Praschan, Chairman

*D01.55.06—Coatings for Plastics*—M.A. Lutterbach, Chairman, reported that a second draft of revised method D 3002 will be prepared for D01.55 ballot.

*D01.55.09—Transfer Efficiency Under Production Conditions*—R.S. Diem, Chairman, reported that the new method for "Determination of Transfer Efficiency Under General Production Conditions," has received society approval and will be published as D 5286. Editorial revisions are under consideration and the chairman will survey users to determine if precision data is available.

*D01.55.10—VOC of Radiation Cured Materials*—E.A. Praschan reported that this new method passed the society ballot and will be published as D 5403.

*D01.55.11—Mar Resistance of Automotive Coatings*—A.E. Rutkiewic has accepted chairmanship of this task group, replacing J.S. Komjathy. The group reviewed two proposed draft methods which will be consolidated prior to performing a round-robin evaluation. Four laboratories have agreed to participate.

*Organizational Meeting on Non-Conductive Coatings for Electrical Protection Equipment*—L.E. Thieben has agreed to chair this new task group, which decided that a new standard is needed to provide guidance for performance of coatings used on electrical equipment. A draft method will be prepared based upon a method currently used by Ontario Hydro-electric.

*D01.55*—E.A. Praschan has succeeded G.R. Pilcher as Subcommittee Chairman.

The membership as of June 2, 1993 is 34. R.S. Herling is a new member. Method D 2199, "Method for Measurement of Plasticizer Migration From Vinyl Fabrics to Lacquers," will be submitted to D01 ballot for reapproval.

## SUBCOMMITTEE D01.56 PRINTING INKS

### J.M. Fetsko, Chairman

*D01.56.02—Lightfastness of Prints*—J. Robins, Chairman, reported on plans to develop a precision statement for D 3424, "Test Method for Lightfastness and Weatherability of Printed Matter." There is only one set of data by "natural daylight under glass" and five sets of carbon arc data.

*D01.56.12—Viscosity of Liquid Inks*—J. Daugherty, Chairman, reported that five laboratories tested the viscosities of four pre-diluted inks and a standard oil with two Shell and two EZ Zahn cups. Repeatability was the same for both types of cups, but reproducibility of the EZ Zahn was surprisingly better than the Shell.

*D01.56.14—Setting of Heatset Inks*—D. Ness, Chairman, distributed a proposed method for running comparative setting tests using the Sinvatrol on wedge plates prints. An exploratory round-robin is planned.

*D01.56.15—Density (wt/gal) of Paste Inks*—P. Ford, Chairman, distributed statistical analysis showing excellent precision of ink densities by the weight-per-gallon cup. On the other hand, precision of the VOC40 method was inversely related to the VOC40 level.

*D01.56.17—Guide for Printing Inks*—J. Fetsko, Chairman, reported that D 5010, "Guide for Testing Printing Inks and Related Materials," was balloted to include the new standards D 5328 on VOC40, D 2090 for clarity of paint and ink liquids, and D 5264 on the Sutherland Rub Tester.

*D01.56.20—Technical Coordination*—B. Blom, Chairman, reported that several NPIRI task forces are working on test methods of interest to D01.56 and recommended that closer cooperation between the two groups be arranged.

*D01.56.21—Print Abrasion*—G. Vandermeersche, Chairman, distributed copies of a proposed standard covering the use of the GV-CAT for testing cans and other metal decorated products. It was suggested that he incorporate the procedure as Part B of D 5181, which covers testing of flat printed matter by the GA-CAT.

*D01.56.22—Drying of Oxidizable Inks*—B. Blom, Chairman, distributed copies of a proposed test method in which the drying of oxidizable inks is evaluated by squalene resistance using a modified Sutherland Rub Tester. In order to develop a precision statement from existing round-robin data, it was suggested that the time to achieve 80% reflectance be adopted as the endpoint. It was also suggested that the manual rub-off method be included even though the precision was poor.

*D01.56.25—Degree of Radiation Curing*—E. Kobylarz, Chairman, reported that according to a review article by A.K. Davies, there are 27 methods of detecting degree of cure in radiation curing. He will examine MEK resistance using the Control Cure Analyzer and manual rubbing.

*D01.56.27—VOC40 of Non-Heatset Paste Inks*—P. Ford, Chairman, reported that the round-robin data discussed in D01.56.15 will be the basis for a revised precision statement for D 5328, "Test Method for VOC40 of Non-Heatset Paste Inks at 40°C."

*D01.56.28—Tinting Strength of Liquid Inks*—D. Ness, Chairman, reported that, when using a cuvette for spectrophotometric determination of tinting strength, readings shifted as much as 10% within 20 minutes. Problems of compatibility with the tinting base must also be resolved.

*D01.56.29—Membership*—J. Daugherty, Chairman, welcomed new members Ed Casserby, of Pennzoil; Dan DeLegge, of Lawter; and Alex Semijalec, of J.M. Huber Corp.

*D01.56.31—Opacity*—A. Semijalec, new Chairman, agreed to conduct a literature survey on methods for measuring opacity of printing inks. Opacity differences can be detected by visual examination of drawdowns but not by instrumental readings on ink films printed at SWOP densities.

## SUBCOMMITTEE D01.57 ARTISTS' PAINTS AND RELATED MATERIALS

### M.D. Gottsegen, Chairman

[Summary report of meeting held April 30, 1993 with the National Art Materials Trade Association in Miami Beach, FL.]

*D01.57.02—Lightfastness of Pigments*—T. Vonderbrink, Chairman. Concurrent D01.57/D01 balloting of a revision to D 4303, alerting users to variabilities between and within exposure procedures, continues. This ballot should resolve a negative by W. Ketola.

T. Vonderbrink reported on progress towards choosing an alternative Fluorescent Lamp Apparatus for D 4303. A homemade unit is currently specified, and the lamps called for are no longer being manufactured. The proposed alternate apparatus, Atlas's HPUV unit, is being considered, and recent test results were distributed; repeatability was good. In the latest round of testing, the control was changed from total radiant exposure to the use of reference colors of known durability.

R. Kinmonth expressed concern regarding the limitations of D 4303 relative to the varying effects of energy from different spectral regions and different sources. For now, revision of D 4303, specifying the new apparatus and using reference colors to control the duration of exposures, is seen as an improvement. T. Vonderbrink agreed to draft changes to D 4303.

*D01.57.04—Specification of Artists' Paints*—A. Spizzo, Chairman. B. Gavett presented a proposal for testing 10 pigments suspected of darkening in mass tone. Manufacturers currently making paints with these pigments will prepare masstone samples for exterior Florida exposure. B. Gavett will provide the exposure panels; G. de Calan will provide spectrophotometer readings in order to quantify the amount of change: and T. Vonderbrink and R. Kinmonth expressed an interest in testing the pigments in the Atlas HPUV. B. Gavett will proceed with test arrangements.

The revisions of D 4302, D 5067, and D 5098, regarding a change in the type size requirement for labeling, has received a negative from J.T. Luke. M. Gottsegen recommended withdrawing the item from balloting until J.T. Luke can attend the next meeting, and D01.57 agreed. Manufacturers were asked to submit comments and further proposals for a more comprehensive revision of this requirement to M. Gottsegen.

J.T. Luke submitted several editorial revisions to the Suitable Pigments Lists (Table 1) of D 4302, D 5067, and D 5098. Most of the changes are corrections to the original list regarding Colour Index Numbers—corrections supplied by the Technical Director of the Colour Index. Several further changes and corrections will have to go to D01.57 ballot and will be distributed to D01.57 before the next meeting.

*D01.57.07—Physical Properties*—R. Gamblin, Chairman. The long-term focus of this group is to become involved in new studies to examine the physical properties of artists' materials. It is hoped that research institutions with related interest will be able to help with the studies. In this regard, D. Stulik, Acting Scientific Program Director with the Getty Conservation Institute, described the efforts of the Institute to develop a comprehensive collection of information on modern materials which could be useful in future conservation work.

A draft of a short survey was distributed by R. Gamblin. The survey's aim is to assess different groups' perceptions of artists' materials, and asks respondents to identify themselves as a conservator, artist, manufacturer, scientist, or educator. The results may help to identify areas for further study, and may also provide information concerning conceptions and misconceptions about certain materials. Members of D01.57 were asked to review the draft and submit comments to R. Gamblin.

*D01.57.08—Toxicity Labeling*—W. Stopford, Chairman, reported that the phrase, "Possible Cancer Agent" was added to the Chronic Hazard Statements Annex Al of D 4326.

*D01.57.10—Consumer Evaluation*—J.T. Luke, Chairwoman. In J.T. Luke's absence, M. Gottsegen presided. D 5383, "Standard Practice for the Visual Determination of the Lightfastness of Art Materials by Art Technologists," and D 5398, "Standard Practice for the Visual Determination of the Lightfastness of Art Materials by the User," are still in society ballot. However, J.T. Luke has submitted some minor changes regarding the naming of the isolating mask used in the assessment portion of the standards, and regarding the Munsell Notation used to describe the color of the mask. These changes will be sent for D01.57 balloting.

J.T. Luke also submitted a research report regarding the development of D 5383 and D 5398 which must be approved by D01.57.

*D01.57.11—Gouache Paints*—T. Takigawa, Chairman, reported that pigments will be retested in Xenon Arc exposure due to an error in the initial tests, and also that several changes have been made in the proposal for the gouache standard as a result of comments received since the last D01.57 meeting.

Many other changes were recommended during D01.57 discussion. Since consensus has not been reached regarding a definition of the different kinds of gouache paints, discussion of their differences has been removed from the proposed standard. It was also proposed that this new standard be made more like D 4302, D 5067 and D 5098, in that only paints containing pigments which have a rating of Lightfastness I or Lightfastness II in Table 1 would be considered in conformance. A second Table 2 will be created to accommodate the other pigments used in gouache paints but which have lightfastness ratings of III, IV, or V, and thus would not be suitable for conformance to the standard. T. Takigawa expects to have another draft of the proposed standard by the next D01.57 meeting.

*D01.57.12—Determination of Toxicity*—W. Stopford, Chairman. C. Jacobson, of the U.S. Consumer Product Safety Commission, reported on enforcement efforts in regard to labeling of art materials for chronic health hazards under LHAMA, which incorporates D 4236. The USCPSC and the Customs Service are working together to target the importation of children's art materials for inspection. C. Jacobson also mentioned that the CPSC would still like to see a relaxation of the requirement in D 4236 that toxicologists be board-certified, but D01.57 declined to act.

The most recent version of the proposed "Test Method for Determining Extractability of Metals from Art Materials" was discussed and further revised. The revised draft will be sent for D01.57 balloting.

The proposed "Standard Test Method for Determining Bioequivalency of Nitroaromatic Amines in Art Materials" need intralaboratory precision testing, but this will be postponed several years until laboratories have had time to gain experience in using the method. The Art and Craft Materials Institute has agreed to manage the testing when the time comes.

W. Stopford reported interest from overseas regarding the adoption of a new ASTM Formaldehyde Analysis Test Method. He is also evaluating a list of official European Community warning phrases which may be appropriate for inclusion in Annex A1 of D 4236.

*D01.57.14—Colored Pencils*—I. Seymour, Chairman. The proposed specification for colored pencils still requires a suitable method for determining light-fastness—relying upon pigment manufacturers' data is not a good option. D01.57 believes that a method should be selected and pencils be tested as finished products. M. Gottsegen recommended considering the "Standard Practice for the Visual Determination of the Lightfastness of Art Materials by Art Technologists." I. Seymour agreed to consider this and other options, and also agreed to further consider appropriate and suitable test substrates.

*D01.57*—A motion to reconfirm C. Jacbson of the U.S. CPSC as an affiliate member of D01.57 was passed.

M. Gottsegen announced that, due to increasing job responsibilities, he would resign as Chairman of D01.57 after the January 1994 meeting. B. Gavett, currently Secretary of D01.57, accepted a nomination as the next Chairman for a limited term, subject to confirmation and appointment by D01.

## DIVISION 60
## PAINT APPLICATION

### SUBCOMMITTEE D01.61 PAINT APPLICATION TOOLS

### F.B. Burns, Chairman

*D01.61.01—Paint Brushes*—T. Sliva, Chairman, reported results from round-robin No. 3 on paint brush leveling and brush cleanup. These results indicated relatively good correlation between the three cooperators, who have returned results to date. A proposal was made to evaluate the effect of reducing the paintout area to 500cm$^2$. These cooperators agreed to try this modification, and to return paintout charts to D/L Laboratories for comparison under the Leneta Leveluminator. Paint types are to be clarified for this procedure.

Cleanup procedure reducing the air-dry (open-time) to 30 minutes appeared to be an improvement. This procedure will include a guide for use of paints with bristling materials of various colors. The chairman will determine the next course of action based on results of the previously mentioned, and will try to develop a first draft of a guide.

*D01.61.02—Paint Rollers*—J.F. Price, Chairman, stated that the next round-robin materials would be in the cooperators' hands by August 1993. Following this, discussion was held on tests for fabrics used to make paint roller covers. It was agreed that a recommendation would be made at the D01.61 meeting to add a new task group to develop tests for the physical characterization of woven paint applicator fabrics.

*D01.61.05—Bulk Density of Filaments and Bristle*—T. O'Brien, who acted as Chairman for W.B. Bond, stated a revised test procedure was issued which incorporates the use of a metal insert in the test. Two cooperators reported improved reproducibility and reduced standard deviations with the modified procedure. Ketema and DuPont will each send out tapered filament samples in each of three cross-sectional configurations. Five cooperators will test these samples using the new test method.

