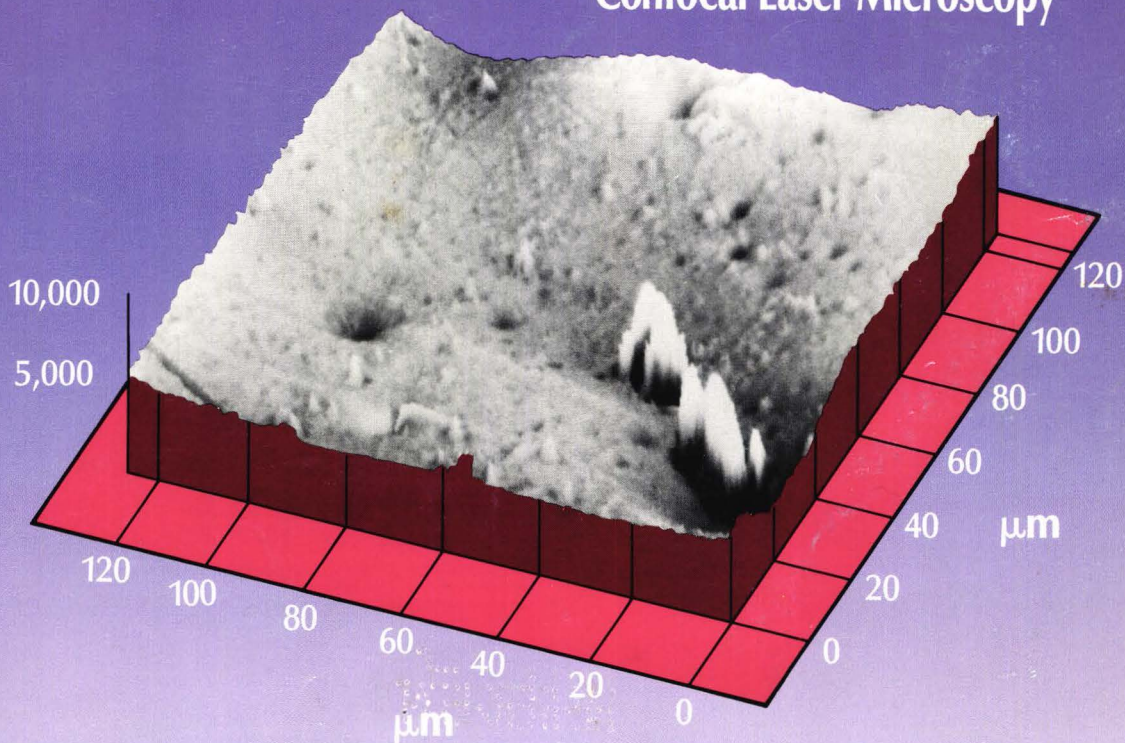


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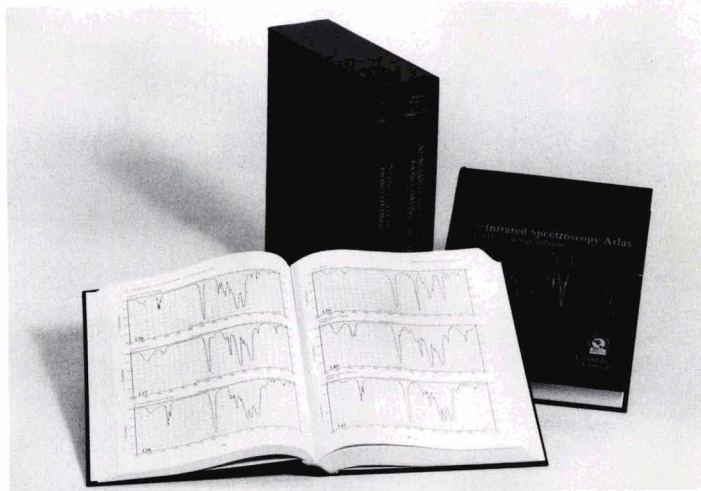
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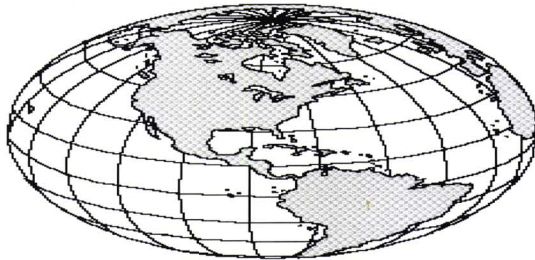
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The Editors invite submission of original research papers, review papers, and papers under the special headings *Open Forum* and *Back to Basics*, and *Letters to the Editor*. All manuscripts will be assumed to be previously unpublished writing of the authors, not under consideration for publication elsewhere.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.**

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**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

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### ...for Roon Foundation Award Competition

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## 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 repetitious 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 solicited. 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 problems 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 8½ 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.

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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.

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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

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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.

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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.

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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.

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- (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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# Comment



## The Seal of Approval

Last month's FSCT Paint Industries' Show saw a return to New Orleans and a record number of exhibiting companies in attendance. Surveyed annually, exhibitors are asked to rate various aspects of their experience at the show. This year the overwhelming response is positive, showing a continuing growth not only in the success of the show, but also in the confidence of the supplier community.

A significant factor in the success of the Paint Show is the growing international attendance. Included among the industry professionals gathered in New Orleans were a large number of overseas visitors. Represented in this number were members of the organizations comprising the Coatings Societies International (CSI). The associations of coatings technologists in the United Kingdom (OCCA), Europe (FATIPEC), Scandinavia (SLF), Japan (JSCM), and Australia (SCAA), and New Zealand (SCANZ) have joined with the FSCT to bring to one table a single voice in coatings technology affairs in the international marketplace.

Discussions of the group have centered on shared concerns: environmental and regulatory issues; new technology and its application; and communication and cooperation were only some of the topics reviewed in New Orleans. One issue, however, spoke directly to the responsibility of the associations to their memberships and the competitive climate in which they must operate.

A consensus of the members of CSI indicated that the mission of the associations included the duty of providing information to their members. Included in this mission is the sponsorship of forums for interaction between their members and the opportunity to learn of new technology, manufacturing equipment, services, etc. In other words, conventions and trade expositions. Several of the member organizations, notably the FSCT, OCCA, SLF, SCAA, and JSCM, do sponsor trade shows in their respective regions, with success.

Of late, however, an increasing number of privately run trade shows in our industry have been aggressively competing for attendance and exhibitors. While we would be undemocratic to rail against the undeniable competitive nature of these expositions, we would suggest that to support attendance and exhibits at CSI-member events would be in the best interests of the industry. For, while resources expended to exhibit and attend private events remain in private pockets, the investments made in support of association-sponsored conventions are returned to the industry and the memberships many-fold, in the form of educational services and activities benefiting the industry and its members.

You will see the CSI logo a lot in the future. It is our seal of approval; your mark of quality. It will designate those events which provide the maximum return on the dollar, or pound, or krone, or mark, or yen, or franc.

If you have any questions or comments, please give us a call.

Robert F. Ziegler  
Secretary General, CSI



# Abstracts of Papers in This Issue

(Translation provided by: Cesar Fuentes Carrasco, of Instituto Mexicano de Tecnicos en Pinturas y Tintas.)

## Defects in Solvent-Free Organic Coatings Studied by Atomic Force Microscopy, Scanning Acoustic Microscopy, and Confocal Laser Microscopy—J. Lange, J.-A. E. Månson, and A. Hult

JCT, 66, No. 838, 19 (Nov. 1994)

Defects in transparent, solvent-free vinyl ether, and allyl ether maleate-functional coatings have been studied using conventional optical microscopy, scanning electron microscopy, atomic force microscopy, scanning acoustic microscopy, and confocal laser microscopy. Vinyl ether films with different curing rates and allyl ether films with different crosslink densities were prepared, and the number and appearance of defects present in the films were compared. The varying curing rates were achieved by using different initiators, whereas the changes in crosslink density were obtained by using monomers with different functionalities. Defects, such as craters and surface crumpling, were observed. Higher curing rates and increased crosslink densities were both found to promote the formation of defects. Atomic force microscopy, scanning acoustic microscopy, and confocal laser microscopy proved to be useful techniques in the characterization of organic coatings, with the latter being particularly well suited for viewing the interior of transparent coatings.

## Epoxidized Polybutadiene: A Novel Prepolymer for Cationically UV-Curable Coatings—F. Cazaux et al.

JCT, 66, No. 838, 27 (Nov. 1994)

Epoxidized polybutadiene was evaluated as a reactive prepolymer for cationically curable photosensitive compositions. Various epoxy or vinyl ether functional reactive diluents were used as viscosity reducers. Compositions with an average content of 5 mmol.g<sup>-1</sup> of polymerizable functions were submitted to viscosity measurements, and their reactivity under UV-irradiation was examined by UV and/or IR spectroscopy. The initiator photolysis can be monitored easily and adequate exposure times were determined by simple kinetic treatment. The polymerization of epoxy monomers was driven to high conversion (over 95%) provided that the film was submitted to a soft thermal post treatment. Coatings of high flexibility and showing good adhesion to aluminum, steel, and glass were obtained by adequate adjustment of the formulations composition. The content in reactive diluent was kept to a low level without loss of curing ability.

## Mechanical Stability of Alkyd Emulsions—Part 2: Influence of Alkyd Properties—G. Östberg and B. Bergenståhl

JCT, 66, No. 838, 37 (Nov. 1994)

The development of waterborne alkyd paints based on alkyd emulsions has been stimulated by more stringent health and environmental regulations. In addition, alkyd paints may have better performance in some applications compared to other types of waterborne paints such as latex dispersions. One problem that needs to be overcome is the limited colloidal stability of alkyd emulsions. It is important that the emulsions are not destabilized when exposed to shear forces, such as those produced by pumps used in the paint industry. An alkyd emulsion consists of alkyd oil droplets dispersed in water and stabilized by a surface active agent (emulsifier). Part 1 of this series described the influence of emulsion droplet size, type, and concentration of the emulsifier on the shear stability. The present study is an additional investigation of the influence of the alkyd properties on shear stability. Increasing the acid value of the alkyd increases the

## Defectos de Recubrimientos Organicos Libres de Solvente Estudiados por un Microscopio de Fuerzas Atomico, un Microscopio de Escaneo Acustico y un Microscopio Laser Focal—J. Lange, J.-A. E. Månson, and A. Hult

JCT, 66, No. 838, 19 (Nov. 1994)

Los defectos en recubrimientos vinil eter libres de solvente y recubrimientos funcionales de alil eter maleato han sido estudiados usando los siguientes equipos; un microscopio óptico convencional, un microscopio de escaneo acústico y un microscopio laser focal. Las películas de vinil eter con diferentes velocidades de curado y películas alil eter con diferentes densidades se preparon y se comparó el número y apariencia de los defectos presentes en las películas. La variaciones de las proporciones de curado se realizaron por el uso de diferentes iniciadores, mientras que los cambios de densidad de entrecruzamiento se obtuvieron por el uso de monómeros con diferentes funcionalidades. Se observaron defectos como cráteres y arrugas de superficie. Se encontró que las altas proporciones de curado e incrementos de densidades de entrecruzamiento son promotores de la formación de defectos. El microscopio de fuerzas atómico, el de escaneo acústico y el laser focal proveen de técnicas utiles en la caracterización de recubrimientos orgánicos, la última técnica mencionada es particularmente conveniente para la observación de recubrimientos transparentes.

## Polibutadieno Epoxidizado: Un Novedoso Prepolimero Para Recubrimientos Curados Cationicamente Por UV—F. Cazaux et al.

JCT, 66, No. 838, 27 (Nov. 1994)

El polibutadieno epoxidizado fué evaluado como un prepolimero reactivo en composiciones fotosensitivas curables catiónicamente. Varios diluyentes con reactivos funcionales epóxi o de vinil eter fueron usados como reductores de viscosidad. Las composiciones con un contenido en promedio de 5 mmol.g<sup>-1</sup> de las funciones polimerizables se sometieron a mediciones de viscosidad y de funciones polimerizables se sometieron a mediciones de viscosidad y de reactividad bajo irradiación UV por espectroscopia UV y/o IR. El iniciador de la fotólisis puede ser fácilmente monitoreado y se pueden adecuar los tiempos de exposición por un simple tratamiento químico. La polimerización de monómeros epóxicos se lleva a una alta conversión (arriba del 95%) que hace que la película sea sujeta a post-tratamiento térmico. Los recubrimientos de alta flexibilidad y que muestran una alta adhesión al aluminio, acero y vidrio fueron obtenidos por ajustamientos de la composición de formulaciones.

## Estabilidad Mecanica de Emulsiones Alquidalicas. Parte 2: La Influencia de las Propiedades Alquidalicas—G. Östberg y B. Bergenståhl

JCT, 66, No. 838, 37 (Nov. 1994)

El desarrollo de las pinturas alquidálicas base agua en emulsiones alquidálicas han sido estimuladas por regulaciones ambientales y de salud cada vez más estrictas. En adición, las pinturas alquidálicas pueden tener un mejor desempeño en muchas aplicaciones comparandolas con otro tipo de pinturas base agua como es el caso de las dispersiones latex. Un problema que necesita ser observado es el límite de estabilidad coloidal de las emulsiones alquidálicas. Es importante que las emulsiones no sean desestabilizadas cuando se

stability of emulsions stabilized with nonionic emulsifiers. Emulsions made using alkyds with shorter oil lengths and higher viscosities were less stable to shear compared to alkyds with higher oil lengths and lower viscosities.

### Interfaces of Polyphenyletheretherketone (PEEK) and Polyphenylene Sulfide (PPS) Coated Zinc Phosphated Steels after Heating-Cooling Cycles in a Wet, Harsh Environment—T. Sugama and N. Carciello

JCT, 66, No. 838, 43 (Nov. 1994)

To evaluate the ability of polyphenyletheretherketone (PEEK) and polyphenylene sulfide (PPS) thermoplastic coatings that protect zinc phosphate (Zn•Ph)-treated steels from corrosion in a wet, harsh environment (1.0 wt% H<sub>2</sub>SO<sub>4</sub>, 3.0 wt% NaCl, and 96.0% wt% water at temperatures from 25° to 200°C), we exposed them in autoclave to attempt heating-cooling cyclic fatigue tests (1 cycle = 12 hr at 200°C + 12 hr at 25°C) up to 90 times. Although no changes in appearance were seen in the PEEK specimens after 60 cycles, extending the cycles to 90 caused the delamination of the coating film from the Zn•Ph. The major reason for this delamination was the degradation of the PEEK polymer caused by its hydrothermal-catalyzed esterification. In the PPS-coating systems, chemical reactions at the interfaces between the PPS and Zn and Fe in the Zn•Ph layer during cycling led to the formation of ZnS and FeS reaction products, which enhanced the Zn•Ph-to-PPS adhesive bond; correspondingly, there were no signs of peeling and separation of the coating after 90 cycles. In addition, because these intermediate reaction products are insoluble at high pH, they minimized the rate of delamination of the PPS coating caused by the cathodic reaction, H<sub>2</sub>O + 1/2O<sub>2</sub> + 2e<sup>-</sup> → 2OH<sup>-</sup>, at the corrosion side of a defect in the film. In contrast, PEEK coatings containing non-reactive Zn•Ph underwent cathodic delamination, because of the susceptibility of Zn•Ph to alkali dissolution. Thus, we believe that combined layers of PPS and Zn•Ph provide great protection to steels in a harsh environment with temperatures up to 200°C.

exponen a fuerzas de corte como son las producidas por bombas utilizadas por la industria de pinturas. Una emulsión alquídica consiste de gotas dispersas de aceite alquídico en agua, siendo estabilizados por un agente activo de superficie (emulsificador). La parte una de este serie describe la influencia del tamaño de la gota de emulsión y el tipo o concentración del emulsificante en la estabilidad de fuerzas. El presente estudio es una investigación adicional de la influencia de las propiedades alquídicas en la estabilidad contra fuerzas de corte.