*D01.61.06—Buckling Resistance of Filaments*—F. Burns acted as Chairman for the meeting in the absence of W.B. Bond. C. Nelson, a co-worker of Bond, reported results from the first round-robin testing using the Ketema test device. Only three of the five cooperators had submitted test results and comments; and filaments were provided only by polymers. Nevertheless, there was relatively good agreement between the three cooperators. Clamp slippage was discussed and recommendations to overcome this problem will be included in the next testing along with other method revisions. Another round-robin test will be initiated before the January meeting.

*D01.61*—F. Burns, Chairman, proposed an expansion in the D01.61 scope to incorporate components of paint applicators as well as paint applicators. A letter ballot will be sent out to the D01.61 members before submission to D01.90. The new task group, entitled "Physical Characterization of Woven Paint Applicator Fabrics," was activated as D01.61.03 with E. Lowder, of Collins & Aikman, as Chairman. It will have its formative meeting in January.

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# January 1993 Subcommittee Reports of ASTM Committee G03

The June meeting of ASTM Committee G03 on Durability of Nonmetallic Materials was held on June 27-30, 1993 at the Franklin Wyndham, Philadelphia, PA. The present membership of G03 is approximately 90.

### Future Meetings

January 23-26, 1994—Crown Sterling, Fort Lauderdale, FL.
June 26-29, 1994—Marriott Crab Tree Valley, Raleigh, NC.
January 22-25, 1995—Hyatt Regency, San Antonio, TX.
June 25-28, 1995—Atlanta, GA.

## SUBCOMMITTEE G03.01 EDITORIAL AND DEFINITIONS

### M.J. Crewdson, Chairman

*G03.01.01—Terminology*—M.J. Crewdson, Chairman. Revisions to Terminology G 113 include an editorial change in the title to correct an error in printing. The correct title is "Terminology Relating to Weathering Tests of Nonmetallic Materials." A revision to Black Box definition will be balloted at the G03.01 level.

*G03.01.02—Conditioning and Handling*—M.J. Crewdson, Chairman. A second draft of the new proposed "Practice for Conditioning and Handling" will be submitted for G03.01 ballot. This incorporates changes suggested in the previous ballot.

*G03.01.03—Statistics*—M.J. Crewdson, Chairman. A first draft of a proposed "Guide to Application of Statistics to Weathering Test Results" will be submitted for G03.01 ballot.

## SUBCOMMITTEE G03.02 NATURAL ENVIRONMENTAL TESTING

### J.S. Robbins, III, Chairman

G03.02 is revising G 24, "Standard Practice for Conducting Exposures to Daylight Filtered Through Glass," to reduce the variability of the glass used in under-glass exposure cabinets. The latest draft of G 24 will include a requirement to pre-age this glass for three months.

A round-robin will be conducted during the Summer of 1993 to intercompare direct-normal ultraviolet irradiance measurements made using a shading-disk technique. If successful, changes will be incorporated into the latest draft of G 90, "Standard Practice for Performing Accelerated Weathering of Nonmetallic Materials Using Concentrated Natural Sunlight."

A status report was distributed concerning a black panel thermometer round-robin which has been in progress. Additional temperature measurements will be performed to test the effect of coating differences on temperature measurements.

G 7, "Standard Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials," will be sent for G03.02 ballot.

## SUBCOMMITTEE G3.03 SIMULATED AND CONTROLLED ENVIRONMENTAL TESTS

### R.M. Fischer, Chairman

R. Fischer reported that the revision to Practices G 23 and G 26 regarding temperature of water used for specimen spray will be on the August 1993 society ballot. The revision of Practice G 53 to include a lamp application appendix passed the March 1993 society ballot.

A draft standard "Practice for Conducting Light-Exposure Tests of Non-metallic Materials Using Horizontal Specimen Tray Xenon Arc Apparatus" passed G3.03 ballot. However, comments received on the G3.03 ballot were technical in nature and must be incorporated into a separate draft for G3.03 ballot.

Negatives on the draft "Guide for Addressing Variability in Natural and Accelerated Testing of Non-metallic Materials" were found persuasive. A revised draft will be prepared for G3.03 ballot.

A draft precision statement for inclusion in practices G 23, G 26, and G 53 was discussed. This precision statement is based on results from round-robin tests which have been conducted during the past seven years.

Inclusion of the precision statement in these standards will be balloted in G3.03.

Negatives on the revision to practices G 23, G 26, and G 53 regarding manufacturers' reference for equipment availability were found persuasive. Revisions based on these negatives will be prepared for G3.03 ballot.

W.D. Ketola reported on format for revision to G3.03 standards to make them performance–based instead of equipment–based. The revisions will be similar in structure to ISO 4892, Parts 1-4, and will be submitted for G3.03 ballot.

## SUBCOMMITTEE G3.05 REFERENCE MATERIALS FOR EXPOSURE TESTS

### W.D. Ketola, Chairman

W.D. Ketola provided a status report on round-robin activities. No additional analysis of the polysulfone round-robin data was done. Several laboratories have not returned the polyethylene specimens. Analysis of polystyrene yellowing data according to Method E691 showed a large reproducibility standard deviation for measurements made by reflectance. The reproducibility standard deviation was better for polystyrene yellowing determined by transmission measurements. The within-laboratory repeatability for yellowing of polystyrene was excellent for both measurement methods. Additional analysis of polystyrene yellowing data from xenon arc exposures by N. Searle showed that the measurement of CIE b* was a major source of the total variability. Although all polystyrene exposures were conducted according to SAE J1960, data from most participating laboratories did not fall within the delta b* specification given in SAE J1960.

The subcommittee discussed and agreed on a format for preparation of a draft, "Standard Guide for the Characterization and Use of Reference Materials in Exposure Tests."

## SUBCOMMITTEE G03.07 LIAISON AND HARMONIZATION

### R.A. Kinmonth, Chairman

Task group members reported on their efforts to harmonize standards prepared by other committees with those prepared by G03. Considerable discussion centered around methods for assuring that U.S. input on natural and artificial weathering testing was gaining consideration in ISO TCs and TGs.

## SUBCOMMITTEE G3.08 SERVICE LIFE PREDICTION

### J.W. Martin, Chairman

This was the inaugural meeting of G3.08 on service life prediction. During this meeting, the scope of the subcommittee was discussed and two technical presentations were given.

The proposed scope of G3.08 is to develop standards useful in facilitating the comparison of laboratory and field/service exposure results and in quantitatively predicting the service life of materials, components, and systems exposed anywhere outdoors. Among these standards will be methods for: (1) uniquely, completely, and with sufficient detail characterizing the initial properties of a material, component, or system; (2) quantifying degradation; (3) designing experiments; (4) developing methods for quantitatively characterizing outdoor exposure environments; (5) analyzing service life data; (6) deriving statistical and mathematical models useful in predicting service life; (7) designing computerized databases for storing and retrieving the collected data; and (8) developing methods for discovering knowledge from the stored data. The work of this subcommittee will be closely coordinated with other standards and non-standards groups.

Technical presentations were given by David Correll, Director of the Smithsonian's Environmental Research Center, who gave a short history of the Smithsonian Institution's activities in monitoring spectral solar radiation; and by David J. Duncan, of Dow Chemical Company, who gave a presentation on the use of computer image processing in quantitatively imaging coating defects.

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# Society Meetings

## CDIC .................................. SEPT.

### *"Clean Air Initiatives"*

President Paul Guevin, Jr., of P.R. Guevin Associates, presented a Past-President's pin to Jim Flanagan (1991-92), of Flanagan Associates, and informed the membership that a pin would be mailed to Alipio R. Rubin, Jr. (1992-93), of Rubin Dispersions.

Donald J. Roettker, of B.H. Roettker Co., Inc., presented the Hüls Gavel to President Paul R. Guevin.

Hugh Lowrey, of Perry & Derrick Co., Inc., the Society Delegate to the recently formed Ohio Paint Council, reported on the OPC's September meeting. He stated that the proposed lead abatement bill, soon to be brought to the Ohio legislature, presents no immediate concern to the industry. No new taxes or further restrictions are contained. Mr. Lowrey said that Title V permits, being considered by Ohio, are as "industry friendly" as permitted by the Federal Government. Ohio EPA will likely not meet the November 15 deadline for drafting regulations to comply with the Federal Clean Air Act of 1990.

The chairman of the OPC, Mike Quinn, stressed the importance of "making our voice known" and the need for total cooperation in establishing solid networks between industry and lawmakers.

The meeting's technical speaker was Jim Bassett, of Eastman Chemical Products. Mr. Bassett's topic was "An Overview of the U.S. EPA Clean Air Initiatives Impacting Solvents."

The speaker reviewed the history of solvent restricting regulations from Los Angeles County's "Rule 66" to the current "Clean Air Act of 1990." Title I of this act, he pointed out, deals with air quality. Areas of "ozone nonattainment" are placed in one of five categories, dependent on the severity of the nonattainment. Title II calls for the establishment of 13 new Control Technique Guidelines (CTGs); in effect establishing rules for 13 heretofore unregulated coatings markets or end-uses. Additionally, noted the speaker, it establishes National Rules for certain coatings types, develops the protocol to the reg/neg process, and updates the 12 existing CTGs. Title III addresses Hazardous Air Pollutants (HAPs). This deals with restrictions of a specific list of chemicals defined as toxic. Mr. Bassett listed the following commonly used coatings solvents: ethylene glycol, MEK, MIBK, toluene, xylene, and ethylene-based glycol ethers. Toxicity status, rather than VOC, is the focus of this regulation, he pointed out; with some of the determinations being highly questionable. Mr. Bassett explained that the listed chemicals are ultimately slated to be done away with by the year 2000. Title VI addresses stratospheric ozone and restricts CFCs and 1,1,1 trichlorethane.

The speaker then addressed related topics: the EPA's voluntary 33/50 Program, the Montreal Protocol, and federal guidelines limiting state regulations.

In conclusion, Mr. Bassett suggested compiling a list of solvents, targeted for eventual elimination by being "attached" to the various regulations. Approaches to achieving this elimination—incineration, replacement, process modification, and low VOC coatings—must be carefully considered.

---

*Q. Please elaborate on the compilation of the HAPs list, which you defined as "arbitrary"?*

A. The SARA 313 list was used and very questionable logic applied to some selections.

John Avery,
*Secretary*

## CHICAGO ........................... SEPT.

### *"Titanium Dioxide"*

Montreal Society member John E. Hall, of Tioxide Canada, presented a technical talk on "Titanium Dioxide, Recent Developments."

Mr. Hall stated that recent technological developments in titanium dioxide ($TiO_2$) have resulted in improved products available to the paint formulator. These include super-durable grades with improved dispersibility and gloss, universal grades with increased formulating latitude, and high-gloss grades for various specialty applications. He reviewed each type of treatment and their effect on durability, dispersibility, gloss, and tinting strength.

The newest development, remarked Mr. Hall, is in ultra-fine $TiO_2$ that allows its use in semi-clear or almost clear coatings for wood, or in plastics or cosmetics, where this non-opacifying grade absorbs UV light to increase product durability by acting as a non-migratory UV absorber.

Clifford O. Schwahn,
*Publicity*

## KANSAS CITY ................... SEPT.

### *"Workforce 2000"*

The Society Officers for 1993-94 were announced as follows: President—Lawrence Murphy, of Tnemac Co.; Vice President—Yvonne D'Arcy, of Cook Composites & Polymers; Secretary—William Porter, of Hillyard Industries, Inc.; Treasurer—Randall Ehmer, of Walsh & Associates; and Society Representative—Norman Hon, of Davis Paint Co.

Kim Nee, of Mozel, Inc., presented President Murphy with the Hüls Gavel, and Norman Hon accepted the Past-President's Pinon behalf of Ms. D'Arcy.

The Society Committee Chairmen for the coming year were introduced: By-Laws—Terryl Johnson, retired; Educational—Peter Cerra, of Tnemac Co.; Environmental—Nick Schmedding, of Tnemac Co.; Joint Meeting—Thomas Hilton, of Naz-Dar/K.C. Coatings; Manufacturing—Terry Barnett, of Naz-Dar/K.C. Coatings; Membership—Nick Dispensa, of Patco Specialty Chemical; Program—Yvonne D'Arcy; Publishing—Robert Leslie, of Tnemec Co.; and Technical—Steve Bussjaeger, of Davis Paint Co.

The first speaker of the evening was Betty Anastasio. Her presentation was entitled, "Workforce 2000."

Ms. Anastasio spoke on the changing corporate outlook and how that impacts the workforce. She predicted that prospective employees will need more technical skills than just a high school diploma, but not a four-year degree.

She discussed a new program called, "Tech-Prep." This program is designed for high school students taking special skills classes and then going on to two years of college for technical training.

The second speaker of the meeting was Edward Orr, of BYK-Chemie. According to Mr. Orr, due to environmental reasons, there has been a shift to higher solids and water-based coatings. This switch has led to other problems: wetting, dispersing, surface flow, and leveling.

Surface tension is much lower for mineral spirits (about 20 dynes/cm) compared to water (approximately 73 dynes/cm), stated the speaker. This means that without an additive, the material will not wet out the surfaces. Mr. Orr stated that new silicon technology can wet down to about 25 dynes/cm without foam generation of a flurosurfactant. He emphasized the need to have an understanding of polysiloxane technology and what it is you want to accomplish.

William Porter,
*Secretary*

## Constituent Society Meetings and Secretaries

**BALTIMORE** (Third Thursday—Martin's West, Baltimore, MD). Albert Holder, U.S. Navy, David Taylor Research Ctr., Code 2841, Annapolis, MD 21402-5067.

**BIRMINGHAM** (First Thursday—Strathallan Hotel, Birmingham, England). P. Hassall, Newtown Industrial Paints Ltd., Silica Rd., Amington Ind. Est., Tamworth, Staffs. B77 4DT, England.

**CDIC** (Second Monday—Location alternates between Columbus, Cincinnati, Dayton, and Indianapolis). John C. Avery, Cintech Industrial Coatings, Inc., 2217 Langdon Farm Rd., Cincinnati, OH 45237-4792 .

**CHICAGO** (First Monday—Sharko's Restaurant, Villa Park, IL). C. David Stromberg, Standard T Chemical, 290 E. Joe Orr Rd., Chicago, IL 60633.

**CLEVELAND** (Third Tuesday—Brown Derby, Independence, OH). Michael A. Wolfe, Seegott Inc., 5400 Naiman Pkwy., Solon, OH 44139.

**DALLAS** (Second Thursday following first Wednesday—Raddison Hotel, Dallas, TX). Paul Kaplan, Cookson Pigments, Inc., 2001 San Miguel Dr., Plano, TX 75704.

**DETROIT** (Second Tuesday—meeting sites vary). Tedd L. Strobehn, Boehle Chemical Inc., 19306 W. 10 Mile Rd., Southfield, MI 48037.

**GOLDEN GATE** (Monday before third Wednesday—alternates between Francesco's in Oakland, CA, and San Francisco's Tennis Club in San Franscisco, CA). Eve Stromquist, Flecto Corp., 100 45th St., Oakland, CA 94608.

**HOUSTON** (Second Wednesday—Houston Medallion Hotel, Houston, TX). Edward E. Boss, Bossco Industries, Inc., P.O. Box 680023, Houston, TX 77268-0023.

**KANSAS CITY** (Second Thursday—Cascone's Restaurant, Kansas City, MO). William T. Porter, Hillyard Industries, Inc., P.O. Box 909, St. Joseph, MO 64502.

**LOS ANGELES** (Second Wednesday—Steven's Steakhouse, City of Commerce, CA). Robert J. Skarvan, McWhorter Inc., 5501 E. Slauson Ave., Los Angeles, CA 90040.

**LOUISVILLE** (Third Wednesday—Executive West Motor Hotel, Louisville, KY). Andrew Traister, Courtaulds Coatings, Inc., 400 S. 13th St., Louisville, KY 40201.

**MEXICO** (Every fifteen days—Gabriel Mancera, Mexico City, Mexico). Sergio Rojas, Pinturas International, S.A. De C.V., Ganaderos 234, Col. Granjas Esmeralda, 09810 Mexico, D.F., Mexico.

**MONTREAL** (First Wednesday—Le Bifthèque Steakhouse, Ville St. Laurent, Quebec). Esther Rouleau McCarthy, Stochem Inc., 1455 32nd Ave., Lachine, Que. H8T 3J1, Canada.

**NEW ENGLAND** (Third Thursday—Sheraton Lexington Hotel, Lexington, MA, and other locations in Massachusetts and Rhode Island). Charles Shearer, ZENECA Resins, 730 Main St., Wilmington, MA 01887.

**NEW YORK** (Second Tuesday—Landmark II, East Rutherford, NJ). Cary Grobstein, Cardinal Color & Chemical, Inc., 50-56 First Ave., Paterson, NJ 07524.

**NORTHWESTERN** (Tuesday following frist Monday—Jax Cafe, Minneapolis, MN). Harold H. Christhilf, The Valspar Corp., P.O. Box 1461, Minneapolis, MN 55440.

**PACIFIC NORTHWEST** (Portland Section—Third Tuesday—Rose's, Portland, OR; Puget Sound Section—Third Wednesday—Barnaby's, Tukwila, WA; Vancouver Section—Third Thursday—Delphi Steak & Pizza, Vancouver). Richard C. Tomczak, Specialty Polymers, Inc., 17316 E. Riverside Pl., Bothell, WA 98011.

**PHILADELPHIA** (Second Thursday—Williamson's Restaurant, GSB Bldg., Bala Cynwyd, PA). Howard Salmon, Akzo Coatings, Inc. 100 Belmont Dr., Somerset, NJ 08873.

**PIEDMONT** (Third Wednesday—Ramada Inn Airport, Greensboro, NC). Bob Bishop, Ashland Chemical Co., P.O. Box 19959, Greensboro, NC 27419.

**PITTSBURGH** (Second Monday—Montemurro's Restaurant, Sharpsburg, PA). W. Ray Lyman, Jr, Ray Lyman & Co. 3462 Hills Church Rd., Export, PA 15632.

**ROCKY MOUNTAIN** (Monday following first Wednesday—Zangs Brewery, Denver, CO). Paul Delmonico, Old Western Paint Co., Inc., 2001 W. Barberry Pl., Denver, CO 80204.

**ST. LOUIS** (Third Tuesday—Salad Bowl Cafeteria, St. Louis, MO). James Lindsley, Akzo Resins, 2904 Missouri Ave., E. St. Louis, IL 62205.

**SOUTHERN** (Gulf Coast Section—third Thursday; Central Florida Section—third Thursday after first Monday; Atlanta Section—third Thursday; Memphis Section—bi-monthly on second Tuesday; and Miami Section—Tuesday prior to Central Florida Section). Robert Wayne West, Thompson & Formby, Inc., 10136 Magnolia Dr., P.O. Box 667, Olive Branch, MS 38117.

**TORONTO** (Second Monday—Cambridge Motor Hotel, Toronto). Kevin Pelling, Inortech Chimie Inc., 4135 LaStrada Hts., Mississauga, Ontario L5C 3V1, Canada.

**WESTERN NEW YORK** (Third Tuesday—meeting sites vary). Marko Markoff, 182 Farmingdale Rd., Cheektowaga, NY 14225.

## LOS ANGELES .................. SEPT.

### *"VOC Determination"*

The following Society Officers for the 1993-94 year were installed: President—V.C. "Bud" Jenkins, Consultant; Vice President—Philip C. Bremenstuhl, of Ashland Chemical, Inc.; Treasurer—John C. Kulnane, of Ameritone Paint Corp.; and Secretary—Robert J. Skarvan, of McWhorter, Inc.

Robert Baklin, of Hüls America, Inc., presented the President's Gavel to Mr. Jenkins.

Mr. Jenkins presented the Society's Past-President's Award to Sandra Dickinson, of Synergistic Performance Corp.

President Jenkins introduced the following Chairmen for the coming year: Environmental—Dave Mugee, of E.T. Horn Co.; Manufacturing—Herbert A. Molano, of Manufacturing Business Systems; Membership—Herbert A. Molano; Program—Philip C. Bremenstuhl; Publicity—V.C. "Bud" Jenkins; and Technical—V.C. "Bud" Jenkins.

Frank Peters, of Dunn-Edwards Corp., reported that there were eight graduates of the Society's "Paint Technology" course during 1992-93. The Outstanding Student Award was given to Steve Russell, of Smiland Paint Co.

Max Wills, of Cal Poly San Luis Obispo, was the technical speaker for the meeting. He reported on a new, more accurate method of VOC determination for coating, resins, etc.

Dr. Wills reviewed the standard method of VOC determination of a given paint sample which is typically done using ASTM Test Method D 3960 or EPA #24. This test method involves running a Karl Fisher titration for water content.

The speaker provided test data on numerous latex and water-based paint samples which displayed the higher degree of accuracy obtained by the test method. Dr. Wills also noted that the test method gave a higher coefficient of variation for paints with very low VOCs of less than 100 g/1.

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*Q. Was a determination made for PM and water mixtures, to look at variances in results at different water levels or take in account the water contamination of the PM which is a hygroscopic solvent?*

A. Methoxy propanol is not particularly hygroscopic. When we do the determination, we buy a gallon of this particular solvent, we dump molecular sieves into it and use it straight out of a bottle. On samples of this particular solvent, we do a Karl Fisher determination which takes about a .1 to .2 ml of Karl Fisher reagent which is less that an mg of water in a 5 ml sample, so that hasn't been a problem area.

Robert J. Skarvan,
*Secretary*

# Future Society Meetings

## Chicago

(Jan. 3)—"Low VOC High Gloss Corrosion Resistant Alternative"—Mike Wildman, McWhorter, Inc.

(Feb. 7)—"Rheological Additives for the Coatings Industry: Past, Present & Future"—Charles W. Glancy, United Catalyst, Inc.

(Mar. 7)—"Improved Rheological & Suspension Characteristics of Epoxy Amine and Water Reducible Coatings with Fumed & Hydrophobic Silica"—Maria Nargiello, Degussa Corp.

(Apr. 4)—"How Good a Color Match Can You Afford?"—John Du, Hüls America.

## Cleveland

(Nov. 16)—"Ethylene Vinyl Acetate Terpolymers and Their Application in Solvent-Free Systems"—Bill Currie, Nacan Products Ltd., and "Operation of the Ohio Paint Council"—John McGough, Ohio Paint Council.

(Jan. 18)—"Cleveland's Lead Initiative"—Judith Zimomra, Cleveland Commissioner of the Environment, Cleveland State University.

(Feb. 15)—"Quality/Waste Reduction Forum"—To Be announced.

(Mar. 15)—"Formulating Coatings with Fluorescent Pigments"—David Heyl, Day-Glo Color Corp., Sterle's Slovenian Country House, Cleveland.

(Apr. 12)—"Faux Finishing—Marblizing, Wood Graining, etc."—Mike MacNeil, Executive Caterers, Mayfield Heights.

(May 18)—BFGoodrich Tour. "Use of Statistical Methods in Emulsion Polymer Design"—Vic Stanislawczyk, BFGoodrich Co., BFGoodrich R&D Center, Brecksville.

## Dallas

(Jan. 13)—"The Making of $TiO_2$"—R.M. Schiller, Kerr-McGee Chemical Corp.

(Feb. 10)—"EPA Clean Air Initiatives"—Jimmy Bassett, Eastman Chemical Co.

(Mar. 3-4)—Southwestern Paint Convention, South Shore Harbour Resort, Houston, TX.

(Apr. 14)—"Waterborne Technologies for Low VOC"—Rich Johnson, Cargill, Inc.

(May 12)—Past-Presidents' Night.

## Golden Gate

(Nov. 15)—"A Solvent Property and Solubility Parameter Calculator"—Thomas M. Larson, Exxon Chemical Corp.

(Jan. 17)—"Improved Hiding in Thin Films"—Michael Issel, Sino-American Pigment Systems

(Mar. 14)—"Aqueous Polyurethane Dispersion for Coatings and Ink"—Richard C. Coogan, ZENECA Resins

(Apr. 18)—"Zero VOC Water-Based Coatings"—Authur Rich, Union Carbide Corp.

(May 16)—"Adhesion of Coatings to Plastic"—Rose Ryntz, Ford Motor Co.

(June 13)—Manufacturing Committee Seminar.

## Louisville

(Nov. 17)—"Oxazoline-Based Moisture Scavengers for High-Solids Coatings"—Thomas L. Johnson, ANGUS Chemical Co.

(Jan. 19)—Past-Presidents' Night.

(Feb. 16)—FSCT Officers Visit.

## Pacific Northwest

(Nov. 18)—"A Solvent Property and Solubility Parameter Calculator"—Thomas M. Larson, Exxon Chemical Corp.

(Jan. 20)—"Improved Hiding in Thin Films"—Michael Isseli, Sino-American Pigment Systems.

(Mar. 17)—"Aqueous Polyurethane Dispersion for Coatings and Ink"—Richard C. Coogan, ZENECA Resins.

(Apr. 21)—"Zero VOC Water-Based Coatings"—Arthur Rich, Union Carbide Corp.

(May 19)—"Adhesion of Coatings to Plastics"—Rose Ryntz, Ford Motor Co.

## Philadelphia

(Dec. 9)—"Formaldehyde in Coatings"—Rudi Moerck, Troy Corp.

(Jan. 13)—Joint Meeting with Philadelphia PCA.

(Feb. 10)—"New Isocyanate Curing Agents for Higher Solids Urethant Coatings"—Richard Wojcik, Olin Corp.

(Mar. 10)—"Good Tests—Bad Testing"—Saul Spindel, DL Laboratories.

(Apr.)—Awards Night.

(May 10)—Manufacturing Committee Presentation.

## Piedmont

(Nov. 17)—"Effects and Use of Silanes in Industrial Coatings"—Jerry Witucki, Dow Corning Corp.

(Feb. 16)—"Manufacture and Usage of Non-Mercurial Biocides in Coatings Production"—Marvin Landau, Hüls America Inc.

## New York

(Jan. 11)—"Novel High Performance Associative Thickener"—David Bryant, RHEOX, Inc.

(Feb. 10)—NYSCT/MNYPCA Joint Legislative Update and Exposition.

(May 10)—PaVaC Awards Night.

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# Elections

## CDIC

### Active

*Mayer, Timothy L.*—Synchem, Dayton, OH.
*Watkins, Edward D.*—Perry & Derrick Co., Inc., Cincinnati, OH.