### Interfaces de Polifeniletertercetona (PEEK) y Polifenileno Sulfida (PPS) en Acero Recubierto de Zinc Fosfatado Despues de Ciclos de Calentamiento-Enfriamiento en un Intemperimetro de Humedad—T. Sugama and N. Carciello

JCT, 66, No. 838, 43 (Nov. 1994)

Para evaluar la estabilidad de recubrimientos termoplásticos con PEEK y PPS para proteger el acero tratado con fosfato de zinc (Zn•Ph) contra la corrosión en un intemperimetro severo de humedad (1.0% en peso de H<sub>2</sub>SO<sub>4</sub>, 3.0% peso de NaCl y 96.00% peso de agua a temperaturas de 25 a 200°C) se expuso en a pruebas de fatiga en autoclaves en ciclos de calentamiento-enfriamiento por arriba de 90 veces, (1 ciclo = 12 hr a 200°C + 12 hr at 25°C). Sin embargo, ningún cambio en la apariencia se observó en lo que respecta a los especimenes de PEEK después de 60 círculos, extendiendo esta prueba a 90 ciclos se originó la delaminación de la película de recubrimiento con Zn•Ph. La mayor razón para esta delaminación fue la degradación del polímero de PEEK causada por su esterificación catalizada hidrotérmica. En los sistemas de recubrimiento PPS las reacciones químicas en las interfaces entre el PPS y Zn y Fe en la capa de Zn•Ph durante el ciclo llevan a la formación de productos de reacción como ZnS y FeS los cuales realzan la adhesión de Zn•Ph a PPS. No hay señas de despellejamiento y separación del recubrimiento despues de 90 ciclos. En adición, debido a que los productos intermedios de reacción son insolubles en altos PH, estos minimizan la proporción de delaminación del recubrimiento PPS causada por la reacción catódica, H<sub>2</sub>O + 1/2 O<sub>2</sub> + 2e<sup>-</sup> → 2OH<sup>-</sup>, en el lado de corrosión de un defecto en la películas. En contraste, el PEEK sufrió una delaminación catódica debido a la susceptibilidad de Zn•Ph a la disolución alcali. Por lo que se cree que combinando las capas de PPS y Zn•Ph se provee de una gran protección al acero en un intemperimetro de humedad con temperaturas por arriba de los 200°C.

Coming Up in the December Issue of the JCT!

## Annual Meeting and Paint Show Wrap-Up Issue



This issue will feature highlights of the FSCT Annual Meeting and Paint Industries' Show held on October 12–14, in New Orleans, LA. Photo displays of the award winning booths, and a complete review of the Annual Meeting, including award winners and special events, will be highlighted.



# PICTORIAL STANDARDS OF COATINGS DEFECTS

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# Committee Activities



## FSCT Educational Coordinating Committee Meets With Society Educational Committee Chairmen

Representatives of Society Educational Committees met recently with members of the Federation's Educational Coordinating Committee in St. Louis, MO. Topics of discussion included: Society-sponsored educational activities; scholarship programs of individual Societies; and an update on FSCT Educational Coordinating Committee activities.

The meeting was chaired by FSCT Education Committee Chairman Don Boyd, of the Pittsburgh Society.

In addition to Mr. Boyd, the following Coordinating Committee members were in attendance: Melinda Rutledge (Los Angeles); Harvest Collier (St. Louis); Gerry Mattson (Southern); and Walter Fibiger (Toronto).

The following Society Educational Committee Chairmen attended: Frank Leo (Chicago); Denise Wolens (Cleveland); Ron Andrus (Detroit); Ed Barrie (Los Angeles); Juan Elizondo (Mexico); H. Mustapha Bacchus (Northwestern); Beverly Spears (Pacific Northwest); Rich Granata (Philadelphia); and Mark Harley (Pittsburgh).

Also attending were the following FSCT Staff Members: Michael G. Bell, Director of Educational Services, and Pat Viola, Director of Publications/JCT Editor.

### Society Reports on Activities

Each Society Educational Committee Chairman was requested to submit a brief summary of the Society's educational activities for presentation at the meeting. The following summarizes the written and/or oral reports submitted.

#### Chicago

Held SYMCO 1994, a one-day seminar which covered the following topics: VOC regulations for coatings, additives for high-solids coatings, chemistry of EVA terpolymers, fundamentals of radiation curing, future trends for UCAR emulsions, powder coatings, and biological contamination control. Sixty-six people attended the meeting. The Society is attempting to develop a cooperative developmental program with DePaul University with the goal being R&D work on high solids. Awarded seven \$2,000 scholarships: two were grant-in-aid awards and five were awarded via a lottery-type drawing.

#### Cleveland

Sponsored a one-day technical symposium with the following two themes: "Advances in Film Characterization" and "Chemistry and Physics of Film Formation." The Society offers speakers for Career Days, Science Fair advisors and judges, and hosts for science field trips and has had an overwhelming response. The Educational Committee has awarded one scholarship this year for \$1,200. Future goals include adding more members to the committee, broadening the base of speakers, and increasing the amount of tours given at local laboratories.

#### Detroit

Re-established its FOCUS Committee in 1994 to enhance the annual technical meeting sponsored by the Society. The result was an increase in the attendance at the seminar from 40 in 1993 to 160 in 1994. The Society also sponsored a course at the University of Detroit-Mercy entitled, "Problem Solving in Coatings Using Analytical Instrument Techniques," conducted by Dr. Manoj Gupta, of BASF Corporation. The Society was an active participant in the Metropolitan Detroit Science Fair and Career Day, where students could learn more about the coatings industry at the Society booth. The Educational Committee is trying to increase the number of talks available on VOC limitation and/or waste management, in addition to attracting more attendees for the monthly meetings of the Society. Scholarships and awards totaling \$4,700 were presented.

#### Kansas City

Four members served as judges at a local science fair and two projects pertaining to the coatings field were selected as winners. The winning students each received a \$100 savings bond and the teachers of the students received \$100 each for use in the science department of their respective schools. The winning entries were displayed at the May meeting of the Society and the winners, along with their parents and their sponsors, were in attendance. The Society donated \$500 to the University of Missouri-Rolla to be used to assist students eligible for degrees in Polymer Science and Coatings. The Society is also seeking ways to stay involved with educational activities despite the shrinking membership base.

#### Los Angeles

Presented 10 scholarships, totaling \$12,050, in the name of the Society and the Cal-Poly Memorial Fund, and contributed \$1,000 to Cal Poly-San Luis Obispo in support of a project on recycling paint. The Paint Technology Course is taught in three trimester parts. The original enrollment was 36 students, with 18 students expected to complete the course this summer. Members participated as guest speakers in high school and two-year college chemistry programs to improve the industry image. Hopes are to expand this program in the fall. The manufacturing seminar is gaining attendees, with 55 members participating this year. The Educational Committee is investigating a bi-annual symposium on water-based coatings and new technologies.

#### Mexico

Held a series of seminars in Mexico City, Monterrey, and Tlaxcala, with the Tlaxcala seminar featuring speakers from three countries. The goals of the Society include holding longer seminars, purchasing a PANORAMA™ Coatings MSDS CD Retrieval System for the membership, and redefining the library policies to attract more members. Membership in the Society is decreasing but the attendance at the seminars is going up.

#### New England

Awarded six scholarships, totaling \$2,500, to college students with an interest in coatings technology, and presented 34 students from the industry as candidates for B.S. and M.S. Degrees at the University of Massachusetts-Lowell. Courses offered in 1993-94 were: Mechanical Behavior of Polymers; Adhesion and Adhesives; Coatings Science and Technology; Colloids; and Polymer Science. Three students completed coatings theses during 1993.

#### Northwestern

Held its annual symposium in March with the following topics covered: design of experiments for coating breakthroughs; rheology modification of water-based paints; current topics in accelerated corrosion testing; and total color management. The Soci-



ety sponsored its annual Educators Night and featured five presentations on education. Scholarships totaling \$2,500 were donated to North Dakota State and a \$400 grant was received from the Federation for scholarship use. Northwestern Society is actively seeking speakers to cover the following subjects at monthly meetings: technologies which are environmentally friendly, lead abatement, efficient dispersion, experimental design, and air quality.

### **Pacific Northwest**

The three sections of the Society had active agendas in 1993-94. The Vancouver Section conducted its annual spring symposium in Victoria, BC. The theme of the event was "Practicality Equals Quality and Efficiency." The Section also sponsored an introductory course with 16 students in attendance. The Puget Sound Section sponsored a course entitled "Basic Coatings for Sales and Marketing Personnel," and the Portland Section sponsored an Environmental Seminar covering HM 181. The Society awarded three \$1,000 scholarships to individuals to attend a course at Cal Poly-San Luis Obispo.

### **Philadelphia**

Held its annual seminar on May 23 entitled, "Filler Pigments, More than Just Dead Space." Approximately 100 attended. The other educational offering, "Basic Coatings Chemistry," is on hold until the instructor can accommodate the time commitment. The Society also sponsors a short course, "Corrosion and its Control by Protective Coatings." This course again will be offered in September 1994. Plans are underway to offer additional courses tailored to the special interest of area members. The Society provides subsidies to its members who attend the "Basic Coatings Chemistry" course.

### **Pittsburgh**

Continues to concentrate its educational efforts in the high schools and presents selected parts of the video, "The Choice." The Society approved a \$1,000 scholarship to send a student to the University of Southern Mississippi. The Society participated in the Pittsburgh Regional School Science and Engineering Fair by providing a judge and a cash award for the program.

### **Rocky Mountain**

Awarded attendance at a short course at the University of Missouri-Rolla to a member of the Society and plans to continue this practice in the future. Funding for the award was from the Society's funds and the Small Society Scholarship program. Rocky Mountain is looking into the possibility of ex-

panding into Arizona and the Society's Executive Committee plans to visit there to review member benefits and also hold a regulatory update meeting.

### **Southern**

The Atlanta Section of the Society has established a grant for research at the University of Southern Mississippi.

### **Toronto**

Sponsors a series of coatings courses at George Brown University. The course runs in three cycles with approximately 20 students enrolled. The Educational Committee has developed a three-volume text for the program and this is being improved before publication. The Society is sponsoring a Distance Learning Program, which was successful in its test run in late 1993. Eventually, this course will be broadcast to eight locations. Investigating whether it should be done as an in-house training program. The Toronto Society financially assisted several students in the coatings program with \$150 per student. This is done to counter the increased tuition at the university due to government cuts. Sponsored a one-day symposium in April entitled, "The Changing Challenges of Coatings Formulation," and had 100 attendees.

### **Educational Coordinating Committee Update**

Chairman Boyd reviewed the status of the Educational Coordinating Committee (ECC) and the programs the committee is developing for the Societies. He began by discussing the relationship of the ECC with the Coatings Industry Education Foundation (CIEF) and said that CIEF distributes funds for equipment grants, scholarships, etc. He added that the colleges and universities are doing well. Mr. Boyd covered the Professional Development Committee's (PDC) relationship with the ECC and said the PDC does the programming for the special events such as the TQM, SPC and Clean Air programs.

He stated that the ECC leverages the efforts of the Societies at the local level and lets them know what FSCT can do to assist them in any educational activity.

Mr. Boyd reviewed the Society Speakers Program and explained how the program will help Societies with their meetings by providing one speaker that will deliver a presentation at the monthly meetings of the society. The program will be funded by FSCT and will include speakers that normally would not be available to deliver a presentation at a monthly meeting.

The Committee is also studying curriculum ideas but has had a difficult time collecting the data for the project. It is proving

to be much more time consuming than originally planned.

Mr. Boyd also discussed the Coatings Science Resource Kit, which was developed in response to requests from various Societies. He covered what has been done so far on the project and said it will be very helpful for members when speaking to schools and community groups. The "Kit" will provide information on what is needed in the way of methods and resources to perform coatings-related experiments.

Mr. Boyd updated attendees on the Small Society Scholarship program. This program is designed to assist Societies with funds for educational-related programs by providing matching funds up to \$400.

### **Mentoring—Reaching Out to Your Community**

Dr. Albert Edney, a Senior Research Chemist at Monsanto in St. Louis, addressed the group on his experiences with mentoring to inner city youngsters of Afro-American descent. He said he became involved because he realized the challenges these children face. He said that 40-50% of the ninth graders in the inner city public schools don't make it to graduation, and in the Black community, mentoring is necessary. Many of the people are learning things on the street that really should be taught in the classroom, with negative results.

Dr. Edney is a member of the Association of Black Scientists (ABS), a 300-member group in St. Louis that touches the community through mentoring.

ABS has established a plan which brings the information to the students. Dr. Edney is currently working with a student who recently won a science project award and has also won a four year scholarship to Morgan State. Through the mentoring program this student has learned that he has the ability to succeed.

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## Keystone Center Officially Concludes AIM Reg-Neg as U.S. EPA and Industry Caucus Fail to Meet Consensus

Despite not reaching a consensus, participants in the architectural and industrial maintenance coatings regulatory negotiation (AIM reg-neg) have been notified by the Keystone Center that the reg-neg has concluded.

According to Keystone, neither the proposals made by the U.S. Environmental Protection Agency (EPA) in June or July 1994, nor the Industry Caucus counter proposal offered in mid-August, has the potential to form the basis for a consensus agreement.

EPA officials have indicated that they expect to release a proposed national AIM rule limiting volatile organic compound (VOC) emissions in early 1995, with a final rule being issued by late 1995 and implementation beginning sometime in 1996. Also, EPA has said that the rule most likely will be based on a phased approach, and it will contain some type of an innovative control mechanism (i.e., fees, preferred products, research grants, etc.). The agency has not discussed potential VOC levels.

Meanwhile, due to the reg-neg's unsuccessful conclusion, many states will not be

able to meet their 15% rate of progress goals, since they had taken credit in their state implementation plans (SIPs) for the VOC emission reductions that EPA had projected would result from the national AIM reg-neg rule.

Therefore, a number of states are moving ahead with the development of their own AIM rules. Currently, AIM coatings rulemaking is underway in seven states. Another 13 states will probably begin development of new or revised AIM rules within the next year. In addition, another 10 states could be forced to initiate rulemaking if their SIPs are deemed inadequate by EPA.

With the reg-neg's conclusion and increased state-level activity, NPCA has undertaken a review of its strategy and policy guidance for dealing with the development of AIM VOC rules and legislation at all levels of government. Recently, the two committees that have shared responsibility for developing association policy in this area—Architectural Coatings and Air Quality Committees—met at NPCA headquarters at separate meetings in September. At

these meetings, the committees discussed the development and implementation of a new AIM VOC strategy and policy guidance for the association which were submitted to the board of directors and executive committee for approval at NPCA's Annual Meeting in October. A key element of the strategy is the formation of a new AIM Steering Committee that is broadly representative of the NPCA membership. The committee would be operational in nature, in that its main function would be to guide and direct NPCA's active involvement with federal, state, and local regulators and other government bodies to ensure that any AIM regulations that may be established are reasonable, e.g., that they are technically and economically feasible.

For information on the reg-neg's conclusion, state AIM regulatory efforts, or the association's new AIM Steering Committee, contact NPCA's Bob Nelson or Jim Sell at (202) 462-6272.

## Second Quarter Powder Coatings Sales Increase 16%

In a report released by the Powder Coating Institute, Alexandria, VA, North American sales of thermostat general decorative powder coatings for the second quarter of 1994 rose 16% in pounds over the same quarter in 1993, and nearly 13% ahead of the 1993 year-to-date pound figure. Total powder sales topped \$124 million for the quarter, a 19.6% increase over the second quarter of 1993, resulting in a 16.9% increase over the year-to-date figure for 1993. Powder coating sales for all of 1994 are projected to approach 210 million pounds.

All powder market categories showed sales increases for both the quarter and the year-to-date. Appliance boasted a 33% quarterly growth and 15% year-to-date. Lawn and garden was up 25% for the quarter and 27% for the year, followed by automotive at 22.4% for the quarter and 18.8% for the year. General metal finishing was up 5% for both the quarter and the year.

Dollar sales of application equipment increased 57% in second quarter comparisons to 1993, and 53% in year-to-date comparisons. Sales of automatic guns showed the most dramatic increase over 1993, nearly

23% for the quarter and 8% in year-to-date comparisons. Year-to-date sales of manual guns showed a modest increase, and sales of portable manual units remained even. Total sales of powder recovery booths increased 19% for the quarter and remained even for the year.

Total powder coating installations each containing one or more booths, either manual or automatic/manual, decreased slightly for the quarter, and for the year-to-date. The industry estimate of total dollar sales of powder coating application and recovery equipment is expected to approach \$78 million in 1994.

## Shell Chemical Expands Resin Units in Texas and Florida

The Shell Chemical Co., Houston, TX, announced the expansion of its Epon® solid resin unit in Deer Park, TX. This unit manufactures solid resin products for many applications, including powder coatings.

As a result of this project, a new Epon resin product form (beads), designed to help

minimize product degradation and cross-contamination, will be available.

In other news, Shell Epi-Resz® waterborne epoxy resins facility in Lakeland, FL, is on stream. A second major expansion for waterborne epoxy resins will be completed by the fourth quarter of 1994.

### U.S. Polymers and Accurez Purchase Shell Division

U.S. Polymers, Inc. and its Accurez Corp. affiliate of St. Louis, MO, have purchased the Paper, Film, and Foil Division of Shell Chemical Co. Terms of the transaction were not disclosed.

Products included in the acquisition include Clear Rez®, Epi-Tex®, Syn-Tex®, and Syn-U-Tex®. These products will be produced in the St. Louis manufacturing facility and marketed through Accurez. U.S. Polymers is a manufacturer of specialty polyesters, alkyds, epoxy esters, and dispersion resins for the paint and coatings industries.

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# Regulatory UPDATE

NOVEMBER 1994

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.

**Environmental Protection Agency**  
**September 30, 1994—59 FR 49888**  
**Acetone; Toxic Chemical Release Reporting; Community Right-to-Know**  
**Action: Proposed rule**

In response to a petition filed by Hoechst Celanese and Eastman Chemical Co., the U.S. Environmental Protection Agency (EPA) is proposing to delete acetone from the list of toxic chemicals subject to Section 313 of the Emergency Planning and Community Right-to-Know of 1986 (EPCRA).

Specifically, EPA is granting this petition by proposing to delist because the agency believes that acetone does not meet any of the EPCRA Section 313 (d) (2) criteria for remaining on the list. However, EPA is also proposing to add acetone to the list of compounds excluded from the definition of "A Volatile Organic Compound" (VOC) under the Clean Air Act. EPA has previously determined that VOCs meet the criteria for listing under EPCRA Section 313. Therefore, finalization of this proposed rule is contingent upon the finalization of the proposed rule to exclude acetone from EPA's definition of a VOC.

Written comments must be received by November 29, 1994, and should be submitted in triplicate to the OPPT Docket Clerk, TSCA Nonconfidential Information Center (NCIC), also known as the TSCA Public Docket Office (7407), Office of Pollution Prevention and Toxics, U.S. EPA, Rm. NE-B607, 401 M St., S.W., Washington, D.C. 20460. Comments should include the document control number for this proposal, OPPTS-400086.

For further information, contact Maria J. Doa, Petitions Coordinator, 202-260-9592, for specific information on this proposed rule. For more information on EPCRA Section 313, call the Emergency Planning and Community Right-to-Know Hotline at 1-800-535-0202. In Virginia call 703-412-9877 or toll free TDD: 1-800-553-7672.

**Environmental Protection Agency**  
**September 30, 1994—59 FR 49890**  
**Lead; Requirements for Lead-Based Paint Activities; Notice of Hearing**  
**Action: Informal hearing**

On September 2, 1994, EPA published a proposed rule governing lead-based paint activities to ensure that individuals engaged in such activities are properly trained, that training programs are accredited, and the contractors engaged in such activities are certified. The proposed rule would also

establish standards for performing lead-based paint activities and require that all lead-based paint activities be performed by certified individuals. When promulgated, the rule would fulfill the mandate of Section 402 (a) (1) of Title IV of the Toxic Substances Control Act (TSCA). Additionally, as part of the proposed rule, EPA has, in accordance with Section 404 (d) of TSCA, developed a proposed Model State Program. When promulgated, this program may be adopted by any state that seeks to administer and enforce a state program under Title IV of TSCA.

EPA was scheduled to hold an informal hearing on October 26 at the Crystal City Hyatt, Arlington, VA.

For transcripts of the hearing or for further information, please call Susan B. Hazen, Director, Environmental Assistance Division (7408), Office of Pollution Prevention and Toxics, U.S. EPA, 401 M St., S.W., Washington, D.C. 20460, (202) 554-1404; TDD: (202) 554-0551. For technical questions, contact Diane Sheridan at (202) 260-0961.

**Environmental Protection Agency**  
**September 19, 1994—59 FR 47980**  
**Hazardous Waste Management System; Testing and Monitoring Activities, Land Disposal Restrictions Correction**  
**Action: Final rule; correction**

EPA is correcting its final regulations which were published Tuesday, August 31, 1993 ("Hazardous Waste Management System; Testing and Monitoring Activities; Final Rule," 58 FR 46040). This action corrects the unintended removal of the text which sets forth the generator waste analysis and recordkeeping requirements of the land disposal restrictions under Subtitle C of the Resource Conservation and Recovery Act of 1976 (RCRA), as amended.

For further information about this correction, contact Kim Kirkland at (202) 260-4761, Office of Solid Waste (Mail Code 5304), U.S. EPA, 401 M St., S.W., Washington, D.C. 20460. For information about the regulatory requirements, contact the RCRA Hotline at (800) 424-9346 or, in the Washington, D.C. metropolitan area, (703) 412-9810.

**Hazardous Materials Transportation**—Legislation reauthorizing the federal hazardous materials transportation safety programs and further deregulating the trucking industry was actually signed into law earlier this fall.

The *Regulatory Update* is made available as a service to FSCIT 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 FSCIT cannot guarantee its completeness or accuracy.



The measure (H.R. 2178—PL 103-113) provides \$75.3 million for fiscal years 1994 through 1997 for programs established by the Hazardous Materials Transportation Act.

Among the provisions in the final version of the bill was the implementation of significant safety changes, including a requirement that the DOT study the use of new highway

technologies to promote safe transport of hazardous materials. Eliminated from the bill was an existing requirement that most interstate trucking companies file their rates with the Interstate Commerce Commission (ICC), a move that diminishes the ICC's role in overseeing interstate trucking.

## States Proposed Legislation and Regulations

### Arizona

*Hazardous Waste (Regulation)*—The Arizona Department of Environmental Quality (DEQ) issued a proposed rule regarding underground storage tanks (USTs). The proposal establishes standards for the installation, operation, and closure of USTs and for the reporting of releases. Contact Martha Seaman, DEQ, (602) 207-2222.

*Occupational Safety & Health (Regulation)*—The Arizona Industrial Commission has announced plans to amend current regulations regarding the reporting of fatalities and multiple hospitalization accidents. Contact Derek Mullins, Industrial Commission, (602) 542-5795.

### Arkansas

*Air Quality (Regulation)*—The Arkansas Department of Pollution Control and Ecology (DPCE) proposed to revise regulations dealing with operating permit fees. The amendments would eliminate certain operating fees and update public notice requirements for operating permits. Contact Mike Porta, DPCE, (501) 562-7444.

### California

*Air Quality (Regulation)*—The California Air Resources Board (CARB) issued a proposed rule that would provide an alternate control plan for volatile organic compound (VOC) emissions from certain consumer products. The voluntary plan would place a limit on aggregate emissions from a group of products, rather than on the VOC emissions from individual products. Contact Floyd Vergara, CARB, (916) 327-1503.

*Graffiti*—CA A. 2240 (McDonald) creates the State Graffiti Clearinghouse in the Office of Criminal Justice Planning and vests specified responsibilities in the clearinghouse relating to graffiti abatement. The governor signed the legislation on September 30.

CA A. 2519 (Karnette) declares that a public entity may include a provision in a public works contract that specifies requirements for anti-graffiti technology in the plans for that project, and may establish a method to finance a graffiti abatement program or a program to deter graffiti entirely. The legislation applies to any situation in which the project is considered to be vulnerable to graffiti and in which the contract will be awarded after January 1, 1996. The governor signed the bill on September 11.

CA A. 2595 (Connolly) requires any minor convicted of possession of a destructive implement with the intent to commit graffiti or willfully affixing graffiti under specified provisions to perform no less than 24 hours of community service, as specified. The bill was signed by the governor on September 15.

CA S. 302 (McCorquodale) authorizes a city to provide for the abatement of any nuisance resulting from the defacement of the property of another by graffiti or any other inscribed material at the expense of the minor creating, causing, or committing the nuisance. It authorizes a city to

adopt an ordinance to make the expense of abatement a lien against property of the minor or the parent or guardian of the minor. The legislation was signed by the governor on September 27.

CA S. 583 (Lewis) adds aerosol paint containers, felt tip markers, and any other marking substance to the list of specified instruments whose use is prohibited if the intent is to commit vandalism or graffiti. The legislation was signed by the governor on September 27.

CA S. 1779 (Bergeson) increases the maximum amount that may be imputed to a parent or guardian for the willful misconduct of a minor to \$25,000. Minors who commit acts of graffiti are required to provide restitution for damages caused. On September 27, the governor signed the legislation.

*Hazardous Waste (Regulation)*—The California Occupational Safety and Health Standards Board (OSHSB) has issued a proposed amendment to current regulations that will require all employers who receive a package, freight container, or transport vehicle which contains a hazardous material to retain any label, placard or marking that is required under the U.S. Department of Transportation's hazardous materials regulations. Under this amendment, such DOT markings, placards, and labels cannot be removed from the incoming package, container, or vehicle until the hazardous material has been removed and the packaging sufficiently cleaned to remove any potential hazard. Contact OSHSB, 1006 4th St., Third Floor, Sacramento, CA 95814-3372.

An emergency rule amending current regulations for corrective action management units (CAMU) was adopted by the California Department of Toxic Substances Control (DTSC). The rule, which went into effect on August 22, 1994, mandates the establishment of these units at RCRA hazardous waste treatment and storage facilities. Additionally, it eliminates disposal requirements, including land disposal restrictions for hazardous materials generated during a corrective action that are managed in a CAMU. Contact Pamela Thompson, DTSC, (916) 324-9933.

*Occupational Safety & Health*—CA S. 1464 (Marks) authorizes the Division of Occupational Safety and Health to prohibit entry to an immediate area of place of employment where it has determined an imminent hazard to employees exists due to the dangerous condition or placement of machinery, or machinery that is improperly guarded. The legislation also sets forth an expedited procedure by which the affected employer may contest the division's order prohibiting entry or use of the area or equipment. The governor vetoed the bill on September 30.

*Toxic Substances (Regulation)*—The California Environmental Protection Agency has announced its intention to revise and republish the list of chemicals known to cause cancer or reproductive toxicity, as required by Proposition 65. Contact California EPA, (916) 445-3846.

### Colorado

*Air Quality (Regulation)*—A final rule dealing with operating permit requirements was adopted by the Colorado De-

partment of Health (DOH). The regulation, among other things, provides exemptions for sources using minor permit modification procedures; creates a \$14.98 per ton fee for regulated pollutants; increases the fee for each filed air pollutant emission notice to \$100; and identifies certain pollutants that are exempt from obtaining operating permits. The rule was effective September 30, 1994. Contact Sara Laumann, DOH, (303) 692-3146.

The Colorado Department of Public Health and Environment (DPHE) proposed a regulation that will allow sources to use emission reduction credits as a method of compliance on a case-by-case basis instead of control technique guidelines (CTGs) or reasonably available control technologies (RACTs). Contact DPHE, (303) 692-2000.

The Colorado DOH has issued a proposal to amend current regulations regarding the definition of "volatile organic compound liquid." The revisions will clarify distinctions between various types of volatile organic compound liquids and provide information on the applicability of the regulations. Contact DOH, (303) 692-3180.

## Connecticut

*Hazardous Waste (Regulation)*—A regulation proposed by the Connecticut Department of Labor (DOL) will adopt federal Occupational Safety and Health Administration (OSHA) standards for general industry which require the retention of Department of Transportation placards or markings on containers of hazardous materials. Contact George Wentworth, DOL, (203) 566-5160.

*Occupational Safety & Health (Regulation)*—The Connecticut Department of Labor (DOL) proposed a rule concerning reporting accidents or fatalities. Under this regulation, employers will have eight hours to report an employee's death or the hospitalization of three or more employees occurring within 30 days of a work-related accident. Contact George Wentworth, DOL, (203) 566-5160.

## Delaware

*Air Quality (Regulation)*—The Delaware Department of Natural Resources and Environmental Control has proposed amendments to a current proposal concerning volatile organic compound (VOC) emissions from automotive refinishing operations. The revision would make the proposal applicable to all refinishing operations, not simply ones which produce 15 pounds per day or more of VOCs. Contact Lee Randolph, DNREC, (302) 323-4542.

*Hazardous Waste (Regulation)*—The DNREC issued a proposed rule which would establish standards for the installation, relining, and closure of underground storage tanks (USTs). In addition, the proposed rule outlines changes to UST confidentiality standards in order to meet federal EPA requirements. Contact Mike Butler, DNREC, (302) 323-4588.

## Florida

*Air Quality (Regulation)*—A rule proposed by the Florida Department of Environmental Protection (DEP) will amend the application procedures for operating permits. Among other things, the regulation will delete the fee category for "major sources," and clarify the fee schedule for facility-wide or multiple emissions unit permits. Contact: Michael Hewett, DEP, (904) 488-0114.

The DEP proposed a regulation to identify the requirements of the state implementation plan (SIP) for ozone non-attainment and adopt public notice and hearing standards for

any SIP revisions. Contact Michael Hewett, DEP, (904) 488-0114.

The DEP issued a proposal to revise current requirements for stationary sources of air pollutant emissions. Under the proposed regulation, sources of hazardous air pollutant emissions are listed, procedures for obtaining non-Title V operating permits are added, and permit notice requirements are revised. Contact Michael Hewett, DEP, (904) 488-0114.

The DEP proposed a rule to amend regulations concerning operating permits for major sources of air pollution in order to clarify local pollution control program requirements. Contact Michael Hewett, DEP, (904) 488-0114.

A proposal issued by the Florida Department of Environmental Protection (DEP) revises monitoring standards of air emissions from stationary sources. Under the proposed rule, test requirements and emissions monitoring standards are modified. Contact Michael Hewett, DEP, (904) 488-0114.

## Iowa

*Hazardous Waste (Regulation)*—The Iowa Comprehensive Petroleum Underground Storage Tank Fund Board (CPUSTFB) has adopted a final rule concerning requirements for installers and inspectors of underground storage tanks (USTs). Under the new rule, testing and licensing procedures will be simpler to implement, and the issuance of temporary licenses will be eliminated. The regulation will go into effect on January 1, 1995. Contact CPUSTFB, (515) 284-1616.

*Occupational Safety & Health (Regulation)*—The Iowa Labor Services Division (LSD) adopted an emergency rule which incorporates, by reference, federal OSHA standards for permit-required confined spaces. The regulation was effective September 14, 1994. Contact LSD, (515) 281-3606.