## CHICAGO

### Active

*Basham, Tom L.*—Moline Paint Mfg. Co., Moline, IL.
*Bostian, Randall J.*—Morton International, Lansing, IL.
*Buchman, Jeffery I.*—Akzo Coatings Inc., Zion, IL.
*Chapelle, Donald C.*—Hallman Paints Inc., Sun Prairie, WI.
*Filippelli, Frank J.*—CPS Inc., Westchester, IL.
*Givens, Dwain T.*—Union Carbide Corp., Alsip, IL.
*Gofron, Kevin J.*—Weber Marking System, Arlington Hts., IL.
*Harris, Robert R.*—McWhorter, Inc., Carpentersville, IL.
*Keck, Mitchell T.*—Morton International, Woodstock, IL.
*McCormick, Michael P.*—Morton International, Lansing.
*McNulty, Michael J.*—Hallman Lindsay, Sun Prairie.
*Mueller, Douglas T.*—Moline Paint Mfg. Co., Moline.
*Nonweiler, Mark A.*—A.P. Nonweiler Co., Inc., Oshkosh, WI.
*Olson, Steven A.*—Hallman Lindsay, Sun Prairie.
*Porter, Cem A.*—Therma-Tru Corp., Butler, IN.
*Puchalski, Robert A.*—Morton International, Lansing.
*Riddick, Wayne T.*—Amoco Chemical Co., Naperville, IL.
*Silvestre, Recto L.*—United Coatings Inc., Kankakee, IL.
*Wahl, Robert F.*—Morton International, Lansing.
*Walsh, Robert E.*—Moline Paint Mfg. Co., Moline.
*West, Kenneth J.*—Therma-Tru Corp., Butler.
*Wheeler, Timothy S.*—Waste Management, Crestwood, IL.

### Associate

*Abbas, Syed H.*—U.S. Pigment Corp., Bloomingdale, IL.
*Berg, Scott R.*—LA Chemicals Ltd., Downers Grove, IL.
*Defalco, Steven J.*—Datacolor International, Westchester, IL.
*Hill, Christopher A.*—Henkel Corp., Willowbrook, IL.
*Jannetto, Philip J.*—Datacolor International, Naperville, IL.
*John, Robert A.*—Datacolor International, Naperville.
*King, Dennis J.*—Dominion Colour Corp., Hinsdale, IL.
*Malmanger, Dennis J.*—Micro Motion, Glenwood, IL.
*Nesom, Wilson F.*—SC Johnson Polymer, Racine, WI.
*O'Rourke, William B.*—Franklin Industrial Minerals, Nashville, TN.
*Raak, Keith A.*—Hilton Davis Co., Buffalo Grove, IL.
*Ryniec, David L.*—LA Chemicals Ltd., Downers Grove.

## DETROIT

### Active

*Atkinson, Timothy J.*—Gougeon Brothers Inc., Bay City, MI.
*Dine, Christopher T.*—BASF Corp., Southfield, MI.
*Hamma, Ralph*—Akzo Coatings Inc., Troy, MI.
*Karloff, Kristina S.*—BASF Corp., Southfield.

### Associate

*Rumer, Roger W.*—Miles Inc., Troy, MI.

### Retired

*Ozols, Atis Jr.*—Detroit, MI.

## KANSAS CITY

### Active

*Bellman, William C.*—Atlas Products, Des Moines, IA.

### Retired

*Korte, Robert*—Oswego, KS.
*Robertson, Ralph* —Valspar Corp., Prairie Village, KS.

## LOS ANGELES

### Active

*Abdelmalek, Mounir Morcos*—Paint Consultant, Glendale, CA.
*Brown, Melba R.*—Major Paint Co., Torrance, CA.
*Burhan, Alida*—Cargill Inc., Lynwood, CA.
*Carrere, Norvell G.*—Sinclair Paint Co., Los Angeles, CA.
*Curtis, James L. Jr.*—Old Quaker Paint Co., Victorville, CA.
*Diaz, Mark*—Coatings Resource Corp., Huntington Beach, CA.
*Dow, William*—Consolidated Color Corp., Hawaiian Gardens, CA.
*Farfan, Luis*—Artex Manufacturing Co., Culver City, CA.
*Forster, Wendell C.*—Morton International, Tustin, CA.
*Gavino, Lourdes G.*—Ameritone Paint Co., Long Beach, CA.
*Haanstra, Lloyd*—Wilshire Paint Co., Fresno, CA.
*Harding, Dana*—Consolidated Color Corp., Hawaiian Gardens.
*Holzrichter, Ed*—Cargill Inc., Lynwood.
*Khosraniani, Aflatoon*—Dura-Deck, Santa Ana, CA.
*Ko, Benita U.*—KB Trading, Montebello, CA.
*Lauricella, G.J.*—TCR Industries, Inc., La Palma, CA.
*Nicholson, Linda L.*—Morton International, Los Angeles.
*Pabich, Dan*—Day-Glo Color Corp., Cudahy, CA.
*Quinteros, Phillip*—Synergistic Performance Corp., Fullerton, CA.
*Rodriguez, Jorge J.*—Morton International, Tustin.
*Rowe, Anton A.*—Hirozon Sports Technologies, San Diego, CA.
*Sayta, Anil R.*—Major Paint Co., Torrance.
*Shah, Bhupendra M.*—Lilly Industrial Coatings, Montebello, CA.
*Sheth, Suresh*—Commercial Chemical, Santa Ana.
*Turk, Brian H.*—Union Carbide Corp., Torrance.
*Waltman, Patrick W.*—Cal-Western Paints, Sante Fe Springs, CA.
*Zywocienski, John M.*—Fine Line Paint Corp., Sante Fe Springs.

### Associate

*Adams, Chuck*—United States Can Co., Commerce, CA.
*Buckley, Andrew*—C.P. Hall Co., Torrance, CA.
*Darden, J. Winn*—Hüls America, Inc., Newport Beach, CA.
*Donohoe, Thomas D.*—Pacific Coast Chemicals Co., Los Angeles, CA.
*Drukenbrod, Mark L.*—Myers Engineering, Bell, CA.
*Galazin, Robert G.*—Henkel Corp., Ambler, PA.
*Gomez, Margarita*—P.T. Hutchins Co., Ltd., Los Angeles.
*Hagge, Don R.*—Union Carbide Corp., Torrance.
*Holst, Britton R.*—Royal Alliance, Fullerton, CA.
*Kowach, Richard*—Hüls America, Inc., Newport Beach.
*Moore, Rae*—P.T. Hutchins Co., Los Angeles.
*Nacorda, Romeo C.*—Old Quaker Paint Co., Victorville, CA.
*Rodriguez, Marté*—P.T. Hutchins Co., Ltd., Los Angeles.
*Severns, John D.*—Datacolor International, Mission Viejo, CA.
*Solt, David G.*—SC Johnson Polymer, Irvine, CA.
*Starks, Edward J.*—Cardinal Industrial Finishes, S. El Monte, CA.
*Young-Smith, Cathy*—McWhorter Corp., Los Angeles.
*Zabrecky, Gordon M.*—Pacific Coast Chemicals Co., Los Angeles.

### Educator/Student

*Chou, John Y.*—Polymerica, Upland, CA.

## NEW YORK

### Active

*Alli, Rasheid Ronald*—Mercury Paint Corp., Brooklyn, NY.

*Gaglani, Kamlesh K.*—Caschem Inc., Carlstadt, NJ.
*He, Z. Alex*—King Industries, Inc., Norwalk, CT.
*Kazmi, Syed Q.*—Troy Chemical Corp., Newark, NJ.
*Muldrow, Charles N.*—Muldrow Associates, E. Windsor, NJ.
*Schlossman, Mitchell L.*—Tevco Inc., S. Plainfield, NJ.
*Woodcock, David S.*—Shell Chemical Co., Short Hills, NJ.

*Associate*

*Batchelor, Robert M.*—United Catalysts Inc., Fair Haven, NJ.
*Fischer, Robert E.*—Pan Chemical Corp., Hawthorne, NJ.
*Gilbert, Darren R.*—Angus Chemical Co., Point Pleasant, NJ.
*Perez, Augustus J.*—Hickson Dumar Inc., Mahopac, NY.
*Scimecca, Frank*—Southern Clay Products, Newtown, PA.
*Smerak, Lance P.*—Sandoz Chemical Corp., Charlotte, NC.
*Tallia, Robert H.*—ChemCentral Corp., Cedar Knolls, NJ.

*Retired*

*Berman, Ben*—Hallandale, FL.
*Feld, Israel*—Red Bank, NJ.
*Luyster, Thomas Jr.*—Saddle Brook, NJ.

## NORTHWESTERN

*Active*

*Annis, Michael J.*—Valspar Corp., Minneapolis, MN.
*Wallace, David R.*—Valspar Corp., Minneapolis.

## SOUTHERN

*Active*

*Beckworth, Frank W.*—Coloray Paint Co., Lawrenceville, GA.
*Braaksma, Marvin*—Anvil Paint & Coatings, Largo, FL.
*Brand, Jack C.*—Spectralab Inc., Pinellas Park, FL.
*Garver, Clyde M.*—Anvil Paint & Coatings, Largo.
*Healey, Thomas*—Anvil Paint & Coatings, Largo.
*Huddleston, Tony A.*—Perma-Chink Systems, Knoxville, TN.
*Jefferson, Earnest*—Gilman Paint Co., Chattanooga, TN.
*Kenknight, Dale L.*—Akzo Coatings Inc., Norcross, GA.
*Lucas, Martin R.*—Grace Davison, High Point, NC.
*Moes, Philip*—Akzo Coatings Inc., Clinton, MS.
*Moore, Paulette*—Gilman Paint Co., Chattanooga.
*Niemi, Robert W.*—Sunbelt Coatings, Inc., Picayune, MS.
*Olson, John H. III*—Southeastern Chemical, Norcross.
*Ribovich, Roger J.*—Akzo Coatings Inc., Norcross.
*Rowe, Richard*—ECC International, Sylacauga, AL.
*Said, M.M.*—Polyplastex Inc., Largo.
*Senentz, Jules A. III*—Sheboygan Paint Co., Cedartown, GA.
*Stansell, Johnny C.*—Gilman Paint Co., Chattanooga.
*Warner, Arthur J.*—Pride Baker Paint Co., Olustee, FL.
*Woody, James E. Jr.*—Gilman Paint Co., Chattanooga.

*Associate*

*Carlson, Dennis L.*—ISK Biotech Corp., Memphis, TN.
*Day, Mike G.*—Van Waters & Rogers Inc., Norcross, GA.

**For information on how to obtain membership in the Federation, call Tori Graves or Marie Wikiera at FSCT Headquaters at 215-940-0777.**

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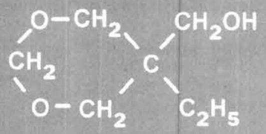

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**Sweden**
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**Fax (int.): +46 435 37067**

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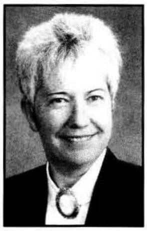
# People

NYCO® Minerals, Inc., Willsboro, NY, has announced the appointment of **Sara M. Robinson** as Director of Sales and Marketing for North America, South America, and Europe. Before joining NYCO, Ms. Robinson was Regional Sales Manager for Heuco-tech, Ltd., and Vice President of Sales and Marketing at Hitox Corp. of America.

A member of the Piedmont Society for Coatings Technology, Ms. Robinson was the recipient of a Women in Coatings Award for her work in technical publications.



**S.M. Robinson**

The American Society for Testing and Materials, Philadelphia, PA, has selected **Joseph M. Behrle** as the 1993 recipient of the Award of Merit. Mr. Behrle, retired from Lilly Industries, Indianapolis, IN, was cited for outstanding contributions and leadership in Committee D-1 in the development of standards for factory coated wood and allied building and preformed products. He has been a member of ASTM since 1959.

Raabe Corp., Menomonee Falls, WI, has named **William Jones** as Color Technician—Research and Development Laboratory. Mr. Jones will be responsible for the development of a high performance exterior touch-up paint product. In addition, he will be developing a training program on paint formulation, paint manufacture, and color science. Mr. Jones held previous positions with Dexter Corp. and Akzo Coatings before joining Raabe.

Degussa Corp., Ridgefield Park, NJ, has named **Reinhard W. Stober** as Vice President and General Manager of the Carbon Black Division within its Pigment Group. Dr. Stober will oversee the Division's North American marketing, sales, purchasing, manufacturing, distribution, engineering, and applied technology-related activities. He will also manage the engineering services in China. He most recently served as Director Business Section Carbon Black within the Inorganic Chemicals Division of Degussa AG in Frankfurt, Germany.

Micro Powders, Inc., Tarrytown, NY, recently announced the promotion of **Warren Pushaw** to the position of Vice President—General Manager. He will manage the domestic sales force, as well as the production, technical support, and quality assurance departments. Mr. Pushaw will oversee MPI's efforts toward obtaining ISO 9002 certification. He has been with the company since 1982, serving most recently as Vice President—Domestic Sales.

Amoco Corp., Chicago, IL, has announced two appointments within its organization. **James E. Fligg** has been elected Executive Vice President and Corporate Officer. He will retain his position as President. Mr. Fligg joined Amoco in 1968 and has held a variety of positions in Australia, Europe, and the United States. In 1989, Mr. Fligg became Executive Vice President of International Operations and Polymers. **Charles E. Wilks** has been named Executive Vice President. In this position, Mr. Wilks will direct activities for Amoco Chemicals Far East, Amoco Chemical Europe, Amoco Chemical Latin America, the Fine Acids business unit, and the Fiber and Film Intermediates business unit. Mr. Wilks, who joined the company in 1966, replaces **L. Richard Flury.**

**Joseph C. Drozd** has assumed the title and responsibilities of Technical Director for Calgene Chemical, Skokie, IL. In his new position, Dr. Drozd will oversee the quality control, quality assurance, product development, research, and technical service for the company.

ISK Biotech Corp., Mentor, OH, has named **Michel DeVleeschauwer** as Industrial Biocides Business Manager at ISK Biotech Europe Ltd. In this capacity, he will manage all Industrial Biocide activities in Europe, excluding regulatory and will continue to act as Administrative Manager of the Brussels, Belgium office.

**Robert E. Davis** has been appointed President and Chief Executive Officer of Rheometrics, Inc., Piscataway, NJ. Mr. Davis was previously President and Chief Operating Officer of both Sequa Corp. and Morton Thiokol.

**Joseph M. Starita,** outgoing President and Chief Executive Officer, will continue as Chairman and will assume the position of Vice President of Technology at Axess, the primary shareholder of Rheometrics. Dr. Starita will focus on the market development of process control and quality assurance systems.

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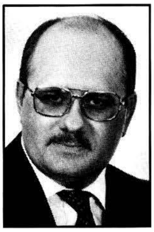
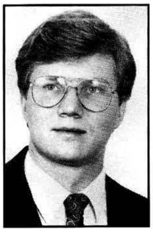
**Solution to October's *"CrossLinks"***



C.S. Phoenix



J.J. Kmetz

Lockhart Chemical Co., Pittsburgh, PA, has announced the addition of four research and development scientists and three marketing professionals. Joining the research and development staff are:

**Norman Ross,** Senior Chemist and Director of New Product Research and Development. Mr. Ross will develop new basic raw materials to achieve product performance improvements;

**Claude S. Phoenix,** Senior Coating Chemist, will specialize in environmental and workplace safety aspects of coating products;

**John J. Kmetz,** Senior Coating Chemist/Group Leader, will focus on the corrosion control and rheology characteristics of coating products; and

**Frank C. Ciccone,** Chemical Technician, will support Dr. Ross's work with various sulfonate compounds.

The new members of the sales and marketing staff are:

**Richard P. Whitehead,** Business Manager, LOCKSOL™ Technologies, will oversee the sales and marketing of the LOCKSOL brand of additives for lubricants;

**Paul A. Drzyzga, Jr.,** Business Manager, LOCKGUARD™ Technologies, will be responsible for sales and marketing of additives for coatings; and

**Hank Odenweller,** Market Development Consultant, is responsible for marketing products to petroleum companies and other users throughout the South and Southeast sections of the United States.

Both Mr. Phoenix and Mr. Kmetz are members of the Pittsburgh Society.

**David Lawson** has joined Cardinal Industrial Finishes, Los Angeles, CA, as its Technical Representative for the New England states. Mr. Lawson is based in Warren, PA.

## CLASSIFIED ADVERTISING

### Plant Manager/Supervisor

Plant Manager/Supervisor wanted. Excellent opportunity for new plant startup. Send resume to Box 120667, Arlington, TX 76012.

The National Decorating Products Association, St. Louis, MO, has announced the appointment of three new Vice Presidents at its headquarters:

**Kathy Lambla-Goodman** has been promoted to Vice President of Sales and Marketing. In her new capacity, she will lead a sales and marketing effort with the sales, marketing, and promotions teams. Ms. Lambla-Goodman previously served as the National Sales Manager for NDPA's trade magazine, *Decorating Retailer*.

The Vice President of Association/Industry Services is **Ron Robertson.** He will lead the membership, education, and show management teams to serve the independent retailers and their suppliers. Mr. Robertson most recently held the position of Membership/Industry Activities Director.

**Gary Spaeth** has been promoted to Vice President of Operations. He will head the accounting, information services, insurance, printing, and personnel departments. Mr. Spaeth was the former Chief Financial Officer.

**Corine Parigian** has joined the staff of K.J. Quinn and Co., Inc., Seabrook, NH, as Technical Manager for the Coatings and Adhesives Division. Ms. Parigian will direct the research and development programs for the company's line of waterbased, solvent based, and 100% solids polyurethane.

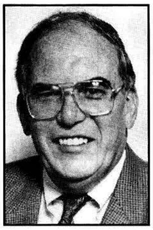
**Jeremy C. Croggon** has been named President of OMYA, Inc., Proctor, VT. Mr. Croggon assumes responsibility for the manufacturing, technical, marketing, and sales of the company's ground calcium carbonate production plants from the Proctor office. He was formerly President, Commercial, ECC International.

**Robin Wells** has joined The Mearl Corp., New York, NY, as a Color and Design Coordinator. In her new position, Ms. Wells will work with the design community and assist in the development of specialty color effects for packaging and other end product applications.

The Kelco Division of Merck and Co., Inc., San Diego, CA, has announced the following personnel changes:

**Jennifer Farnady**—Technical Service Representative, Algin Product Development; **Franne Ficara**—Division Counsel and Merck Legal Dept. member; **Alan King**—Manager, Gellan Gum Technical Service; **Martha Llaneras**—Technical Representative; **Howard Roberts**—Marketing Manager Textiles; **George Sanderson**—Senior Research Fellow; **Gail Trimble**—Project Manager, R&D Technical Service, Food Applications, R&D; and **Dana Zdanis**—Manager, Food Applications Development.

**John P. Lynch** has joined Daniel Products Co., Jersey City, NJ, as Vice President, Chief Financial Officer. Mr. Lynch's goal is to increase growth and adhere to the standards of quality for the company's products and customer service. Mr. Lynch is a former partner in Business Solutions International, Inc., his prior experience includes senior financial and operations management positions in both the chemical and financial services industries, encompassing acquisitions, financing, risk management, and strategic planning.



J.P. Lynch

# Obituary

**James W. Donnelly, Sr.,** Past-President of the Southern Society, died on July 6, 1993 in Atlanta, GA. He was 62.

Mr. Donnelly attended Auburn University and graduated from Georgia State University. He served in the U.S. Army from 1950 until 1952.

Mr. Donnelly's career in the coatings industry, which spanned 40 years, began with the Glidden Co. in 1953. He worked as Plant Manager and Plant Superintendent for 19 years. Following his years at Glidden, Mr. Donnelly worked at National Lead for six years as Salesman and Regional Sales Manager. At the time of his death, he had completed 13 years with Deeks and Co., Inc., where he worked as Sales Manager and Vice President.

Mr. Donnelly joined the Southern Society for Coatings Technology (SSCT) in 1964 where he immediately became active in various committee work. He was elected as an officer of the Society early in his career while working for the Glidden Co. and served as Secretary, Treasurer, Vice-President, and President-Elect. However, due to leaving Glidden and beginning work for a supplier, the rules of the Southern Society prevented Mr. Donnelly from serving as President. Nevertheless, he continued to be active as an associate member. Due to recent rule changes in the SSCT, Mr. Donnelly was elected to the Board of Directors and was elected as Society President for the 1990-91 year. Mr. Donnelly was an active member of the Atlanta Paint and Coatings Association, where he served as President.

Mr. Donnelly is survived by his wife, Marilyn; two sons, James, Jr. and David; one daughter, Carol; and a mother, brother, sister, and grandson.

## Organic Coatings Papers/Posters Solicited For 20th International Conference in Greece

The Organizing Committee of the Annual International Conference in Organic Coatings Science and Technology has issued a call for papers/posters. The conference will be held on July 4-8, 1994, Athens, Greece.

The meeting will feature the presentation of papers via the poster sessions. They will be introduced by rapporteurs who will highlight the major themes from the posters. A plenary discussion led by specialists in the subjects areas will follow the poster session. In addition, a limited number of invited and contributed oral papers will be delivered. Appropriate topics include, but are not limited to: polymers (waterborne, emulsion, powder, cure studies) rheology, testing, pigments, corrosion, and coatings technology, and films—formation, properties, surface characterization.

Potential authors are invited to submit titles and a one-page abstract by April 1, 1994. Additional information may be obtained from Angelos V. Patsis, Director, Institute for Materials Science, State University of New York, New Paltz, NY 12561.

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## "Advances in Emulsion Polymerization" Short Course Scheduled for June 6-10

The 25th annual one-week short course, "Advances in Emulsion Polymerization and Latex Technology," will be offered at Lehigh University, Bethlehem, PA, during the week of June 6-10, 1994.

The course, which is aimed at engineers, chemists, other scientists and managers who are involved in emulsion work and for those who wish to develop expertise in the area, provides an in-depth study of the synthesis and properties of high polymer latexes. The subject matter includes a balance of theory and applications as well as a balance between chemical and physical problems.

The course fee is $900 for the entire week or $300 per day for any part. Further information can be obtained from Dr. Mohamed S. El-Aasser, Emulsion Polymers Institute, Lehigh University, 111 Research Drive, Bethlehem, PA 18015.

## Pacific Northwest Society for Coatings Technology's 47th Annual Meeting and Symposium

### "PRACTICALITY EQUALS QUALITY AND EFFICIENCY"

### April 28-May 1, 1994
### Victoria, B.C., Canada

The 1994 Annual Meeting and Symposium of the Pacific Northwest Society is scheduled for April 28-May 1, 1994 at the Empress Hotel in Victoria, B.C., Canada.

The theme of this event is "Practicality Equals Quality and Efficiency," and the Technical Committee is seeking papers covering topics that address current and new technologies that facilitate manufacture, enhance performance and quality of coatings. The deadline for one-page abstracts is **December 15, 1993.**

Those interested in submitting a paper should contact: Dave Pasin, 12255 King George Hwy., Surrey, B.C., Canada V3V 3R9 or Yvon Poitras, 8380 River Rd., Delta, B.C., Canada V4G 1B5.

## Western Coatings Societies' 22nd Symposium and Show

### February 20-22, 1995
### San Francisco, CA

The 22nd Western Coatings Societies' Symposium and Show "New Opportunities—Challenges & Solutions," is scheduled for February 20-22, 1995, at the Hilton Hotel and Towers, San Francisco, CA. The Golden Gate Society for Coatings Technology is hosting this biennial event.

For more information on the 22nd Western Coatings Societies' Symposium and Show, contact: Gordon Pioch, c/o Eureka Chemical Co., P.O. Box 2205, S. San Francisco, CA 94083.

## Los Angeles Society Paint Technology Course Underway

The Los Angeles Society is sponsoring a course on Latex Architectural Finishes—Formulation and Raw Materials. This course, which is currently underway, covers latex paint formulation and raw materials. Some of the topics to be discussed include:

—History of paint;

—Paint mathematics;

—Paint personality;

—Formulation dynamics;

—Properties of latex emulsions and manufacturing;

—White hiding and extender pigments;

—Nine latex paint additives;

—Colored pigments—inorganic and organic;

—Latex wall sealers, wood undercoats, exterior wood primers, and block fillers; and

—Latex house paints, latex exterior stains, elastomeric roof coating, deck paints, latex semi-gloss enamels

For more information on the Paint Technology Course, contact Frank Peters, Dunn-Edwards Corp., 4885 E. 52nd Pl., Los Angeles, CA 90040.

# Book Review

## KIRK-OTHMER ENCYCLOPEDIA OF CHEMICAL TECHNOLOGY, FOURTH EDITION, VOLUME 6, $C_2$-CHLORO-CARBONS TO COMBUSTION TECHNOLOGY

Executive Editor: Jacqueline I. Kroschwitz
Editor: Mary Howe-Grant

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605 Third Ave.
New York, NY 10158-0012 (1993)

Reviewed by
Percy E. Pierce
Consultant
Monroeville, PA 15146

The multivolume *Kirk-Othmer Encyclopedia of Chemical Technology* has become a standard reference work in public, academic, and research and development libraries. It has undergone periodic revisions to update its contents and is now in its Fourth Edition. The recently revised Volume 6, *$C_2$-Chloro-Carbons to Combustion Technology* has been issued and carries on the tradition established by its predecessors.

*The Encyclopedia of Chemical Technology* articles are written by individual contributors with experience and knowledge in the topic area. The topic coverage is logically organized and gives an overview of important issues to the chemical technologist. The topics are well referenced, providing a direction for those who desire to delve more deeply into the topic. The references include patents as well as books, monographs, and journal articles, a feature that is especially useful to the industrial researcher. Cross references to material of interest in other volumes are provided throughout the text.

Volume 6 is rich in topics and information of interest to the coatings technologist. The articles on "Coated Fabrics," "Coatings," and "Coatings, Marine" are obvious subjects of interest to JCT readers.

The article on "Coated Fabrics" discusses the textile component, polymeric coating component, processing, economic aspects, testing, and health and safety factors of coated fabrics.

The articles on "Coating Processes" are arranged in three sections. The "Coating Processes (Survey)" section covers knife and blade coating, wire-wound rod coating, roll coating, gravure coating, dip coating, extrusion and slot coating, slide coating, curtain coating, dip and spin coating, and vacuum deposition techniques. Air impingement dryers and contact dryers are also discussed. Modelling methods for simulation of coating and drying processes are mentioned. The section on "Coating Processes (Powder Technology)" covers thermoplastic coating powders, thermosetting coating powders, manufacture, application methods, economic aspects, test methods, environmental and energy considerations, and health and safety factors. This article has an extensive bibliography that covers patents, monographs, and research articles on powder coatings. The section on "Coating Processes (Spray)" discusses airless atomization, supercritical atomization, air atomization, electrostatic atomization, rotary atomization, electrostatic spraying, and economic aspects.

The article on "Coatings" written by Zeno W. Wicks, Jr. provides a comprehensive overview of the field of organic coatings. This article, especially recommended to JCT readers, covers film formation, resins, volatile components, pigments, flow, film properties, adhesion, exterior durability, corrosion control, mechanical properties, architectural coatings, product coatings, special purpose coatings, and economic aspects. It contains a balanced blend of coating fundamentals and practical insights on formulation and raw material selection. End use requirements for architectural, industrial, automotive, wood product, autorefinish, etc., coatings are presented with an intelligent discussion of the issues that influence the selection of application methods, resins, pigments, solvents, and additives to satisfy customer requirements. The article is extensively referenced to primary and secondary sources of specialized coating information, including the *FSCT Series* on coatings.

The article on "Coatings, Marine" written by Robert F. Brady, Jr. and Richard W. Drisko gives a detailed discussion of marine coatings with emphasis on corrosion protection, environmental concerns, surface preparation, marine coating types, application methods, and coating selection for various types of marine structures. The article references include military specifications.