## Kansas

*Air Quality (Regulation)*—The Kansas Department of Health and Environment (DHE) issued a proposed rule that will establish an operating permit program, as required by the Clean Air Act. The regulation will increase emission fees for air pollutants, and enact new source performance standards, national emissions standards for hazardous air pollutants (NESHAP), and maximum achievable control technology (MACT) for hazardous air pollutants. Contact Chuck Layman, DHE, (913) 926-1579.

*Toxic Substances (Regulation)*—The Kansas DHE proposed a regulation to amend the current Community Right-to-Know Rule. The proposal specifies certain chemicals that are exempt from reporting fees. Contact Jon Flint, DHE, (913) 296-1690.

## Kentucky

*Air Quality (Regulation)*—The Kentucky Department of Environmental Protection (DEP) has announced its intention to adopt federal standards for determining whether the state implementation plan (SIP) conforms to federal requirements. Contact John Hornback, DEP, (502) 573-3382.

The Kentucky DEP proposed a regulation that establishes standards for VOC emissions from beverage can surface coatings operations that are at least as stringent as federal requirements. Contact John Hornback, DEP, (502) 573-3382.

*Water Quality (Regulation)*—The Kentucky DEP adopted a final rule which amends regulations dealing with analytical requirements for VOCs. The rule establishes, among other things, standards for maximum contaminant levels for certain chemicals, procedures for determining compliance with

those contaminant levels, and criteria for granting waivers from analysis requirements. The regulation went into effect on July 27, 1994. Contact John Smither, DEP, (502) 564-3410.

### Louisiana

*Air Quality (Regulation)*—The Louisiana Department of Environmental Quality (DEQ) has issued a final rule which establishes the procedures for banking emission reduction credits. The regulation will allow companies to store emission reduction credits for future use or to meet the 15% emission reduction required by the Clean Air Act. A schedule for submitting credit applications, and the procedures for using and transferring credits, are included. The rule was effective on August 20, 1994. Contact David Hughes, DEQ, (504) 765-0399.

The Louisiana DEQ proposed a rule that will apply reasonably available control technologies (RACT) to control emissions from VOC storage. The regulation applies to sources emitting 50 tons per year or more of VOCs in certain districts in the state. Contact Patsy Deaville, DEQ, (504) 765-0399.

### Maryland

*Lead (Regulation)*—The Maryland Department of Licensing and Regulation (DLR) adopted a final regulation which incorporated, by reference, federal OSHA construction industry standards regarding occupational exposure to lead. The rule was effective September 26, 1994. Contact DLR, (410) 333-4184.

*Occupational Safety & Health (Regulation)*—The Maryland DLR issued a final rule which adopts federal OSHA requirements for permit-required confined spaces (PRCS). It also revises the section dealing with atmospheric monitoring and allows employers to select appropriate testing instruments. The regulation went into effect September 26, 1994. Contact DLR, (410) 333-4184.

### Massachusetts

*Air Quality (Regulation)*—The Massachusetts Department of Environmental Protection (DEP) issued a final rule that establishes standards for operating permits. The regulation, which went into effect on August 12, 1994, details procedures for the submittal and review of operating permit applications, for modifications of existing permits, and for permit reporting requirements. Contact Robert Donaldson, DEP, (617) 292-5619.

A proposed rule of the Massachusetts DEP amends the current state implementation plan for ozone. The regulation includes specific requirements for the labeling of automotive refinishing containers. Contact Marc Cohen, DEP, (617) 292-5630.

### Minnesota

*Air Quality (Regulation)*—The Minnesota Pollution Control Agency (PCA) proposed a regulation to incorporate, by reference, the requirements of the federal operating permit regulation, nitrogen oxide emission reduction requirements, and continuous emission monitoring requirements for acid rain affected sources under Title IV of the Clean Air Act. Contact Norma L. Coleman, PCA, (612) 296-7712.

The Minnesota PCA issued a proposal relating to the development of an air emission operating permit program to

implement the requirements of the Federal Clean Air Act which will include existing source application deadlines and source operation during transition requirements. Contact Andrew Ronchak, PCA, (612) 296-3107.

The Minnesota PCA issued a final regulation which incorporates the national ambient air quality standards for lead and particulate matter (PM-10), as required by the Federal Clean Air Act amendments. The rule went into effect September 13, 1994. Contact Norma Coleman, PCA, (612) 296-7712.

An amendment to current regulations regarding air emission compliance was proposed by the Minnesota PCA. Under this proposal, enhanced monitoring systems will be used in issuing compliance certifications and in establishing evidence of air emissions violations. Contact Norma Coleman, PCA, (612) 296-7712.

### Montana

*Occupational Safety & Health (Regulation)*—The Montana Department of Labor and Industry (DLI) proposed a regulation which will require a safety committee for every employer with more than five employees. In addition, the rule defines the composition, role and duties of the safety committees. Contact John Maloney, DLI, P.O. Box 8011, Helena, MT, 59406-8011.

### New Hampshire

*Occupational Safety & Health (Regulation)*—The New Hampshire Department of Labor (DOL) has repealed certain provisions of its safety and health program regulations. The repealed sections required employers of 10 or more employees to implement written on-the-job safety and health programs, and to establish program specifications. This rule is effective from September 2, 1994 until September 2, 2000. Contact Arthur Flebotte, DOL, (603) 271-6297.

### New Jersey

*Air Quality (Regulation)*—The New Jersey Department of Environmental Protection (DEP) has proposed a rule to assess penalties for violations of the Air Pollution Control Act. The regulation specifies penalties for general air pollution violations, for submitting false or inaccurate permit applications, and for failing to report releases of air contaminants. Contact Janis Hoagland, DEP, (609) 292-0716.

*Graffiti*—NJ A. 765 (Catania and DeCrocce) revises the law concerning graffiti. On September 26, the bill was amended on the assembly floor.

NJ S. 1374 (Girgenti) requires the suspension of a driver's license and community service for acts of graffiti. Introduced on September 19, the bill was sent to the Senate Committee on Law and Public Safety.

*Lead*—NJ A. 2079 (Gregory-Scocchi and Warsh) establishes the Children's Immunization and Lead Screening Program in the Department of Health and appropriates funds for that purpose. Introduced on September 12, the bill was sent to the Assembly Committee on Appropriations.

NJ A. 2130 (Gregory-Scocchi and Felice) mandates health insurance benefits for treatment of lead poisoning. The bill was introduced on September 19 and referred to the Assembly Committee on Insurance.

*Lead (Regulation)*—The New Jersey Environmental Health Services issued a proposed rule which establishes standards for the permitting of lead abatement workers, lead abatement supervisors, lead inspector/risk assessors, and lead



abatement planner/project designers. It specifies the educational and experimental requirements for each discipline, the application process to obtain a permit, the required training course and examination, and renewal procedures. In addition, the regulation requires that all lead abatement workers must be certified by the Department of Health. Contact Joseph D. Eldridge, M.P.H., Environmental Health Services, CN 360, Trenton, NJ 08625-0360.

### New Mexico

*Air Quality (Regulation)*—The New Mexico Environmental Improvement Board (EIB) proposed a rule which requires federal agencies that are planning or implementing general actions to affirmatively show that the action would conform to the state air quality implementation plan, as required by the Federal 1990 Clean Air Act Amendments. Contact Phyllis Ludi, Air Quality Bureau, (505) 827-2971.

The New Mexico EIB has proposed a regulation which requires sources of air pollutant emissions to comply with the enhanced monitoring rules promulgated by the U.S. EPA as required by the Federal 1990 Clean Air Act Amendments. Contact Phyllis Ludi, Air Quality Bureau, (505) 827-2971.

The New Mexico EIB issued a proposed rule that establishes maximum achievable control technology (MACT) standards for sources of hazardous air pollutants (HAPs) by incorporating new federal emission rules. Contact Phyllis Ludi, Air Quality Bureau, (505) 827-2971.

### New York

*Air Quality (Regulation)*—The New York Department of Environmental Conservation (DEC) adopted an amendment to current regulations which establishes emission standards and control requirements for various categories of facilities and satisfies mandates of the 1990 Clean Air Act with respect to the ambient air quality standards for ozone. The amendment went into effect on September 23, 1994. Contact William Delaware, DEC, (518) 457-3200.

*Recycling (Regulation)*—The New York DEC has announced that it will continue consideration of a proposed rule to revise regulations for the use of the official state recycling emblem. The revisions will alter the required percentage of recycled content for items using the emblem and will simplify the registration process. Contact Peter Pettit, DEC, (518) 457-3966.

### North Carolina

*Lead (Regulation)*—The North Carolina Department of Environment, Health and Natural Resources (DEHNR) proposed a regulation which provides for consistency with current recommendations from the Centers for Disease Control and Prevention regarding the definition for "elevated blood lead level," allows laboratory reporting by computer generated report, provides for separate rules regarding notification and abatement requirements, and establishes lead poisoning prevention in child programs. Contact Grady L. Balentine, Department of Justice, (919) 733-4618.

### Ohio

*Air Quality (Regulation)*—The Ohio Environmental Protection Agency (EPA) issued a proposed rule to amend deficiencies in the current state implementation plan (SIP) for VOC reductions. Under the new SIP proposal, can coating and miscellaneous metal parts coatings operations will be subject to more stringent emission limits. Additionally, the

proposal will amend the emission exemption levels for coatings for miscellaneous metal parts. Contact Tammy Saunders, Ohio EPA, (614) 644-2270.

### Oklahoma

*Air Quality (Regulation)*—The Oklahoma Department of Environmental Quality (DEQ) proposed a regulation to correct deficiencies in its air quality implementation plan. Under this proposal, sources of certain regulated pollutants would be required to pay a fee, and modified emissions monitoring requirements would be used to establish state implementation plan violations. Contact Scott Thomas, DEQ, (405) 271-5220.

### Pennsylvania

*Graffiti*—PA H. 3045 (Wogan) prohibits the sale of aerosol spray paint cans and broad-tipped markers to minors and regulates the display of such items. The bill was introduced on September 27 and referred to the House Committee on Judiciary.

*Lead*—PA H. 1865 (McNally) requires training and certification of individuals engaged in lead-based paint activities, and the licensure of lead contractors. The legislation sets the standards for minimum training requirements for lead abatement occupations, and for accreditation and licensure fees. In addition, the bill creates the Lead-Based Paint Abatement Advisory Committee. The legislation was amended and released from the House Committee on Labor Relations on October 4.

### Rhode Island

*Air Quality (Regulation)*—The Rhode Island Department of Environmental Management (DEM) issued a proposal to establish an operating permit fee program, including an annual fee based on a source's potential emissions. Additionally, the regulation would institute operating permit program requirements pertaining to permit content and procedures for permit applications. Contact Douglas McVay, DEM, (401) 277-2808.

### South Carolina

*Occupational Safety & Health (Regulation)*—The South Carolina Department of Labor, Licensing and Regulation (DLLR) issued a proposed rule to revise general industry standards for permit-required confined spaces, personal protective equipment, and retention of Department of Transportation placards and markings to conform to federal requirements. Contact DLLR, Public Information Office, (803) 734-9612.

### Texas

*Air Quality (Regulation)*—The Texas Natural Resources and Conservation Commission (TNRCC) has proposed a regulation that would grant the Commission the authority to adopt rules consistent with the policy of the Federal Clean Air Act. The provisions would include applicability; flexible permit application; application review schedules; emission caps and individual emission limitations; implementation schedule for additional controls; significant emission increases, limitation on operational changes and alterations; distance limitations, compliance history, public notice and comment; and flexible permit fees and permit renewals. Contact TNRCC, (512) 239-1500.

## Utah

*Hazardous Waste (Regulation)*—The Utah Division of Occupational Safety & Health issued a proposed rule which requires employers who receive a package, transport vehicle, freight container, motor vehicle, or rail freight car which contains a hazardous material and which is required to be marked or labeled, in accordance with U.S. Department of Transportation regulations, to retain such markings of labels. Contact Don Anderson, Office of Occupational Safety & Health, (801) 530-6897.

## Vermont

*Occupational Safety & Health (Regulation)*—A rule proposed by the Vermont Department of Labor and Industry (DLI) will incorporate, by reference, federal OSHA requirements for reporting fatalities or multiple hospitalizations. Contact John Roorda, DLI, (802) 828-2765.

## Virginia

*Occupational Safety & Health (Regulation)*—The Virginia Department of Labor and Industry (DLI) issued a final rule to amend current regulations for permit-required confined spaces (PRCS) which alters provisions for atmospheric monitoring. The rule went into effect on October 1, 1994. Contact John Crisanti, DLI, (804) 786-2384.

The final rule of the Virginia DLI adopts federal standards for personal protective equipment. The rule was effective October 1, 1994. Contact John Crisanti, DLI, (804) 786-2384.

## Washington

*Air Quality (Regulation)*—The Washington Clean Air Authority (CAA) has adopted a proposal which updates Article 4, "Air Operating Permits" to be consistent with the new state law on air operating permits. The new regulation went into effect on September 23, 1994. Contact David A. Lauer, Benton Franklin Counties Clean Air Authority, (509) 943-3396.

The Washington Northwest Air Pollution Authority (NAPA) issued a proposed rule which brings the current

NAPA regulations up to date by amending, adding, and deleting sections to reflect changes in the Washington Clean Air Act, Washington Administrative Codes, federal new source performance standards, and national emission standards for hazardous air pollutants. The proposal adds provisions that promote effective air pollution control and raises fees to reflect added costs of performing duties. Contact NAPA, Mt. Vernon, WA, FAX (206) 428-1620.

The Washington Department of Ecology (DOE) issued a final rule that institutes fees for RACT analysis and determination. Under this regulation, which went into effect September 15, 1994, DOE may increase fees based upon the complexity of the analysis, reduce fees for small businesses and sources assisting with category determinations, and institute a fee system for review of proposed new sources. Contact Audrey O'Brien, DOE, (206) 407-6875.

*Hazardous Waste (Regulation)*—The Washington Department of Labor and Industries (DLI) proposed a rule to incorporate federal standards which require hazardous materials to carry placards and other markings during transportation and at stationary facilities. Contact Merle Larson, DLI, (206) 956-5519.

*Occupational Safety & Health (Regulation)*—The Washington Department of Labor and Industry (DLI) announced its intention to revise current regulations regarding permit-required confined spaces by adopting federal OSHA standards. Contact Marcia Holt, DLI, (206) 956-5530.



In late September, the New York State Department of Environmental Conservation (DEC) began circulating a proposed architectural and industrial maintenance coatings (AIM) VOC regulation for comment. The most stringent state AIM regulatory proposal to surface since the cessation of the federal AIM Reg-Neg process, among other things, it calls for reducing the VOC content of general non-flat coatings to 150 grams per liter in 1996; for additional reductions to 100 grams per liter in 1999; and finally 50 grams per liter in 2002. For additional information, contact NPCA's Bob Nelson or Jim Sell at (202) 462-6272.

# Defects in Solvent-Free Organic Coatings Studied by Atomic Force Microscopy, Scanning Acoustic Microscopy, and Confocal Laser Microscopy

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Defects in transparent, solvent-free vinyl ether, and allyl ether maleate-functional coatings have been studied using conventional optical microscopy, scanning electron microscopy, atomic force microscopy, scanning acoustic microscopy, and confocal laser microscopy. Vinyl ether films with different curing rates and allyl ether films with different crosslink densities were prepared, and the number and appearance of defects present in the films were compared. The varying curing rates were achieved by using different initiators, whereas the changes in crosslink density were obtained by using monomers with different functionalities. Defects, such as craters and surface crumpling, were observed. Higher curing rates and increased crosslink densities were both found to promote the formation of defects. Atomic force microscopy, scanning acoustic microscopy, and confocal laser microscopy proved to be useful techniques in the characterization of organic coatings, with the latter being particularly well suited for viewing the interior of transparent coatings.