A good general introduction to color principles is provided in the article on "Color." An excellent overview is given of color fundamentals, color vision, color order systems, colorimetry, and color measuring instruments. A distinctive feature of this article is its discussion of the fifteen causes of color. The author in a clear and concise way explains the physical mechanisms of color formation in gases, solids, and liquids. The discussion ranges from the color of incandescent lighting to the changing color of liquid crystals through the red of ruby and the green of emerald. The article is well referenced to books and papers that are considered standard sources by color specialists who deal with coatings applications.

The article on "Colloids" is mainly of academic interest and does not deal specifically with the many practical applications of colloid science to coatings or chemical technology. The article has a comprehensive bibliography of the significant articles and monographs on colloid science. It is worthwhile reading for the individual with an interest in the foundations of colloid science and the current directions of fundamental work in this important area.

The articles on "Chlorocarbons and Chlorohydrocarbons," "Chromium Compounds," "Clays," and "Cobalt Compounds" besides presenting a balanced overview of their respective topics contain useful information on pigments, driers, and solvents used in the coatings industry.

The articles on "Colorants for Ceramics," "Colorants for Food, Drugs, Cosmetics, and Medical Devices," and "Colorants for Plastics" contain useful information on pigments and colorants. However, the pigments used in organic coatings receive better coverage in other volumes of the *Kirk-Othmer Encyclopedia.* The "Colorants for Food, Drugs, Cosmetics, and Medical Devices" article is noteworthy because it covers regulatory issues that protect the health and safety of the public.

The articles on "Color Photography" and "Color Photography, Instant" provide an interesting and colorful overview of a complex technology. These products based on multilayer photosensitive coatings show that coating science is not restricted to the products of the coating industry most familiar to JCT readers. The article on "Color Photography" has clear discussions of color vision, additive color mixing, subtractive color mixing, and three-color photography.

The section on "Chromogenic Materials" is a source of new opportunities for coatings applications utilizing color changes induced by the effect of electricity, stress, light, and heat on films. Many of the systems and materials discussed have found

*(Continued on page 80.)*

## Surfactants Guide

A 40-page guide to surfactants and their role in improving the manufacture, stabilization, and formulation of latex polymers has been published. In addition to summary descriptions of several performance chemicals products, the four-color publication employs a wide array of graphs and charts and written explanations to illustrate test results and the impact of surfactants on a variety of the final latex properties. For a free copy of "Products for the Manufacture, Stabilization, and Formulation of Latex Polymers," write BASF Corp./Performance Chemicals, 100 Cherry Hill Rd., Parsippany, NJ 07054.

**Circle No. 200 on Reader Service Card**

## Waterborne Acrylic

A new waterborne acrylic emulsion, offering fast developing print resistance in industrial wood finishing applications, is the topic of a product release. This product is designed for high volume applications involving wood furniture, kitchen cabinets, and other wood components, where premium print resistance, clarity, gloss, chemical, and stain are critical. For more information or samples of NeoCryl XA-6051, contact ZENECA Resins, Industrial Coatings, 730 Main St., Wilmington, MA 01887.

**Circle No. 201 on Reader Service Card**

## Dispersion Mill

Information on a high speed rotor-stator dispersion mill is now available. This product can be utilized as a small batch mill for the large factory or a production mill for the smaller plant. To obtain more information on the Kady Model OC, contact Kinetic Dispersion Corp., 127 Pleasant Hill Rd., P.O. Box 847, Scarborough, ME 04070-0847.

**Circle No. 202 on Reader Service Card**

## Heat Seal Coatings

A product release introduces polyolefin polymers supplied in aliphatic solvent that offers fast rates of solvent loss and a reduction in volatile aromatics. This product is designed to metal foil lamination and can be used as an adhesion improver for paints, inks, and adhesives. Contact Ron McGregor, Siber Hegner Group, County House, 221-241 Beckenham Rd., Beckenham, Kent, BR3 4UF, UK.

**Circle No. 203 on Reader Service Card**

## Bulk Container

A new 330-gallon intermediate bulk container designed for the shipment of liquid products such as foods, paper treatments, water-based adhesives, and paint additives is introduced through literature. To obtain more information on the Sonobulk® 330, contact Ron Rakey, Sonoco Products Co., Intermediate Bulk Container, 100 Alex St., Lavonia, GA 30553.

**Circle No. 204 on Reader Service Card**

## Biocide Line Guide

A guide to assist the formulator of paint and coatings systems with the selection and proper use of a company's products is available. Products covered include those used in solvent borne stains and coatings, low solids aqueous systems, latex coatings, high-solid alkyd paints and primers, latex emulsions, metal-working fluids, textile processing solutions, and aqueous or solvent borne systems. To receive a copy of "Tips and Techniques," which offers selection of the Troysan® Polyphase® products, contact Troy Corp., P.O. Box 366, 72 Eagle Rock Ave., East Hanover, NJ 07936-0366.

**Circle No. 205 on Reader Service Card**

## Powder Wetting

Literature highlights a mixer developed for use in applications where a vortex is required to rapidly wet-out and disperse large volumes of powders into liquids. The product is suited for rapid dispersion of large volumes of powder or absorption of materials that are difficult to wet out and that have a tendency to float or raft. Additional information on the V Range mixer may be obtained from Silverson Machines, Inc., P.O. Box 589, East Longmeadow, MA 01028.

**Circle No. 206 on Reader Service Card**

## Additives, Bases, and Coatings

A line of functional additives, formulating bases, and semi-finished coatings, that feature environmentally safe water-based products are the topic of a product release. They are designed for environmental protection and provide worker safety in manufacturing, shipping, and use. For more information on the LOCKGUARD™ product line, write James A. Giammarco, Vice President, Marketing, Lockhart Chemical Co., 2873 West Hardies Run Rd., Gibsonia, PA 15044.

**Circle No. 207 on Reader Service Card**

## Cost Estimator

A slide chart for estimating and comparing costs of liquid finishing and powder coatings is available. One side of the chart allows the user to estimate the applied cost of liquid finishing and the other side of the chart provides the corresponding variables for estimating the applied coat of a powder coated finish. To receive the slide chart, write Jeff Palmer, The Powder Coating Institute, 1800 Diagonal Rd., Suite 370, Alexandria, VA 22314.

**Circle No. 207 on Reader Service Card**

## Rheological Tests

A vapor barrier system that enables creep, elastic recovery, and dynamic mechanical tests to be run on fast drying liquid coatings is the topic of a product release. The new system was designed to prevent evaporation and drying by creating a vapor barrier that controls the sample environment. For more information on the new vapor barrier system for the Stress Rheometer, contact Don Becker, Product Manager, Rheometrics, Inc., One Possumtown Rd., Piscataway, NJ 08854.

**Circle No. 208 on Reader Service Card**

## Atomic Spectroscopy Supplies

A 68-page catalog provides ordering information for a company's atomic absorption, inductively coupled plasma-optical emission spectroscopy, and ICP-mass spectrometry instrumentation. The catalog also highlights a line of high-quality standards prepared from high-purity metals and salts, with full certification and includes pricing and overnight ordering information. To order the 1993-1994 Perkin-Elmer Atomic Spectroscopy Supplies Catalog, write PE XPRESS, 761 Main Ave., Norwalk, CT 06859-0012.

**Circle No. 209 on Reader Service Card**

## Emulsion Defoamer

A new silicone emulsion defoamer for paint and coatings, lacquer, ink, and special purpose applications has been introduced in a product release. This product was designed to enhance handling and storage capabilities, as well as system compatibility. To request DREWPLUS L-410 defoamer literature and a sample, contact Marketing Services, Drew Industrial Division, One Drew Plaza, Boonton, NJ 07005.

**Circle No. 210 on Reader Service Card**

### Microscopy Attachment

A new slide-on attenuated total reflectance attachment is introduced through literature. This attachment reportedly allows for a new range of surface analyses, eliminates scatter losses, and the Contact Alert accessory provides an audible sound and visual indication that contact has been established between the objective and the sample. For more information on the Slide-On ATR Attachment, contact Debbie Esposito, Spectra-Tech, Inc., 652 Glenbrook Rd., Bldg. 8, P.O. Box 2190-G, Stamford, CT 06906.

**Circle No. 211 on Reader Service Card**

### Fragmentation Accessory

A new accessory that provides fragmentation and structural information directly from a mass spectrometer for details on structure, sequence, and post-translational modifications is highlighted in a product release. Customer trials reportedly demonstrated successful fragmentation of glycopeptides and peptides up to 10 kDalton. For more information on FAST™, write Bruker Instruments, Inc., Manning Park, Billerica, MA 01821.

**Circle No. 212 on Reader Service Card**

### Pigment Grind Aids

A data sheet discussing a company's solvent-free, low foaming pigment grind aids designed for use in aqueous systems is available. These surfactants are utilized in formulations and reportedly include greater color development for maximum tint strength, reduced grind time, and dispersion stabilization. For a copy of "Surfynol® Surfactants for Pigment Grinding," contact Air Products and Chemicals, Inc., Performance Chemicals Division, 7201 Hamilton Blvd., Allentown, PA 18195-1501.

**Circle No. 213 on Reader Service Card**

### Capillary Rheometer

A rheometer designed to permit the use of capillaries, slits, and dies to provide rheological information is introduced through literature. This device describes the behavior of materials under production, processing, and application conditions where extremely high shear rates, up to $10^6$ $sec^{-1}$, often occur. This product reportedly determines the viscosity of materials by identifying the time that a certain amount of sample, under a preset pressure condition, needs to flow through a capillary, slit, or die of known dimensions. To obtain more information on the HVA6 Rheometer, contact Paar Physica USA, Inc., 1090 King Georges Post Rd., Ste. 607, Edison, NJ 08837.

**Circle No. 214 on Reader Service Card**

### Viscosity and pH Control

Technical information has been released on a portable viscometer that measures and controls viscosity and pH of water-based fluids in process applications. The two-piece instrument consists of the viscometer probe and detachable second probe with a temperature sensor and sealed pH sensor. To learn more about TT220 viscosity and pH control system, contact Brookfield Engineering Laboratories, Inc., Dept. NR-99, 240 Cushing St., Stoughton, MA 02072.

**Circle No. 215 on Reader Service Card**

### Colorimeter Software

Literature describes a software package that has been developed for use with an online colorimeter. The software is used to analyze in-process color control of paper, plastics, textiles, and foods and offers on-the-spot color evaluation and pass/fail capabilities. Additional information on the SpectraQC software package may be obtained from Minolta Corp., Instrument Systems Div., 101 Williams Dr., Ramsey, NJ 07446-1293.

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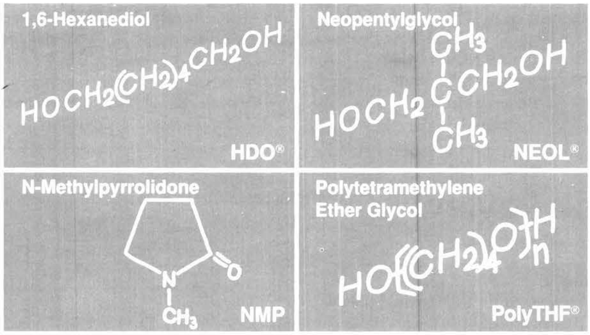

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### Experimental Design

The Design of Experiments (DOE) methodology, which allows engineers and scientists to evaluate many variables simultaneously, is clarified in a new booklet. This publication provides answers to the 12 most commonly asked questions about DOE and covers topics like when and how to use DOE, its link with quality, and the role of the computer and DOE software. For a complimentary copy of this publication, contact DuPont Quality Management & Technology Center, Louviers Bldg., Room 33W53, P.O. Box 6090, Newark, DE 19714-6090.

**Circle No. 217 on Reader Service Card**

### Lid Placer

Information on a lid placer for placing up to 120 friction type plugs per minute is now available. A screw feed separates and continually moves stored lids into position, as one is being placed. The basic unit is set up for single size from 1/2 pint to gallons and change kits are available to accommodate additional sizes. To receive information on the High Speed Lid Placer, contact Ideal Manufacturing & Sales Corp., 1118 O'Neill Ave., Madison, WI 53704.

**Circle No. 218 on Reader Service Card**

### Impeller Selection

A brochure illustrating how a range of impellers provides a variety of flow and shear characteristics has been published. Each of the major impellers is pictured, along with a brief description of its function and appropriate applications. For a free copy of "Matching the Impeller to Your Process," contact LIGHTNIN, 135 Mt. Read Blvd., Rochester, NY 14603.

**Circle No. 219 on Reader Service Card**

### Ink Jet System

A small character ink jet system that allows users to automate their on-line product marking process has been introduced in a product release. This product reportedly utilizes proven and affordable drop-on-demand printhead technology by delivering a highly legible mark on most surfaces at speeds of up to 200 feet per minute and can print four message lines of 1/16 in., 3/32 in., or 1/8 in. alphanumerics, or special characters. Additional information on the PES Series Small Character Ink Jet System may be obtained from Diagraph Corp., 3401 Rider Trail South, St. Louis/Earth City, MO 63045-1110.

**Circle No. 220 on Reader Service Card**

### Interferometry Software

Literature describes analytical, control, and human interface software used to drive a family of noncontact interferometers that has extended its use to encompass a 3-D imaging surface structure analyzer. This software is windows based and features a simple point-and-click graphic user interface and includes interactive control of the interferometer, data processing features, sample logging formats, and Statistical Process Control capabilities. Additional information on MetroPro™, write Zygo Corp., Laurel Brook Rd., P.O. Box 448, Middlefield, CT 06455-0448.