## INTRODUCTION

The occurrence of defects in organic coatings is an old and well-known problem. A wide variety of defects, including phenomena such as cratering, crumpling, convection cells, and orange peel have been described in the literature.<sup>1-3</sup> In addition to ruining its appearance, the defects often impart poor mechanical and protective properties to a coating film.

The reason for the formation of defects is usually found either in the application of the film, in the interfacial properties between the film and the substrate, or in the curing process. Important factors in this context are the composition of the coating (presence of solvent, particles, etc.) as well as its viscosity and surface tension. The interfacial properties between the film and the substrate also play a role, as well as

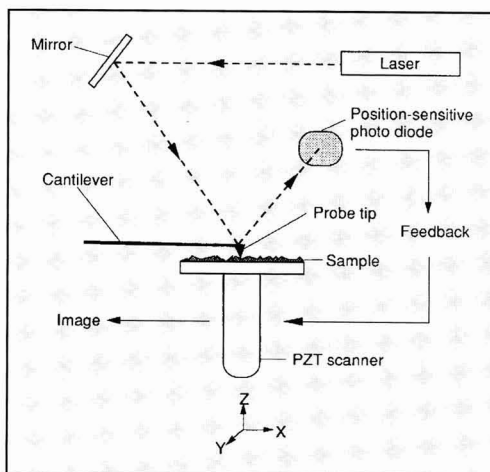


Figure 1—Set-up for atomic force microscopy. The size of the sample and the probe is greatly exaggerated compared to the rest of the figure

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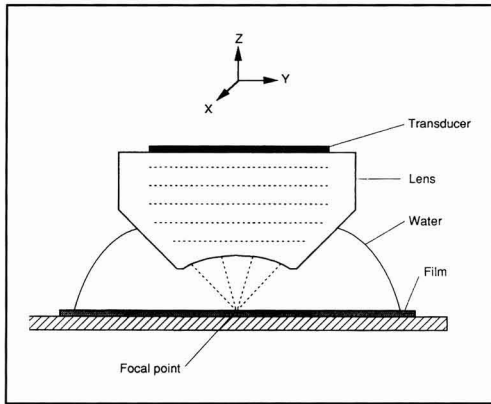


Figure 2—Principle of acoustic microscopy

do the curing conditions.<sup>1,4</sup> Furthermore, if residual stresses build up in the film during cure, the formation of defects may serve as a stress-releasing mechanism.<sup>5</sup>

Most coatings in which defect formation has been studied so far have been solvent-borne, slowly drying (curing) systems. Possible causes for defects have been identified and different remedies suggested.<sup>1-4,6</sup> The advent of new solvent-free coating systems with the possibility of rapid curing now raises the issue to what extent these systems will be prone to defect formation. Furthermore, if defects do occur, will they be of the same kind and due to the same processes as in solvent-borne systems? Another question is what role the special features of these new systems (i.e., the rapid curing) will play in this regard.

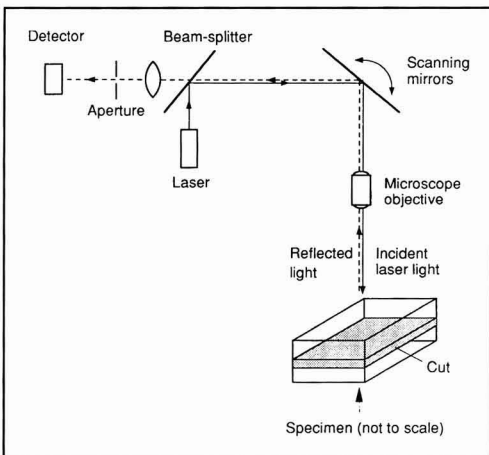


Figure 3—Confocal laser microscope scanner. Two mirrors are used for scanning the laser beam across the specimen (only one is shown here). The size of the specimen is greatly exaggerated compared to the rest of the figure

Atomic force microscopy, scanning acoustic microscopy, and confocal laser microscopy are three recently developed techniques for material characterization. They are all non-destructive and permit the surface of a sample, and in some cases (acoustic and confocal microscopy) its interior, to be examined.<sup>7-9</sup> An atomic force microscope uses a sharp probe sensitive to interatomic forces to gather information about the surface topography. In acoustic microscopy, acoustic waves are employed for the imaging, which means that the contrast will be due to variations in the elastic properties in the specimen. A confocal microscope uses light, as does a conventional light microscope, but offers the advantage of increased resolution and three-dimensional imaging.

In this work, defects present in transparent, solvent-free coatings, have been examined using conventional optical microscopy, SEM, atomic force microscopy, scanning acoustic microscopy, and confocal laser microscopy. The influence of rate of cure and monomer functionality on defect formation have been investigated, and the usefulness of atomic force microscopy, scanning acoustic microscopy, and confocal laser microscopy compared to conventional techniques are demonstrated.

## CHARACTERIZATION METHODS

### Atomic Force Microscopy

Atomic force microscopy (AFM) employs the atomic interaction between a probe and the sample to collect topographic information from the sample surface.<sup>7</sup> A very sharp probe tip is used to limit the interaction area on the surface being studied. The tip is at the free end of a cantilever or known force constant, and the contact force between the sample and the tip is determined by measuring the deflection of the cantilever as the tip makes contact with the surface (see Figure 1). The deflection is measured using a laser and a position-sensitive photodiode. The contact forces between the tip and the surface fall off very quickly with increasing distance from the surface. This keeps the interaction confined to the very end of the probe tip, which allows high resolution imaging. A constant cantilever deflection (constant contact force) is maintained by a feedback circuit constantly adjusting the position of the sample. The sample is raster scanned and the vertical position of the sample is monitored.

### Scanning Acoustic Microscopy

The working principle of the scanning acoustic microscope (SAM) has been thoroughly described by Briggs.<sup>8</sup> The core of the microscope is a single-crystal sapphire lens with a piezoelectric transducer attached to it. When a high frequency is applied to the transducer, ultrasonic waves are generated. These waves pass through the lens and focus on the sample (see Figure 2). Due to the poor propagation of acoustic waves in air, a coupling fluid, e.g., water, is used between the lens and the sample.

A portion of the acoustic energy is transmitted into the sample and a portion is reflected back. This reflected energy is picked up by the lens and the transducer, and the intensity of the reflected signal plotted as a function of position of the

lens above the sample. The lens thus works both as a transmitter and a receiver. To obtain an acoustic image, the lens is mechanically scanned over the sample. By bringing the lens closer to the sample, it is possible to focus the acoustic waves inside the sample, thereby viewing the interior of the specimen.

The main source of contrast in an acoustic microscope is the variation in elastic properties (velocity of sound) in the specimen. This makes it possible to examine specimens which are opaque to light. There is also a possibility of optically detecting invisible structures. The resolution in acoustic microscopy depends on the ultrasonic frequency. Surface imaging may be done at frequencies up to 1.2 GHz which gives a resolution of 1  $\mu\text{m}$ , roughly corresponding to the resolution of an optical microscope. The penetration depth at this frequency is, however, small. At 200 MHz (a frequency which permits focusing below the sample surface), the resolution is 10  $\mu\text{m}$  at the surface and deteriorates as one proceeds downwards. The resolution in acoustic in-depth imaging is thus poor compared to optical microscopy.

### Confocal Scanning Laser Microscopy

The particular confocal microscopy set-up used in this work, as well as the general principle, has been described by Carlson et al.<sup>9</sup> A confocal laser microscope scanner is composed of a conventional optical microscope, a laser light source, a couple of scanning mirrors, and a detector which is attached to a computer (see Figure 3). The sample is irradiated with a narrow laser beam through the microscope objective. The reflected proportion of the incident light is recorded and stored in the computer. An image is obtained by scanning the laser beam over the sample surface with the aid of the mirrors. Since both the incident light and the outgoing light is focused (hence the name confocal microscopy), stray light is reduced and the in-depth resolution becomes very high. This makes it possible to perform optical sectioning of the sample by focusing at different depths, thus ignoring information from the layers above and below the chosen depth. The information from several such sections may be combined to one single three-dimensional image. The three-dimensional image, or rather assembly of data points, can then be digitally processed, e.g., filtered, and different projections calculated.

## EXPERIMENTAL

### Instrumentation

A Perkin-Elmer series 7 DSC was used for the calorimetry experiments. Dynamic mechanical experiments were performed with a Rheometrics RSA 2 dynamic mechanical analyzer. Optical images were taken with an Olympus PMG 3 light microscope using reflected light. The scanning electron microscope was a JEOL JSM 5400. The atomic force microscope used was a SFM-BD2 from Park Scientific Instruments Inc. For the acoustic imaging, an ELSAM Leitz scanning acoustic mi-

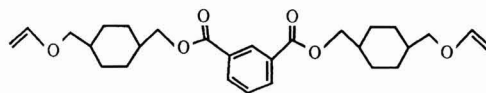


Figure 4—Vinyl ether monomer

croscope was used. The confocal optical images were obtained with a confocal microscope constructed at the Department of Physics IV at the Royal Institute of Technology in Stockholm, Sweden.<sup>9</sup>

### Samples

Two series of samples were studied. The first series was prepared from a vinyl ether monomer (see Figure 4). This substance was made by reacting 1,4-dimethylolcyclohexane monovinyl ether with isophthalic acid chloride as described by Lapin et al.<sup>10</sup> The monomer was cationically cured with thermal initiation. Two different onium salt initiators,<sup>11</sup> phenacyl tetramethylene sulfonium hexafluoro antimonate (1) and *a*-methyl benzyl tetramethylene sulfonium hexafluorophosphate (2) were used. One percent per weight of (1) gave full cure in 0.5 min at 115°C as measured by DSC, while 0.1% per weight of (2) gave full cure in 10 min at the same temperature. Films of the monomer with initiator (1) and (2) in the concentrations mentioned were prepared on glass substrates and cured at 115°C for 45 min. In this way, fast cured and slowly cured vinyl ether samples were obtained. The thickness of the films was approximately 30  $\mu\text{m}$ .

The second series was prepared from three different allyl ether maleate-functional monomers. They had similar structures but differed in functionality, i.e., the number of reactive groups per molecule. The structure of the monomers is shown in Figure 5. Monomer synthesis has been described by

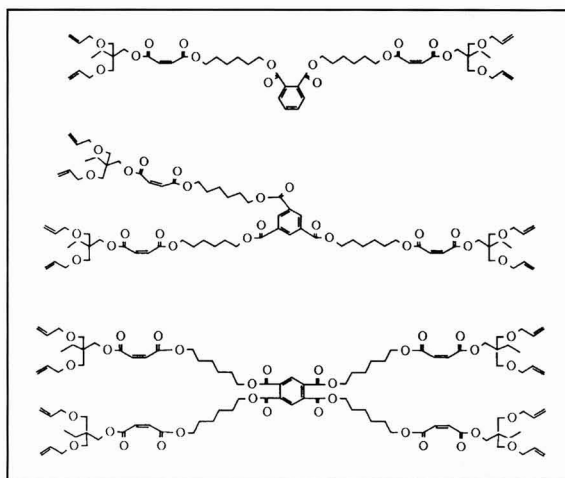


Figure 5—Allyl ether maleate-functional monomers with different functionalities

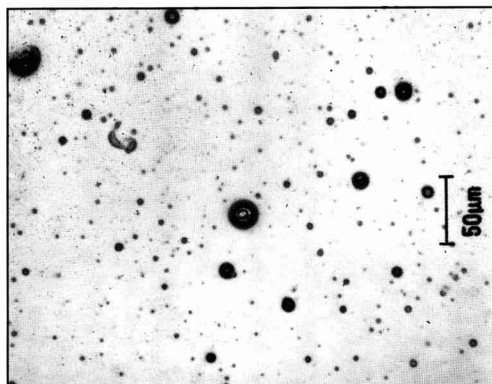


Figure 6—Optical microscopy image of the fast cured vinyl ether sample

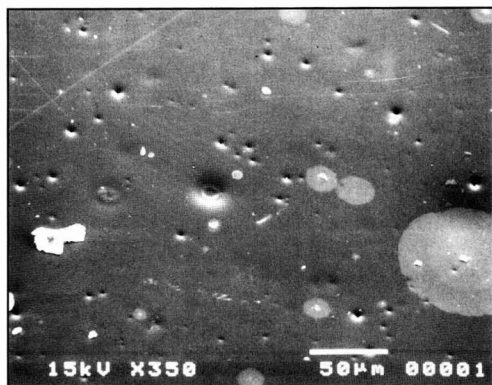


Figure 7—SEM image of the fast cured vinyl ether sample

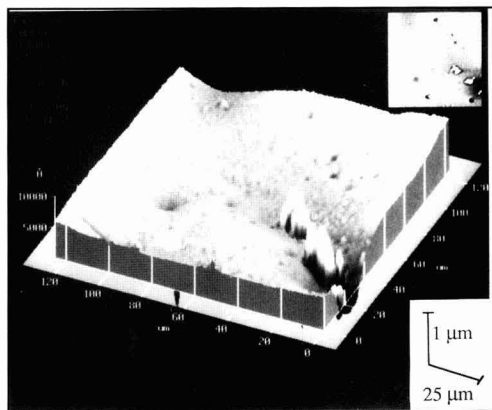


Figure 8—Atomic force microscopy image of the fast cured vinyl ether sample. The protruding objects in the lower right corner are artifacts due to the sample sticking to the probe

Johansson et al.<sup>12</sup> A thermally initiated radical curing procedure, reported to give full cure in about 50 min at 80°C,<sup>12</sup> was used. Monomer with a mixture of initiators was applied on glass substrates and cured at 80°C for 60 min. This gave three samples with different crosslink densities. All films were roughly 30 µm thick.

### Atomic Force Imaging

For the atomic force imaging, a representative area of the sample was chosen with the aid of an optical microscope. The probe tip was then lowered so as to come in contact with the sample. Subsequently, scans were run, and the information collected transformed into three-dimensional pictures with the aid of a computer.

### Acoustic Imaging

In a typical experiment, the specimen was placed under the microscope and viewed optically, using the optical microscope built into the acoustic microscope. This helped in choosing the imaging area, as well as in roughly focusing the microscope. Then, the acoustic lens was put into place, and the coupling fluid (water) applied between the lens and the sample. Subsequently, the distance between the lens and the sample was adjusted until the acoustic waves came into focus on the surface. Surface imaging was then performed with an ultrasonic frequency of 1.2 GHz and in-depth imaging with a frequency of 200 MHz.

### Confocal Imaging

For the confocal imaging, the specimen was placed under the microscope and a suitable area chosen by manual inspection of the surface. Then a number of sections, each one about 1 µm thick, were recorded so as to cover the whole film thickness. The assembly of sections, together creating a three-dimensional image, was digitally gradient filtered and converted into a vector representation. Finally, a surface-shadowed projection from an angle of 40° was calculated.

## RESULTS AND DISCUSSION

### Characterization Techniques

A representative optical image of a fast-cured vinyl ether film is shown in Figure 6. From this picture it is evident that there are some kind of essentially circular structures present in the film. It is difficult, however, to discern the nature of the structures. They could be bubbles, particulate inclusions, or simple holes or depressions. Since the film is transparent, it is equally difficult to say at which level they are situated, i.e., inside the film or at the surface.

A typical scanning electron microscopy (SEM) image of the same film (see Figure 7) provides additional information on the surface structure. The presence of various kinds of holes, dimples, and craters is clearly visible. Apart from that, the surface appears smooth. SEM is well reputed as being a highly surface sensitive characterization technique. An SEM image is, however, not an exact reproduction of the surface but rather an indirect representation, susceptible to changes

in the position and the angle of the sample.

Atomic force microscopy is a characterization technique that allows the physical topography of a sample to be directly visualized. A typical AFM picture of the fast cured vinyl ether film (presented in *Figure 8*) confirms the information provided by the SEM picture. Again holes, depressions, and craters are visible, as is a certain amount of waviness in the surface. It may be noted that this unevenness did not appear in the SEM or optical microscopy pictures.

In regards to sub-surface features (e.g. possible inclusions or bubbles), SEM and atomic force microscopy naturally give little information. In order to image the interior of the film, one is either forced to employ techniques like cryogenic cutting or metallographic polishing, or to use another method of characterization.

Scanning acoustic microscopy is a method that offers the possibility of in-depth as well as surface imaging. A problem with acoustic in-depth imaging is, however, the limited resolution. Viewing a 30  $\mu\text{m}$  thick film with a resolution of, at best, 10  $\mu\text{m}$  makes it difficult to tell from what depth the contrast occurs. An SAM image of the surface of the fast cured vinyl ether sample is presented in *Figure 9*. Images from the interior of the film showed similar structures but with a lower resolution. The existence of circular structures at the surface is again indisputable, as is the irregularity of the surface. The unevenness observed in the atomic force experiments is thus supported by the scanning acoustic microscopy.

Confocal laser microscopy is also a technique which enables the interior of a specimen to be viewed provided that it is transparent to light. A confocal laser microscopy image of the fast cured vinyl ether film is shown in *Figure 10a*. Here, multiple sections all the way from the top to the bottom of the film, have been combined, the data filtered to enhance borderlines, and a surface-shadow projection calculated. The computer-generated surface shading simulates a light source illuminating the object's surface. The image is presented at a slight angle, which is why the edge is visible at one end. The unevenness of the surface is evident, as is the presence of craters, dimples, and holes. The reason why the unevenness is so pronounced as compared to the AFM picture in *Figure 8*, is the high in-depth resolution of the confocal microscope in combination with the digital image processing. Together, these two features render the confocal microscope extremely sensitive to surfaces.

To detect the possible presence of structures inside the film, another projection was made (see *Figure 10b*). In this picture, the sections corresponding to the top and bottom film surfaces have been removed. In other words, only the central section of the film is viewed. Again the image is

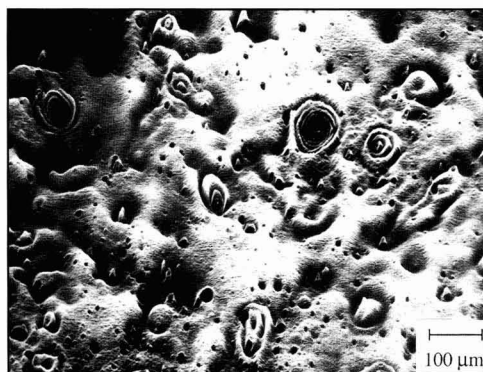


Figure 9—SAM image of the fast cured vinyl ether sample

presented at a slight angle, leading to the edges being visible. Clearly, there is not much contrast in this part of the film. A projection of the bottom surface (not shown here) revealed a very slight unevenness and no craters. Therefore, it may be concluded that the structures seen in *Figure 6* are indeed defects located mainly at the top surface of the film.

An overview of the different characterization techniques used, as well as the particular information provided by each method, is presented in *Table 1*.

### Influence of Curing Rate on Defect Formation

The slowly cured vinyl ether sample presented in *Figure 10c* resembles the fast-cured sample (*Figure 10a*), but with less pronounced defects. The types of structures, i.e., craters, depressions, and general crumpling, are the same, but they became more pronounced with increased curing rate.

Craters and dimples, as well as bumps, sinks, and a crumpled surface, are well-known in solvent-borne systems.<sup>2,4</sup> The craters have been attributed to inhomogeneous curing, i.e., formation of gel particles and subsequent variations in surface tension around the particle.<sup>4</sup> Surface crumpling has been explained by the formation of a solid skin on the film in combination with surface tension gradients or convection cells.<sup>2</sup>

It is possible that the defects in the coatings described in the present work were caused by similar processes, in spite of the fact that the coatings were prepared without solvents. There is also a difference in size which has to be accounted for; the defects observed in the solvent-free coatings are only

Table 1—An Overview of the Characterization Techniques

Technique	Source of Contrast	Sample Requirements	Penetration Depth/In-Depth Resolution
Optical microscopy .....	Refractive index	Transparent	Surface and bulk/ = 10 $\mu\text{m}$
Scanning electron microscopy .....	Surface topography	Resist vacuum	Surface
Atomic force microscopy .....	Surface topography	Not to soft	Surface
Scanning acoustic microscopy .....	Density/elastic modulus	Resist water	Surface and bulk/10 $\mu\text{m}$
Confocal optical microscopy .....	Refractive index	Transparent	Surface and bulk/1 $\mu\text{m}$



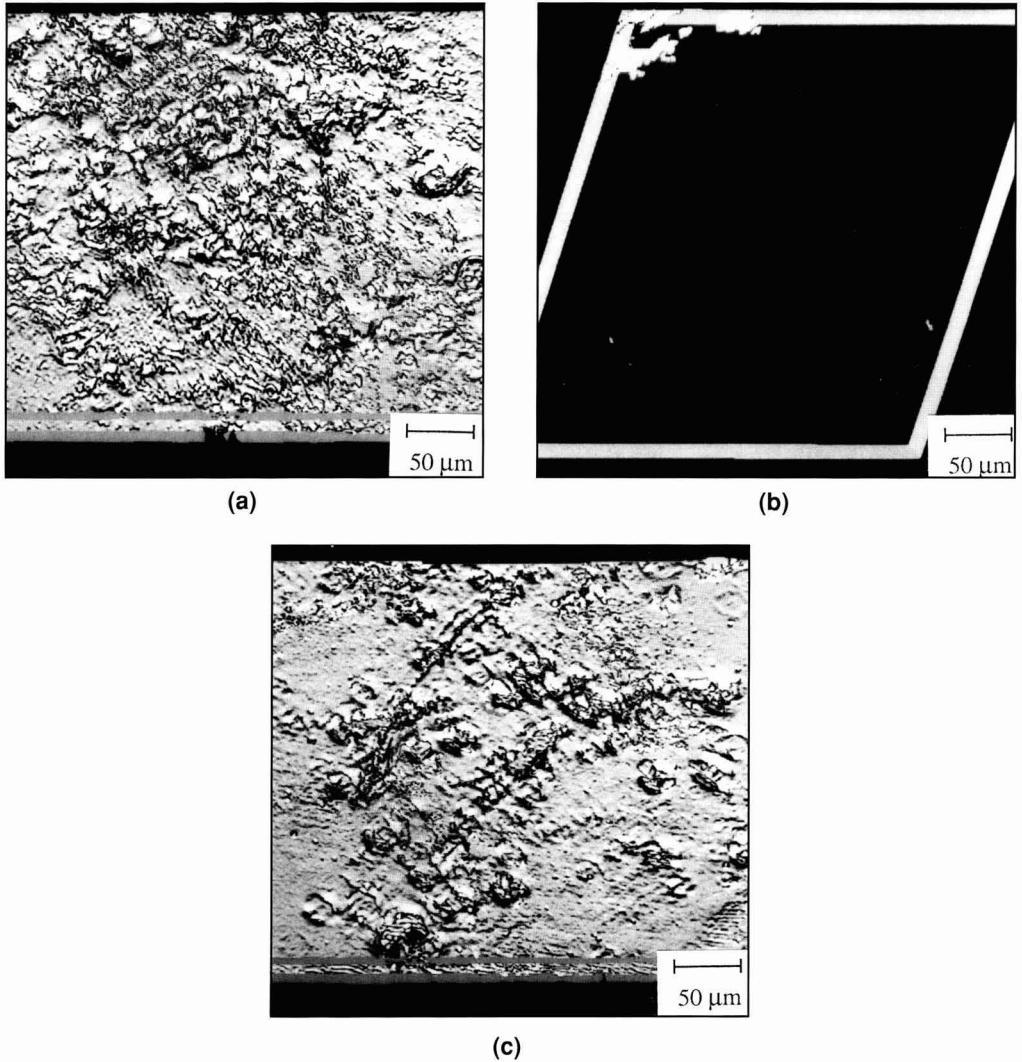


Figure 10—Confocal laser microscopy image of the (a and b) fast and (c) slowly cured vinyl ether samples. (a) and (c) are surface-shaded projections at an angle of 40°. In image (b) the top and bottom surfaces are not included, i.e., the image presents the middle 15  $\mu\text{m}$  of the film

one-tenth the size of the ones described in the solvent-borne coatings. Clearly, however, there is a difference in number and intensity of defects between the films cured at different rates, so the processes responsible for the defects were somehow related to the rate of cure. On the other hand, the shape, size, and general appearance of the defects seem quite independent of curing speed.

#### Influence of Monomer Functionality (Crosslink Density) on Defect Formation

Confocal images of coatings prepared from three allyl ether maleate functional monomers with different functionalities are presented in Figure 11. The most densely crosslinked film (Figure 11a) exhibits pronounced cratering

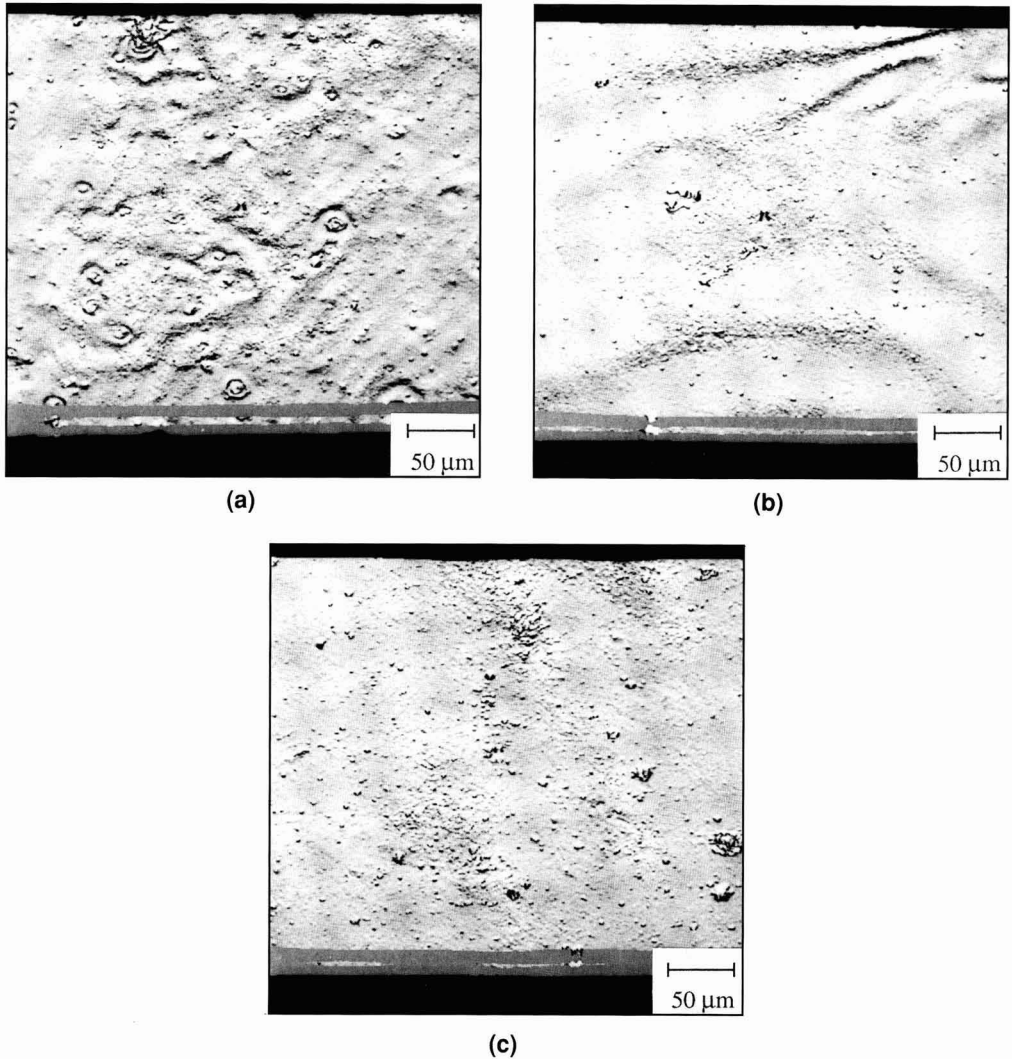
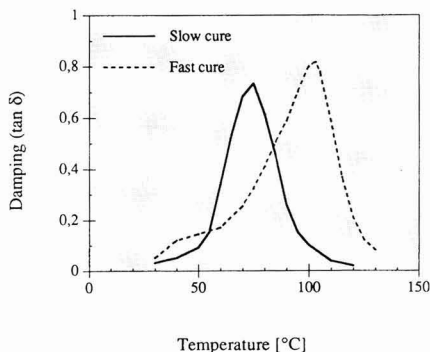


Figure 11—Confocal optical microscopy images of (a) the most crosslinked, (b) the less crosslinked, and (c) the least crosslinked sample. The images are surface-shaded projections at an angle of  $40^\circ$

and a significant amount of surface crumpling. The less densely crosslinked film (Figure 11b) shows some surface crumpling but no cratering, while the least crosslinked film (Figure 11c) is all but flat (the small spots present in all three images are dust particles).

Again, the defects (i.e., crumpling and cratering) are possible effects of gel particles and skin formation in com-

bination with surface tension gradients or convection cells. The disparity between the films with different crosslink densities is, however, greater than in the case of varying curing rates, since not only the intensity but also the nature of the defects changed. Therefore, there seems to be a critical crosslink density above which craters occurred. The surface crumpling, however, appears to be a more general



**Figure 12—Damping (tan δ) versus temperature for the slow and fast cured vinyl ether films measured using dynamic mechanical analysis at a frequency of 1Hz and a strain of about 0.1%**

phenomenon—the degree of crumpling rising with increased crosslinking.

### Comparability of Systems

A question that naturally arises is whether there is any connection between different curing rates and different crosslink densities. It is natural to assume that a higher density of functional groups gives a more intense reaction, and therefore a higher curing rate. This is also the case for the three allyl ethers.<sup>12</sup> Consequently, in this case increased monomer functionality is compared to an increase in curing rate.

With dynamic mechanical analysis, looking at the width of the damping peak at the glass transition temperature, it is possible to obtain a measure of the crosslink density.<sup>13</sup> In this way the difference between the allyl ether samples was verified.<sup>12</sup> Such an analysis, performed on the fast and the slowly cured vinyl ether samples, is presented in Figure 12. The difference in the width of the damping peak, as well as the higher glass transition temperature, reveals that the fast-cured sample had a higher crosslink density than the slowly cured one. A higher rate of cure, as achieved in this work for this vinyl ether, was thus accompanied by an increase in crosslink density.

Increased curing rate and increased monomer functionality should therefore affect the formation of defects in similar ways. This is indeed what is observed and in both cases the defects became more pronounced. The apparent variation in nature of the defects with crosslink density, but not with rate of cure, may be due to the limited comparability of the two sets of samples. The crosslink densities, as measured by dynamic mechanical analysis, are difficult to compare since the width of the damping peak depends not only on the crosslink density but also on the chemical structure. Furthermore, curing rates of vinyl ethers are generally higher than those of allyl ethers; the slowly cured vinyl ether thus cured much faster than the most crosslinked allyl ether. It is also quite possible that the formation of defects was influenced by other factors in addition to curing rate and crosslink density, and that these forces varied between the two sets of samples.

### CONCLUSIONS

Surface defects, such as surface crumpling and formation of craters, occurred when solvent-free vinyl ether and allyl ether maleate functional coatings were cured. The defects were similar in appearance but smaller in size compared to defects in solvent-borne coatings described in literature.