**Circle No. 221 on Reader Service Card**

### Spray Booth

A spray booth designed to reclaim high-solids thermosetting and UV curable coatings is the topic of a data sheet. The system reportedly provides the capability to capture light colors on one set of collectors and dark colors on another which allows the material to be reformulated and color adapted for various color. Contact JBI, Inc., Spray Booths and Systems, P.O. Box 38, 801 Norway Rd., Osseo, WI 54758.

**Circle No. 222 on Reader Service Card**

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## Book Review

*(Continued from page 77)*

application in products ranging from electrical displays to self-darkening plastic lenses.

Because of the interdisciplinary nature of the organic coatings field, information of interest to the coating technologist can be found under a variety of topic headings some of which might be overlooked by even the most careful and experienced reader. The information in the article on "Coffee" will enhance each JCT reader's appreciation of this most essential substance consumed in large quantities in the coatings industry during meetings and coffee breaks.

This reviewer regrets that he did not have the opportunity to review the CD-ROM version of the *Kirk-Othmer Encyclopedia of Chemical Technology*. CD-ROM technology would facilitate the searching of the rich mine of technical information that abounds in this excellent work for the chemical technologist. I recommend the revised *Kirk-Othmer Encyclopedia* to JCT readers as a general source of up-to-date useful information on chemical technology suitable for the needs of the coating chemist. Judging from the recently issued Volume 6, the fourth revised edition is a worthy update and has a place in the reference section of every library.

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## Letter to the Editor

To the Editor

I read with great interest the Letter to the Editor by Milton Glaser regarding a languishing technique of using amino alcohols in the preparation of alkyds.

In the late sixties, I had prepared a number of oxazoline derivatives for making water soluble polymers, oxazoline modified polyesters (U.S. 3, 438, 943), UV stabilizers and alkyd intermediates. In one of our studies, we prepared a special alkyd using (tris)hydroxymethylaminomethane as well as 2-amino-2-methyl propanediol. These alkyds had very good corrosion resistance. In fact, we were able to produce a product BLOXRUST®, a rust resistant primer marketed by the O'Brien Corporation, (now Fuller-O'Brien Corp.)

Mr. Glaser is certainly correct in bringing this to the attention of our readers. I trust that this may spark the imagination of those synthetic polymer chemists out there to reevaluate the role of these very functional aminoalcohols.

Here is another proof that history repeats itself, especially in the coatings industry.

Dr. Thomas J. Miranda
Adjunct Professor
Indiana University—South Bend

# Coming Events

## FEDERATION MEETINGS

*For information on FSCT meetings, contact Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422 (215) 940-0777, FAX: (215) 940-0292.*

### 1994

(May 12-15)—Federation "Spring Week." Spring Seminar on the 12th and 13th; Incoming Society Officers Meeting on the 14th; Board of Directors Meeting on the 15th. Marriott City Center Hotel, Minneapolis, MN.

(Oct. 12-14)—72nd Annual Meeting and 59th Paint Industries' Show. New Orleans Convention Center, New Orleans, LA.

### 1995

(Oct. 9-11)—73rd Annual Meeting and 60th Paint Industries' Show. Cervantes Convention Center, St. Louis, MO.

### 1996

(Oct. 23-25)—74th Annual Meeting and 61st Paint Industries' Show. McCormick Place North, Chicago, IL.

## SPECIAL SOCIETY MEETINGS

### 1994

(Feb. 9-11)—21st Annual Waterborne, Higher-Solids, and Powder Coatings Symposium. Sponsored by the Southern Society for Coatings Technology and The University of Southern Mississippi (USM). New Orleans, LA. (Robson F. Storey or Shelby Thames, Co-Organizers, WBHS&PC Symposium, Dept. of Polymer Science, USM, P.O. Box 10076, Hattiesburg, MS 39406-0076).

(Mar. 3-4)—Southwestern Paint Convention. Sponsored by Houston and Dallas Societies. South Shore Harbour Resort & Conference Center, League City (Houston), TX. (Joseph Caravello, Guardsman Products, Inc., 137 N. Hall Dr., Sugarland, TX 77478; (713) 242-9473).

(Apr. 27-30)—Pacific Northwest Society. 47th Annual Spring Symposium. The Empress Hotel, Victoria, B.C., Canada. (Ed Linton, Cloverdale Paint Inc., 6950 King George Hwy., Surrey, B.C. V3W 4Z1, Canada; (604) 596-6261).

## OTHER ORGANIZATIONS

### 1993

### *North America*

(Nov. 15-16)—"Fundamentals of HPLC." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education—Chemistry, KSU, P.O. Box 5190, Kent, OH 44242-0001).

(Nov. 16-18)—"Liquid and Powder Coatings Operations: A Comprehensive Seminar on Finishing Processes with a Special Emphasis on Pollution Prevention." Sponsored by University of Berkeley Extension. University of New Orleans, New Orleans, LA. (Continuing Education in Engineering, University Extension, University of California, 2223 Fulton St., Berkeley, CA 94720).

(Nov. 18-19)—"Rheological Short Course on Applications, Theory, and Instrumentation," sponsored by Paar Physica USA. Houston, TX. (Lisa Covell, Paar Physica USA, 1090 King Georges Post Rd., Ste. 607, Edison, NJ 08837).

(Nov. 19-21)—46th Annual National Decorating Products Show. Sponsored by National Decorating Products Association (NDPA). Indiana Convention Center, Indianapolis, IN. (Ruth Wilms, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Nov. 30-Dec. 3)—"Compliance with Air Quality Regulations for Paint, Coatings, and Printing Facilities with Special Sessions on Title V Permitting and Title III Air Toxics." Sponsored by University of Berkeley Extension. University of New Orleans, New Orleans, LA. (Continuing Education in Engineering, University Extension, University of California, 2223 Fulton St., Berkeley, CA 94720).

(Dec. 1-2)—"Statistical Process Control for Liquid and Powder Coating." Symposium sponsored by Society of Manufacturing Engineers (SME). Cincinnati, OH. (Barbara Hamlin, SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121-0930).

(Dec. 7-8)—"Finishing Automotive Plastics." Symposium sponsored by Society of Manufacturing Engineers (SME). Dearborn, MI. (Barbara Hamlin, SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121-0930).

(Dec. 7-8)—"Design of Experiments for Paint & Coatings." Symposium sponsored by the Society of Manufacturing Engineers (SME). Hyatt Regency O'Hare, Rosemont (Chicago), IL. (Susam Mahalik, SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121-0930).

### *Europe*

(Nov. 15-17)—"Powder Coatings." Paint Research Association's (PRA) 13th International Conference. Brussels. (Conference Secretary, PRA, 8 Waldegrave Rd., Teddington, Middlesex TW11 8LD, United Kingdom).

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(Nov. 23-25)—Conchem '93. International Exhibition and Conference sponsored by Reed Exhibition Companies. KKA Congress and Exhibition Centre, Karlsruhe, Germany. (Diane R. Tiberio, Reed Exhibition Companies, 999 Summer St., P.O. Box 3833, Stamford, CT 06905-0833).

(Dec. 6-8)—"Styrenics '93." MBS Conference sponsored by Maack Business Services. Swissôtel Zürich, Switzerland. (Maack Business Services, Moosacherstrasse 14, CH-8804, AU/ Zürich, Switzerland).

### Asia

(Dec. 16-18)—"Paintindia '93." Nehru Centre, Bombay, India. (Pankaj D. Dutia, Colour Publications Pvt. Ltd., 126-A, Dhuruwadi, A. V. Nagwekar Marg, Prabhadevi, Bombay 400 025, India).

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### 1994

### North America

(Jan. 15-18)—RCMA 1994 Annual Conference & EXPO. Sponsored by the Roof Coatings Manufacturers Association (RCMA). Le Meridien at San Diego, Coronado, CA. (Sally Choquette, RCMA Meetings Coordinator, RCMA, 6000 Executive Blvd., Ste. 201, Rockville, MD 20852-3803).

(Jan. 22-25)—6th Sanibel Conference on Mass Spectrometry.Sponsored by American Society for Mass Spectrometry (ASMS) and ACS Division of Polymer Chemistry. Sundial Beach & Tennis Resort, Sanibel, FL. (ASMS, 815 Don Gaspar, Santa Fe, NM 87501).

(Feb. 11-13)—37th Annual Southern Decorating Products Show. Sponsored by National Decorating Products Association (NDPA). Georgia International Convention & Trade Center, College Park (Atlanta), GA. (Ruth Wilms, NPDA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Feb. 18-19)—110th PDCA Paint & Paper Pro Show. Sponsored by the Painting and Decorating Contractors of America (PDCA). Anaheim, CA. (Mary DePersig, Trade Show Manager, PDCA, 3913 Old Lee Hwy., Ste. 33-B, Fairfax, VA 22030).

(Feb. 20-24)—Second North American Sympopsium on Recent Advances in Polymer Blends and Alloys. Sponsored by the American Chemical Society, PMSE Division. Hilton Head, SC. (A.V. Patsis, State University of New York, New Paltz, NY 12561).

(Feb. 21-23)—Annual Meeting of The Adhesion Society and "Fundamentals of Particle Adhesion," symposium.Clarion Plaza Hotel, Orlando, FL. (Prof. J.G. Dillard, Dept. of Chemistry, Virginia Polytechnic Institute and State Unviersity, College of Arts and Sciences, Blacksburg, VA 24061-0212).

(Mar. 5-6)—38th Annual Canadian Decorating Products Show. Sponsored by National Decorating Products Assoication (NDPA). International Centre, Toronto, Ontario, Canada. (Ruth Wilms, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Mar. 13-18)—"High Solids Coatings" Symposium. Sponored by American Chemical Society Division of Polymeric Materials: Science and Engineering. San Diego, CA. (Frank N. Jones, Coatings Research Institute, Eastern Michigan University, 430 W. Forest St., Ypsilanti, MI 48197 or George R. Pilcher, Akzo Coatings, Inc., P.O. Box 147, Columbus, OH 43216-0147).

(Mar. 14-16)—Seventh Annual Conference on Industrial Lead Paint Abatement and Removal. Sponsored by Steel Structures Painting Council (SSPC). Hyatt Regency, Greenwich, CT. (SSPC, 4516 Henry St., Ste. 301, Pittsburgh, PA 15213-3728).

(Mar. 23-25)—Electrocoat '94. Sponsored by *Products Finishing Magazine*. Hyatt Orlando, Orlando, FL. (Cindy Goodridge, Gardner Management Services, 6600 Clough Pike, Cincinnati, OH 45244).

(Mar. 28-Apr. 1)—"Basic Composition of Coatings." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(Apr. 17-19)—"Volatile Organic Compounds (VOCs) in the Environment" Symposium. Sponsored by ASTM Committee E-47. Montreal, Que., Canada. (Wuncheng Wang, Symposium Chairman, U.S. Geological Survey, WRD, P.O. Box 1230, Iowa City, IA 52244).

(Apr. 18-22)—"Introduction to Paint Formulation." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(Apr. 20-27)—Surface Treatment '94. Hannover Fairgrounds, Hannover, Germany. (Hannover Fairs USA Inc., 103 Carnegie Center, Princeton, NJ 08540).

(Apr. 23-24)—Eastern Decorating Products Show. Sponsored by National Decorating Products Association (NDPA). Garden State Convention & Exhibit Center, Somerset, NJ. (Ruth Wilms, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(May 1-5)—RadTech '94 North America. Conference sponsored by RadTech International North America. Walt Disney World Dolphin Hotel, Orlando, FL. (Chris Dionne, RadTech International North America, 60 Revere Dr., Suite 500, Northbrook, IL 60062).

(May 2-4)—"Fundamentals of Adhesion: Theory, Practice, and Applications." Lake Mohonk, New Paltz, NY. (A.V. Patsis , State University of New York, New Paltz, NY 12561).

(May 2-4)—"Polymer Blends & Alloys: Phase Behavior, Characterization, Morphology, Alloying Technology." Lake Mohonk, New Paltz, NY. (A.V. Patsis , State University of New York, New Paltz, NY 12561).

(May 2-4)—"Basic Coatings for Sales and Marketing Personnel." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(May 2-5)—"Science & Technology of Pigment Dispersion." Lake Mohonk, New Paltz, NY. (A.V. Patsis , State University of New York, New Paltz, NY 12561).

(May 4-5)—"Paint Volatile Organic Compounds (VOC)." Course sponsored by American Society for Testing and Materials (ASTM). Chicago, IL. (Tina Falkenstein, ASTM, 1916 Race St., Philadelphia, PA 19103).

(May 8-13)—37th Annual Technical Conference of the Society of Vacuum Coaters (SVC). Westin Hotel/Copley Place, Boston, MA. (SVC, 440 Live Oak Loop, Albuquerque, NM 87122-1407).

(May 16-20)—"Introduction to Paint Formulation." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(June 6-10)—"Advances in Emulsion Polymerization and Latex Technology." Short course sponsored by Lehigh University, Bethlem,

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PA. (Mohamed S. El-Aasser, Emulsion Polymers Institute, Lehigh University, 111 Research Dr., Bethlehem, PA 18015).

(July 11-15)—MACROAKRON '94 International Symposium. Sponsored by the International Union of Pure and Applied Chemistry. University of Akron, Akron, OH. (Dr. Joseph P. Kennedy, Chairman of the Organizing Committee, or Cathy Manus-Gray, Symposium Coordinator, College of Polymer Science and Polymer Engineering, The University of Akron, Akron, OH 44325-0604).