Increased rate of cure produced more pronounced defect, but did not change their appearance, i.e., both craters and surface crumpling occurred independently of curing rate. Increased crosslink density (monomer functionality) made the defects more pronounced and changed their appearance. Hence, surface crumpling was present to a various extent at all crosslink densities, whereas craters occurred only above a certain level of crosslinking. Increased curing rates and increased monomer functionality thus promoted the formation of defects in similar ways.

Atomic force microscopy and scanning acoustic microscopy were found to be useful methods for examining the surface of coatings, and showed to be more sensitive to surface roughness than either conventional optical microscopy or SEM.

Confocal laser microscopy proved to be a versatile tool for examining the surface as well as the interior of transparent coatings. The method offers very high surface contrast in combination with good in-depth resolution, features which made it particularly well-suited to locate and identify the defects present in the films.

### ACKNOWLEDGMENT

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# Epoxidized Polybutadiene: A Novel Prepolymer for Cationically UV-Curable Coatings

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Epoxidized polybutadiene was evaluated as a reactive prepolymer for cationically curable photosensitive compositions. Various epoxy or vinyl ether functional reactive diluents were used as viscosity reducers. Compositions with an average content of 5 mmol.g<sup>-1</sup> of polymerizable functions were submitted to viscosity measurements, and their reactivity under UV-irradiation was examined by UV and/or IR spectroscopy. The initiator photolysis can be monitored easily and adequate exposure times were determined by simple kinetic treatment. The polymerization of epoxy monomers was driven to high conversion (over 95%) provided that the film was submitted to a soft thermal post treatment. Coatings of high flexibility and showing good adhesion to aluminum, steel, and glass were obtained by adequate adjustment of the formulations composition. The content in reactive diluent was kept to a low level without loss of curing ability.

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## INTRODUCTION

UV-initiated cationic polymerization of epoxy and vinyl ether derivatives appears to be a valuable alternative to the UV-curing of coatings by a free radical process.<sup>1</sup> By comparison to acrylic compositions, cationically curable systems exhibit several advantages arising from the following features: compositions are of low odor and low viscosity, the process is less sensitive to oxygen, and cured films show good adhesion and contain lower amounts of extractable.

However, serious restrictions to their development for coatings applications lie in the limited number of reactive prepolymers and diluents available on the raw materials market. Considering the higher cost and the insufficient quantity of available data for orienting the development of new compositions, it is easy to explain the reluctance of formulators to change to cationic UV-curing. Additionally, the thermal post treatment for achieving complete cure and stress relaxation makes the curing process more complicated.

Several efforts have been made in recent years to offer new reactive diluents: novel vinyl or propenyl ethers of high reactivity are now available.<sup>2,3</sup> Concerning reactive prepolymers, it appears that besides epoxy functional bisphenols and novolacs as hydrocarbon prepolymer representatives, and epoxy functional polysiloxanes as soft but very particular prepolymers, there is an evident lack of choice. Polyurethanes with vinyl ether end-groups have been reported recently.<sup>4</sup> The present contribution focuses on the study of compositions based on epoxidized polybutadiene associated with conventional reactive diluents. This objective is to evaluate this prepolymer which may provide the formulator with a new starting material for soft coatings.

## EXPERIMENTAL

### Materials

Epoxidized polybutadienes Polybd 605 and Polybd 600 and two epoxy functional reactive diluents, limonene dioxide (EPO1) and 1,2-epoxydodecane (EPO4), are commercial grades from Elf-Atochem. Obtained from Union Carbide were 3,4-epoxycyclohexylmethyl, 3',4'-epoxycyclohexane-

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## Equipment

UV spectra were obtained from a Uvikon 930 spectrophotometer. Infrared spectra were obtained from Perkin Elmer spectrophotometers (FTIR 2000 or IR-882). Dynamic viscosities were measured on a Haake Rotovisco RV100. The samples were submitted to static UV light (900 watt xenon Hanovia lamp supplied with a 40 A current intensity) or to dynamic UV irradiation (mimicry system of Primarc—dose control by Compact radiometer).

## Characterization of Cured Coatings

Coatings adhesion to substrates was measured by cross-cut tape tests according to ASTM D 3359 (excellent adhesion is noted as 5B and poor adhesion is noted as 0B). The gloss of the coatings was measured by specular reflection at 20° (Rhopoint from Novogloss). Pencil hardness was determined according to ASTM 3363, with Faber Castell 9000 pencils covering the range 6B-6H. Flexibility was assessed by T bend tests. It is quantified by the minimum number of half-bends measured to avoid cracking of the coating. Solvent resistance was measured by the methyl ethyl ketone double rubs test.

## RESULTS AND DISCUSSION

The epoxidized polybutadiene used for this study can be represented roughly by the general structure shown in *Scheme 1*. It results from the peracid oxidation of hydroxytelechelic polybutadiene. As a consequence of the butadiene polymerization process, the average hydroxy functionality is actually slightly above 2.<sup>5</sup> Two grades are commercially available. They essentially differ by the functionality and by the viscosity of the polymers (*Table 1*).

Compounds containing hydroxy functionalities are known to participate in the polymerization process as transfer agents. Polyols can also act as crosslinkers connecting the chains by step reactions catalyzed by acids. The presence of hydroxy groups on the tested polybutadienes enables this additional curing process to take place.<sup>6</sup> Low molecular weight or polymeric additives with hydroxy functionality permit the properties of the cured material to modify. They take place in the network by the epoxy alkoxy addition reaction and are generally chosen and used for their plasticizing effect.<sup>7</sup>

**Scheme 1—Structure of the reactive materials**

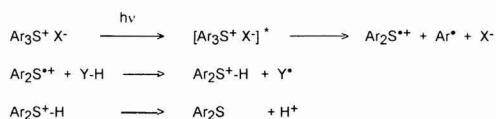
carboxylate (EPO2), and cyclohexene oxide (EPO3). Vinyl ethers were obtained from ISP triethyleneglycoldivinylether (VE1), 1,4-butanediol monovinylether (VE2), and 1,4-bis-vinylloxymethylcyclohexane (VE3). Photoinitiators FX512 (3M) and UVI-6974 (Union Carbide) are 50 wt% solutions of triarylsulfonium salts in  $\gamma$ -butyrolactone and propylene carbonate, respectively. The content of photoinitiators in the compositions is expressed in wt% of the commercial solution. The formulated compositions were stored in dark rooms and allowed to rest for 24 hr before use. Steel and aluminum panels (QD36 and A36) were purchased from Q-Panel. Liquid films to be irradiated were coated on the desired substrate with calibrated bar coaters. Transparent plates were used for spectroscopic monitoring of the reactions (Suprasil for UV and NaCl for IR measurements). A non-ionic fluorinated surfactant (FC430 from 3M) was used as wetting agent.

**Table 1—Main Structural Characteristics of Epoxidized Polybutadienes and Reactive Diluents Used for This Study**

Reactive Material	Functionality Type/Number	Molecular Weight (g.mol <sup>-1</sup> )	Content in Reactive Functions (mmol.g <sup>-1</sup> )	Viscosity 25°C (mPa.s)
Polybd 605 .....	epoxy -	4400 <sup>a</sup>	3.8	25 000 <sup>b</sup>
Polybd 600 .....	epoxy -	4000 <sup>a</sup>	2.2	5 500 <sup>b</sup>
EPO1 .....	epoxy -2	168	11.9	—
EPO2 .....	epoxy -2	252	7.94	350
EPO3 .....	epoxy -1	98	10.2	—
EPO4 .....	epoxy -1	184	5.43	5.8
VE1 .....	vinyl ether -2	202	9.90	2.7
VE2 .....	vinyl ether -1	116	8.62	5.4
VE3 .....	vinyl ether -2	196	10.2	5.0

(a) Weight-average molecular weight deduced from GPC analysis with polystyrene calibration.

(b) Significant differences in the degree of branching may account for the higher viscosity of Polybd 605 with regard to Polybd 600.



Scheme 2—Photodecomposition of triaryl sulfonium salts

To permit fast curing, mixtures of epoxy functional prepolymers and diluents must contain oxirane groups at a sufficiently high level. A generally admitted rule recommends epoxy contents greater than  $5 \text{ mmol.g}^{-1}$ .<sup>8</sup> The addition of reactive diluents containing cationically reactive epoxy or vinyl ether functions allows the adjustment of this prime parameter.

The viscosity is reduced by this means, while the properties of the cured film may also be controlled to some extent by the nature and amount of the reactive diluents. Adhesion, toughness, solvent resistance, and weatherability can be very sensitive to this factor.<sup>9</sup> We used the mono- and difunctional reactive diluents depicted in *Scheme 1*. Their main characteristics appear in *Table 1*.

### Viscosity of the Formulations

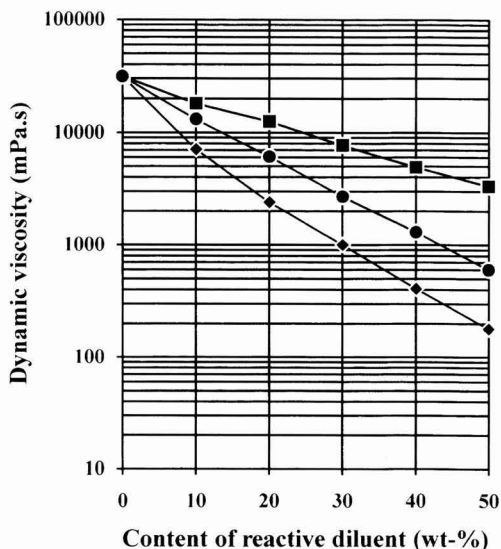
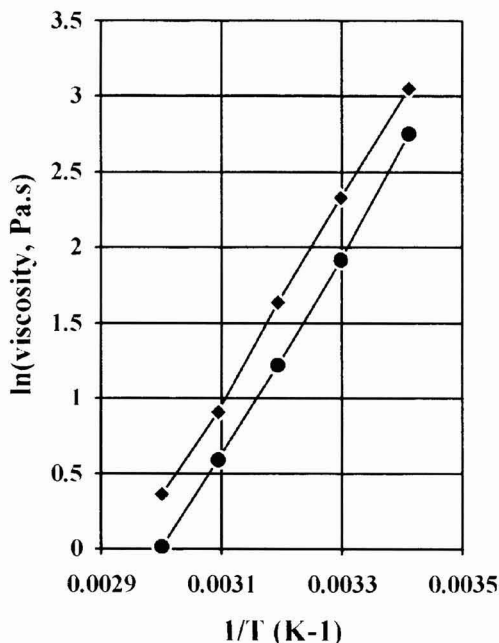
To determine the viscosity range covered by formulations containing a sufficient amount of reactive functions, we blended the prepolymers with various reactive diluents so as to obtain the value of  $5 \text{ mmol.g}^{-1}$ . The viscosities measured at  $25^\circ\text{C}$  are shown in *Table 2*. The low content of epoxy groups in Polybd 600 requires high proportions of reactive diluents and the resulting viscosities range from 0.3 to 1.4 Pa.s. For Polybd 605, the amount of added diluent generally is kept below 20 wt%.

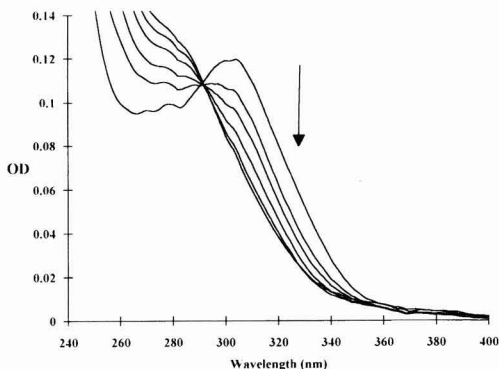
The changes in viscosity of mixtures containing Polybd 605 and a proportion of reactive diluents ranging from 0 to 50 wt% are plotted on the curves of *Figure 1*. VE1 appears to be a very efficient reducer, the viscosity being cut by a factor of 13 with only 20 wt% of vinyl ether added. At a dilution of 50 wt%, the viscosity of the mixture blended with EPO2 still extends 3 Pa.s.

Alternatively, reduction of the viscosity can be obtained at low content in reactive diluent by moderate heating of the formulations. The temperature dependence of the dynamic viscosity was also examined with compositions C1 and C4. The corresponding plots of *Figure 2* indicate that, for temperatures ranging from 20 to  $60^\circ\text{C}$ , the Arrhenius law is obeyed with an activation energy  $E^{\#}$  of 795 and  $800 \text{ J.mol}^{-1}$  for C1 and C4, respectively. Although the two solvents are significantly different in their structures, the values of  $E^{\#}$  are very close. They can be used to reasonably predict the viscosity of the other mixtures at a given temperature.

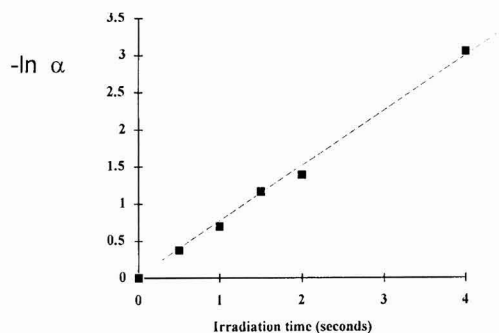
### Reactivity of Photosensitive Compositions

In this chemistry, onium salts initiate the curing process by generating very strong Brønsted acids upon photodecomposition. The main steps of this reaction are depicted in *Scheme 2*.<sup>10</sup> We prepared the photosensitive formulations by

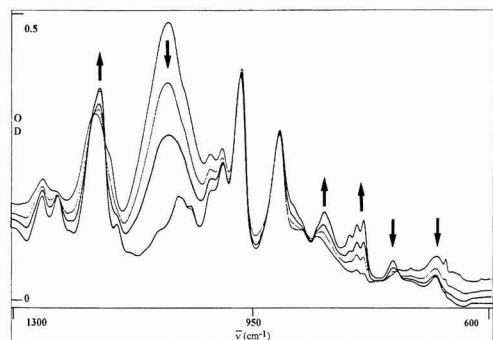
Figure 1—Dynamic viscosity at  $25^\circ\text{C}$  of various blends Polybd 605-reactive diluent: EPO1 (●), EPO2 (■), VE1 (◆)Figure 2—Arrhenius plot of temperature dependence of the dynamic viscosity: mixtures of Polybd 605 and reactive diluent EPO1 (●), VE1 (◆) containing  $5 \text{ mmol.g}^{-1}$  of reactive functions



**Figure 3—Changes in the UV spectra of a photosensitive composition (Polybd 605 71 wt%/EPO2 29 wt%/FX512 4 phr) upon irradiation under the Hanovia lamp. Irradiation time = 0, 0.5, 1, 2, 4, and 8 sec. Wet thickness 25 μm**



**Figure 4—Plot of variation of  $-\ln \alpha$  versus time of a photosensitive composition (Polybd 605 71 wt%/EPO2 29 wt%/FX512 8 phr) upon irradiation under the Hanovia lamp. Wet thickness 10 μm**



**Figure 5—Changes in infrared spectra for the photosensitive composition (Polybd 605 71 wt%/EPO2 29 wt%/FX512 4 phr) upon irradiation under the Hanovia lamp. Wet thickness 10 μm. Irradiation time: 0, 0.5, 1, 2 sec**

using commercial solutions of mixed triaryl sulfonium salts associated with counter anions of poor nucleophilicity, namely hexafluorophosphate ( $\text{PF}_6^-$ ) for FX512 initiator, from 3M, and hexafluoroantimonate ( $\text{SbF}_6^-$ ) for initiator UVI-6974, from Union Carbide.