(July 18-20)—"Basic Coatings for Sales and Marketing Personnel." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(Nov. 13-17)—Third North Ameican Research Conference on Organic Coatings Science and Technology. Sponsored by the American Chemical Society, PMSE Division. Hilton Head, SC. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

## Australia

(Apr. 5-8)—1994 Australian Master Painters Convention. Sponsored by The Federation of Master Painters & Signwriters of Australia. Sydney Convention & Exhibition Centre, Darling Harbour. (Total Concept Conference, P.O. Box 1221, Crows Nest, NSW, 2065).

## Asia

(May 18-19)—Fourth Asia/Pacific Coatings Show. Organized by the Paint Research Association. Hong Kong Convention and Exhibition Center, Hong Kong, China. (Jane Malcolm-Coe, FMJ International Publications Ltd., Queensway House, 2 Queensway, Redhill, Surrey, RH1 1QS, England).

## Europe

(Mar. 14-18)—"Recycle '94." Forum and Exposition. Sponsored by Maack Business Services. Congress Centre, Davos, Switzerland. (Maack Business Services, Moosacherstrasse 14, CH-8804 AU/Zürich, Switzerland).

(June 5-9)—XXIInd FATIPEC Congress and Exhibition. Budapest Convention Centre, Budapest, Hungary.

(June 13-16)—"Science and Technology of Pigment Dispersion." Luzern, Switzerland. (A.V. Patsis, State University of New York, New Paltz, NY 12561).

(June 14-17)—Workshop on "Polymer Blends and Alloys." Luzern, Switzerland. (A.V. Patsis, State University of New York, New Paltz, NY 12561).

(June 20-22)—16th International Conference on Advances in the Stabilization and Controlled Degradation of Polymers. Luzern, Switzerland. (A.V. Patsis, State University of New York, New Paltz, NY 12561).

(June 21-22)—Surfex '94. Exhibition organized by Surfex Ltd., a wholly owned subsidiary of the Oil & Colour Chemists' Association. Harrogate, North Yorkshire. (H. Pooley, Surfex Ltd., Priory House, 967 Harrow Rd., Wembley HAO 2SF, England).

(July 4-8)—20th International Conference on Organic Coatings Science & Technology. Athens, Greece. (A.V. Patsis, State University of New York, New Paltz, NY 12561).

(Sept. 28-30)—"Fluorine in Coatings." Conference organized by the Paint Research Association in conjunction with Chemical-Polymer and Chemserve (UMIST). Salford, England. (Conference Secretary, Paint Research Association, 8 Waldegrave Rd., Teddington, Middlesex TW11 8LD, UK).

## Pacific Rim

(Apr. 14-16)—Paint Show '94. Sponsored by Japan Paint Manufacturers Association, Japan Paint Commerce Association, Japan Painting Contractors Association, and Nippon Toryo (Paint) Club. Nippon Convention Center, Tokyo, Japan. (Paint Show '94 Executive Committee, Overseas Operation, c/o Space Media Japan Co., Ltd., Asahi Bldg. 4F, 2-31-3 Taito, Taito-ku, Tokyo 110 Japan).

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# 'Humbug' from Hillman

A recent issue of the *New York Times* contained a column title "A Way With Words," compiled by Jim Koch, in which he featured the inimitable Mae West "as a writer of her own material, as well celebrated for her lines as her curves."

—"A man has more character in his face at 40 than at 20—he has suffered longer."

—"I like a man that's good, but not too good. For the good die young, and I hate a dead one."

—"When I'm good I'm very good but when I'm bad, I'm better."

—"Men like a woman with a past—because they hope history will repeat itself."

—"It's not the men in my life that count—it's the life in my men."

—"It's better to be looked over than overlooked."

—"Give a man a free hand, and he'll try to put it all over you."

—"Between two evils I always pick the one I haven't tried before."

—"I wrote the story myself. It's all about a girl who lost her reputation and never missed it."

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I was pleased to get a note from an old and very good friend, Moe Bauman, which included an article from the *Salt Lake Tribune*. It obviously is NOT typical Humbug fare but I was so impressed with its message that I thought it was well worth sharing with you. It begins with an Editor's note:

"In 1854, President Franklin Pierce made an offer to buy Indian tribal lands in the Northwest. We are printing, in part, the reply of Chief Seattle, leader of the combined Duwanism and Suquamish tribes. It is widely considered the most moving and profound statement of the environment ever made."

By Chief Seattle (1854):

How can you buy or sell the sky, the warmth of the land? The idea is strange to us.

If we do not own the freshness of the air and the sparkle of the water, how can you buy them?

Every part of this earth is sacred to my people. Every shining pine needle, every sand shore, every mist in the dark woods, every clearing and humming insect is holy in the memory and experience of my people.

We will consider your offer to buy our land. But it will not be easy. This shining water that moves in the streams and rivers is not just water but the blood of our ancestors. The water's murmur is the voice of my father's father. The rivers are our brothers, they quench our thirst. The rivers carry our canoes and feed our children.

If we decide to accept. I will make one condition . . . the white man must treat the beasts of this land as his brothers.

I have seen a thousand rotting buffalo on the prairie, left by the white man who shot them from a passing train. I am a savage and I do not understand how the smoking Iron Horse can be more important than the buffalo that we kill only to stay alive.

This we know . . . the earth does not belong to man. Man belongs to the earth. All things are connected like the blood which unites one family.

Even the white man, whose God walks and talks with him as friend to friend, cannot be exempt from the common destiny. We may be brothers after all. We shall see. One thing we know, which the white man may one day discover . . . our God is the same God. He is the God of man and his compassion is equal for the red man and the white. This earth is precious to Him, and to harm the earth is to heap contempt on its creator.

We know that the white man does not understand our ways. One portion of the land is the same to him as the next; for he is a stranger who comes in the night and takes from the land whatever he needs.

He treats his mother, the earth, and his brother, the sky, as things to be bought, plundered, sold like sheep or bright beads.

There is no quiet place in the white man's cities . . . And what is there to life if a man cannot hear the lonely cry of the whippoorwill or the arguments of the frogs around a pond at night? . . . The Indian prefers the soft sound of the wind darting over the face of a pond, and the smell of the wind itself, cleansed by a mid-day rain or scented with pinon pine.

The whites, too shall pass; perhaps sooner than all other tribes. Contaminate your bed and you will one night suffocate in your own waste.

But in your perishing you will shine brightly, fired by the strength of the God who brought you this land and, for some special purpose, gave you dominion over this land and the red man.

That destiny is a mystery to us, for we do not understand when the buffalo are all slaughtered, the wild horses are tamed, the secret corners of the forest heavy with the scent of many men, and the view of the ripe hills blotted by talking wires.

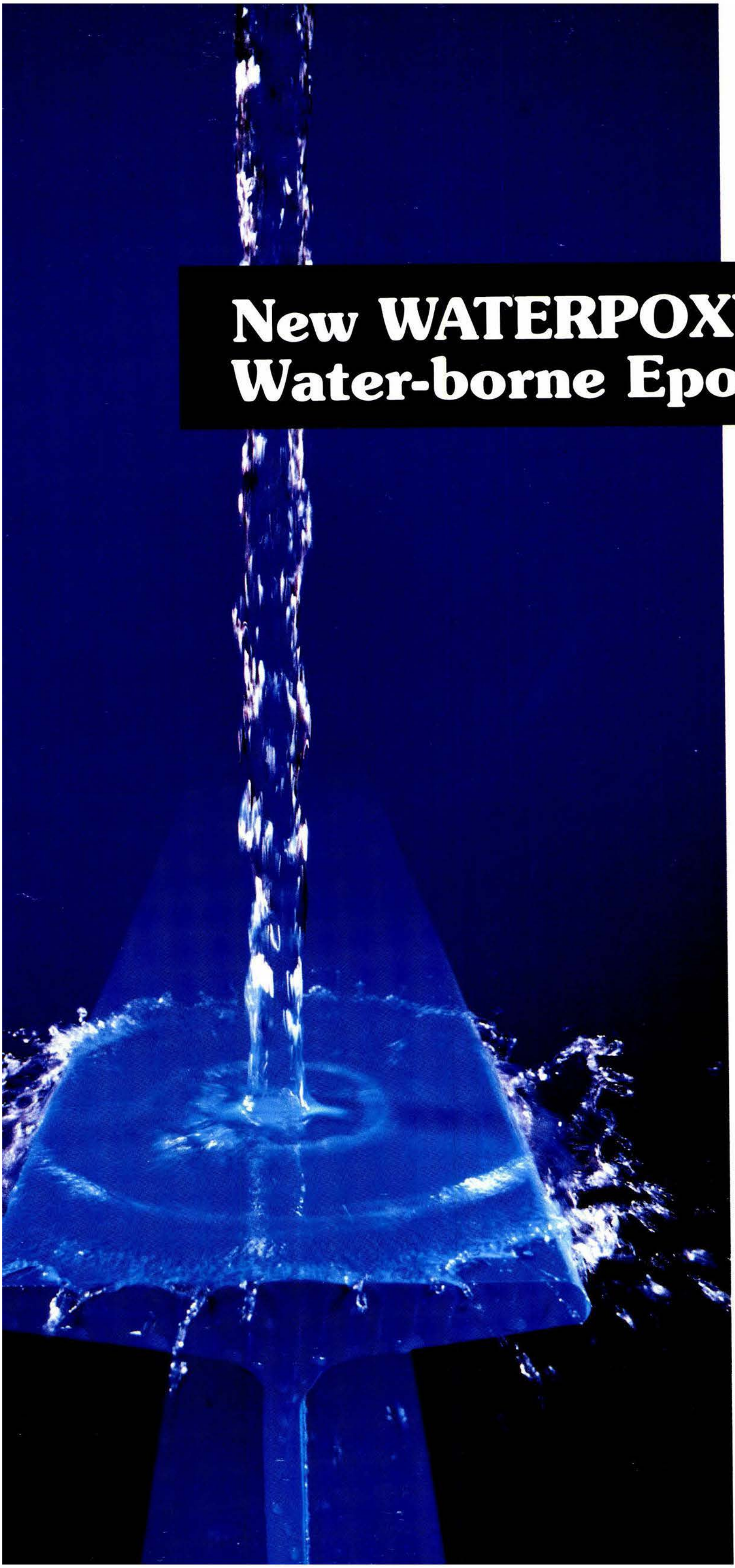
Where is the thicket? Gone. Where is the eagle? Gone. The end of living and the beginning of survival.

● ● ●

I don't know if Chief Seattle, in fact, wrote the above. I'm not sure there was a Chief Seattle of the whatchamacallit tribes. Even if it was written by Kevin Costner's script writer, it's worth a thought or two!

But as a final note, it might be appropriate to mention a quote attributed to Ben Franklin that was sent to me by Milt Glaser, "Men and melons are hard to know."

*—Herb Hillman*
*Humbug's Nest*
*P.O. Box 135*
*Whitingham, VT 05361*



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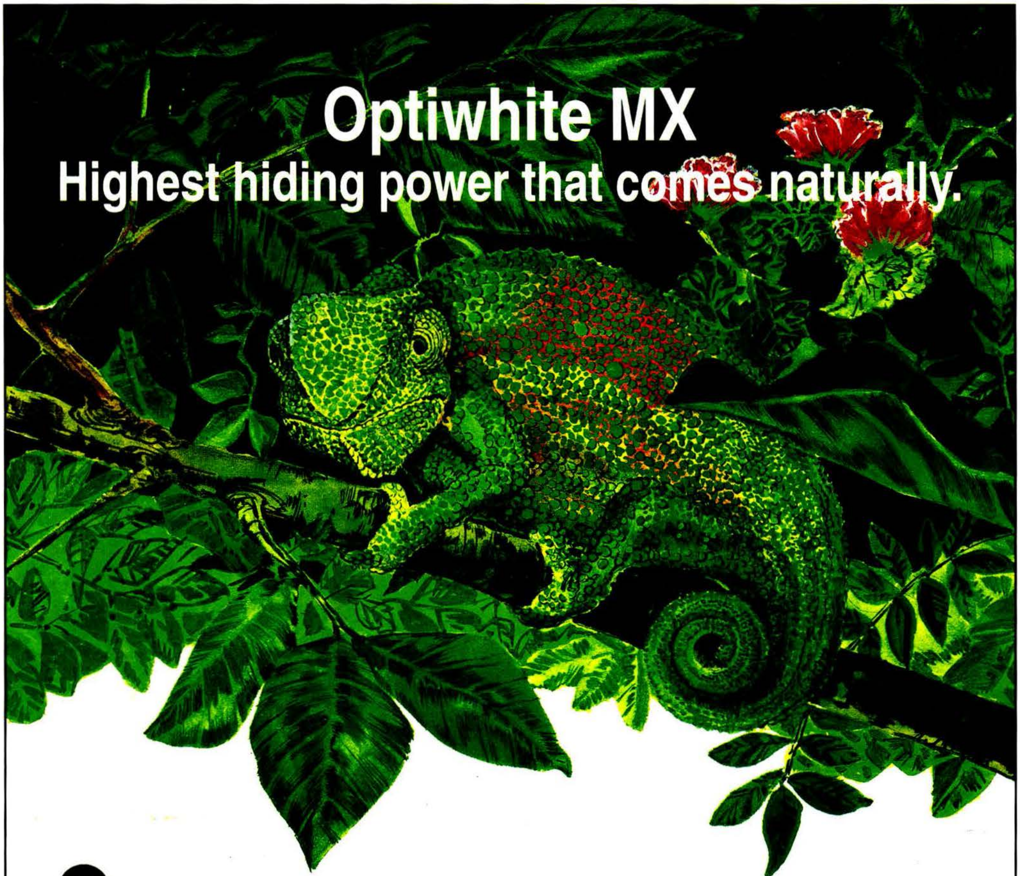

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