The absorption of light at the maximum wavelength (305 nm) was examined by UV-spectroscopy of an epoxy formulation containing 4 wt% of FX512. The values measured for films of thickness between 10 and 50 μm were in the range of 0.20-0.75 units of absorbance. Screening effects that limit light penetration in the film depth are thus acceptable at this concentration. Although the films are affected by some bleaching upon irradiation, direct and accurate measurements of the apparent rate constant in the reactive formulations proved to be impossible, since the photoproducts absorb also in the band of the initiator. The presence of an isobestic point at 290 nm in the spectra of UV-irradiated compositions tends to indicate that the photodecomposition follows an unperturbed process (Figure 3). Some corrections can be made with the assumption that the decomposition is achieved when no significant change is observed after additional exposures. The molar fraction  $\alpha$  of unreacted photoinitiator at instant  $t$  is then given by the relationship:

$$\alpha = \frac{[\text{DO}_{305 \text{ nm}}]_t}{[\text{DO}_{305 \text{ nm}}]_0} = \frac{[\text{DO}_{305 \text{ nm}}]_t - [\text{DO}_{305 \text{ nm}}]_{\infty}}{[\text{DO}_{305 \text{ nm}}]_0 - [\text{DO}_{305 \text{ nm}}]_{\infty}}$$

As indicated by the linearity of the curve  $-\ln \alpha$  plotted against time (Figure 4), the decay of the initiator concentration follows an expected first order kinetics. The lifetime measured under the Hanovia xenon lamp for compositions with 4 wt% of initiators and film thickness ranging from 10 to 50 μm was about one second. The exposure durations were thus fixed at 20 times this value (20 sec and more) to ensure complete photolysis of the initiator in the cured samples.

Changing the initiator concentration may also cause some modifications of the nature and the amounts of the aromatic photolysis products.<sup>11</sup> These are the results of recombination of radicals, and the change in concentration of these species usually affects the products' distribution. As a consequence, the UV-spectra can be greatly modified and internal filter effects must be checked for the various concentrations to be used. At low concentration of onium salts (2-4%) and for reasonable thicknesses (5-50 μm), the first order kinetic law was obeyed as a consequence of the low initial optical density.

Because of the specific feature of photoinitiated cationic polymerizations where the ratio of the propagation rate to the photoinitiation rate is small in comparison to acrylic based free radical systems, special care must be paid to the reactivity control of both initiator and monomers in the two consecutive steps. IR spectroscopy proves to be a very powerful tool for this purpose.<sup>12,13</sup> The 700-1200  $\text{cm}^{-1}$  region of the spectra recorded from a photosensitive epoxy formulation (Figure 5) contains information concerning the various processes taking place upon irradiation. It is possible to monitor the decrease of sulfonium salt concentration with the band at 845  $\text{cm}^{-1}$ , the decrease of epoxy function with the band at 790  $\text{cm}^{-1}$ , and the resulting increase of the ether band at 1100  $\text{cm}^{-1}$ . The kinetic results obtained by UV spectroscopy were confirmed for low to moderate contents in photoinitiator

concentration with half-lives of about one second under the Hanovia lamp.

Irradiating the samples with a given dose in one exposure, or several smaller doses separated by a few minutes for recording the spectrum, was shown to have little effect on the conversion. The two curves showing the progress of the epoxy polymerization initiated under a low power laboratory source can be compared in *Figure 6*. The limited differences can be explained by the small propagation rate at room temperature and the small interval of time separating the measurements. The addition of the doses enables studying the dose-conversion relationship with a reasonable number of experiments. The kinetic results presented in this article were obtained by accumulating doses on one original sample.

Increasing the photoinitiator content in compositions based on Polybd 605 and EPO2 was shown to increase the initial rate of epoxy polymerization. The values of  $(R_p)_0$  calculated from the slopes of the curves in *Figure 7* are 0.11, 0.24, and 0.48  $\text{mmol}\cdot\text{g}^{-1}\cdot\text{s}^{-1}$  for initiator concentrations of 2, 4, and 8 wt%, respectively. The initial enhancement of the extent of polymerization does not change the ultimate conversion reached. In the composition containing the greatest amount of FX512, the limiting conversion of epoxy groups was shown to reach a value of 95%, almost unchanged with regard to the value measured with 4 wt% of initiator. The compositions tested for their properties as cured coatings were prepared with 4 wt% of FX512.

We have already mentioned the somewhat low rate of polymerization of the epoxy groups. The benefit of post thermal treatments to achieve efficient curing of the liquid films is well-known. Polybutadiene and its derivatives are, however, subject to some degradation upon heating.<sup>14</sup> The conditions recommended for aromatic or cycloaliphatic epoxy materials (120°C and above<sup>15</sup>) were expected to be detrimental to our formulations. The extent of the post polymerization under moderate thermal treatment was thus examined in order to determine the proper temperature and time of activation. Heating the samples at 100°C after a six-second exposure under the Hanovia lamp was shown to cause limited darkening of the film in some cases. The higher sensitivity of films containing VE1 indicates that the

**Table 2—Dynamic Viscosity at 25°C of Various Blends of Epoxidized Polybutadienes and Reactive Diluents Containing 5  $\text{mmol}\cdot\text{g}^{-1}$  of Reactive Functions**

Composition	Prepolymer (amount - wt%)	Reactive Diluent (amount - wt%)	Viscosity at 25°C (Pa.s)
C1	Polybd 605 - 85.2	EPO1 - 14.8	10.68
C2	Polybd 605 - 65.4	EPO2 - 34.6	8.40
C3	Polybd 605 - 81.2	EPO3 - 18.8	3.62
C4	Polybd 605 - 80.3	VE1 - 19.7	3.12
C5	Polybd 600 - 71.1	EPO1 - 28.9	1.06
C6	Polybd 600 - 44.8	EPO2 - 55.2	1.29
C7	Polybd 600 - 65.0	EPO3 - 35.0	0.29
C8	Polybd 600 - 63.6	VE1 - 36.4	0.28

reactive diluent may have a determining influence on the thermal behavior of the coatings. After 10 min at 130°C, photocatalytically cured films of unblended Polybd showed no sign of degradation. Thermal post treatments at 80°C for 20 min offered an acceptable compromise yielding a conversion of epoxy functions exceeding 95 mol% (*Figure 8*). IR spectra also indicate that the initiator is thermally decomposed but slowly under the mentioned conditions.

### Properties of UV-Cured Films

The properties of polybutadiene based films cured as clear coats were evaluated with Polybd 605. This epoxidized polybutadiene exhibits high content in polymerizable functions and gives reactive blends of convenient viscosity. Polybd 600 appeared better suited for pigmented or filled formulations, the properties of which are strongly dependent on the presence of the inorganic filler and on the relatively high amount of reactive diluent.

The strong influence of the nature and content of epoxy functional diluents in the compositions can be analyzed from the results shown in *Table 3*. As expected, long chain monofunctional epoxide EPO4 yields tacky films. On increasing the diluent concentration, the strong plasticizing effect overcomes the enhancement of epoxy group concentration and the resulting effects on reactivity and network structure. On the other hand, reactive blends containing di-

**Table 3—Tackiness<sup>a</sup> and Adhesion<sup>b</sup> of Polybd 605 Compositions<sup>c</sup> Blended with Epoxy Reactive Diluents after Irradiation<sup>d</sup>**

Reactive Diluent (wt%)	Concentration of Epoxy Functions ( $\text{mmol}\cdot\text{g}^{-1}$ )	Thermal Post-Treatment at 70°C				
		0 min	5 min	10 min	20 min	30 min
EPO4 (25)	4.2	+3B	+3B	+3B	+4B	+4B
EPO4 (50)	4.6	+ <sup>e</sup>	+ <sup>e</sup>	+ <sup>e</sup>	+ <sup>e</sup>	+ <sup>e</sup>
EPO4 (74)	5.0	+ <sup>e</sup>	+ <sup>e</sup>	+ <sup>e</sup>	+ <sup>e</sup>	+ <sup>e</sup>
EPO1 (7)	4.3	+3B	+3B	+4B	+4B	+4B
EPO1 (15)	5.0	+2B	+2B	+2B	-2B	-2B
EPO1 (22)	5.6	-1B	-1B	-1B	-2B	-2B
EPO2 (25-50)	4.8 - 5.9	-1B	-1B	-1B	-1B	-1B

(a) Wet thickness 25  $\mu\text{m}$  on glass plates; tacky (+); tackfree (-).

(b) Cross-cut tape test.

(c) Photoinitiator: 4 phr of FX 512.

(d) UV exposure: 33 sec under the Hanovia lamp.

(e) Adhesion not measurable.



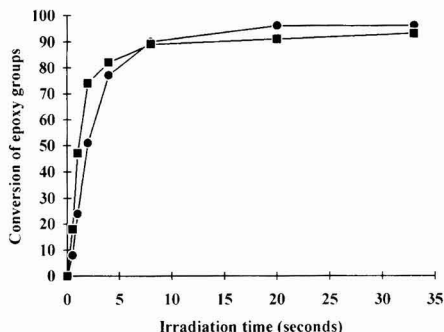


Figure 6—Comparison of the conversion of epoxy groups under UV exposure delivered as single dose (■) or accumulated increments (●) for a photosensitive composition (Polybd 605 71 wt%/EPO2 29 wt%/FX512 4 phr) upon irradiation under the Hanovia lamp. Wet thickness 25 μm

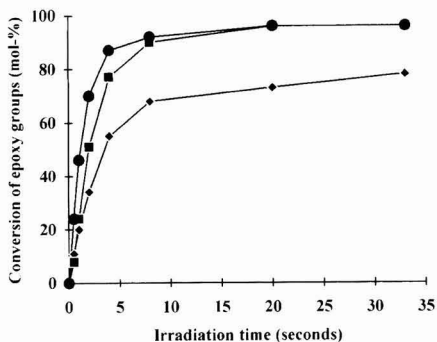


Figure 7—Conversion profile of epoxy groups for a photosensitive composition (Polybd 605 71 wt%/EPO2 29 wt%/FX512 2 (◆), 4 (■) and 8 (●) phr) upon irradiation under the Hanovia lamp. Wet thickness 25 μm

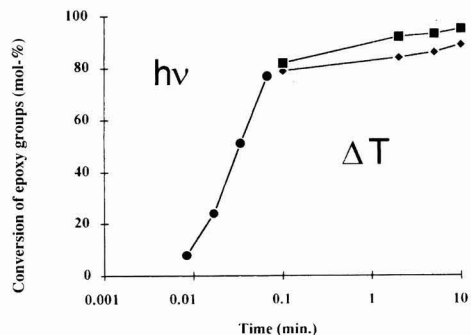


Figure 8—Thermal activation at 45°C (◆) and 80°C (■) of the conversion of epoxy groups after 6 sec of UV exposure under the Hanovia lamp for a photosensitive composition (Polybd 605 71 wt%/EPO2 29 wt%/FX512 4 phr). Wet thickness 25 μm

functional cycloaliphatic EPO2 give tack-free films immediately after irradiation, but the films are poorly adherent in the absence of any surfactant. The properties of cured films containing EPO1 are shown to be very sensitive to composition and thermal effects. Increasing the concentration of EPO1 from 7 to 22 wt% improves the curing of the film but at the expense of the post irradiation adhesion. Nevertheless, both properties are improved by a soft thermal post-treatment. This series of results also demonstrates the good inherent adhesion of Polybd 605 based coatings of low content in reactive diluents, as well as the versatility of difunctional epoxides to control the properties of the films.

Vinyl ether diluents were also tested in compositions of an overall content of 5 mmol.g<sup>-1</sup> of epoxy and vinyl functions. A first evaluation of the reactivity of these diluents blended with Polybd 605 was performed by measuring the degree of cure of the formulations appearing in Table 4. The coatings containing hexafluorophosphate sulfonium salts are tacky on exiting the UV-tunnel. Even with UVI-6974 that generates stronger acids, two of the three diluents still yield insufficient cure that is brought to completion after 12 hours of dark cure. The reactivity increases in the order VE3 < VE2 < VE1.

#### Adhesion on Glass, Aluminum, and Steel: Effect of Fluorinated Surfactants

The combined effects of a thermal post-treatment and the concentration of surfactant on the adhesion on glass plates are exemplified on the diagrams of Figure 9 measured for Polybd 605 - EPO1 mixtures. The same influence was observed on steel or aluminum plates that presented a slightly better adhesion (one digit above the values given for coated glass plates) on the average.

Compositions prepared with 0.5 wt% of fluorinated surfactant and the more promising reactive diluents show excellent adhesion to steel or aluminum plates. This is observed after curing initiation in the minicure tunnel at 50 mJ.cm<sup>-2</sup> and subsequent thermal activation at 70°C for 15 min. Increasing the UV dose to 400 mJ.cm<sup>-2</sup> does not change the results of the tests performed before heating the coatings. The thermal treatment is not sufficient with VE3. This confirms the lower reactivity of this diluent. The crosslinking polymerization nevertheless goes on and the coatings exhibit hardness 3H or harder grade after one to two days of dark

Table 4—Influence of the Nature of the Vinyl Ether Diluent on the Reactivity of Liquid Films<sup>a</sup>

Reactive Diluent <sup>b</sup> (wt%)	Curing Conditions			
	FX 512 <sup>c</sup>		UVI-6974 <sup>c</sup>	
	After Irradiation <sup>d</sup>	+ 12 hr of Dark Cure	After Irradiation <sup>d</sup>	+ 12 hr of Dark Cure
VE1 (22)	+	-	-	-
VE2 (24)	+	-	+	-
VE3 (21)	+	+	+	-

(a) Wet thickness 25 μm on glass plates: tacky (+); tackfree (-).  
 (b) Mixtures of Polybd 605 blended with the indicated reactive diluent.  
 (c) Photoinitiator content: 4 wt%.  
 (d) UV exposure: 50 mJ.cm<sup>-2</sup> in the minicure tunnel.

Table 5—Overview of the Coatings Properties Evaluated One Day after Curing<sup>a</sup>

Reactive Diluent <sup>b</sup>	Photoinitiator	Gloss	Pencil Hardness	Flexibility <sup>c</sup>	Solvent Resistance <sup>c</sup>
—	UVI-6974	107.5	2H	3/2 T	95
EPO2 (15)	UVI-6974	115.4	3H	5/2 T	>100
EPO2 (15)	FX512	87.3	H	3/2 T	45
VE2 (24)	UVI-6974	103.7	H	3/2 T	50
VE2 (24)	UVI-6974 <sup>d</sup>	118.3	3H	3/2 T	>100
VE1 (20)	UVI-6974	106.5	HB	2 T	20
VE1 (20)	FX512	70.8	H	5/2 T	15
VE3 (19)	UVI-6974	108.9	F	2 T	35

(a) Photoinitiator content: 4 wt% - Wet thickness: 25  $\mu\text{m}$  on steel plates - UV exposure: 50  $\text{mJ}\cdot\text{cm}^{-2}$  in the Minicure tunnel.

(b) Mixtures of Polybd 605 blended with the indicated reactive diluent.

(c) T bend and MEK rub tests.

(d) Plus thermal post-treatment: 2 min @ 100°C.

and cold cure. This need for extra time to achieve correct curing is also observed for compositions containing VE1 at concentrations exceeding 25 wt%.

The preceding results indicate that sufficient cure and adhesion can be obtained by combining composition effects (the nature and amount of photoinitiator and diluent) and treatment effects (UV and thermal curing). One of the major reasons for using epoxidized polybutadiene lies in the possibility of preparing flexible coatings with correct resistance to mechanical and chemical aggression. The selected data of Table 5 show that undiluted Polybd already provides a good compromise between flexibility on one hand, and pencil hardness and solvent resistance on the other. However, the high viscosity of the resin requires blending the prepolymer with diluent. The properties measured for the various systems at an overall concentration in polymerizable functions of 5  $\text{mmol}\cdot\text{g}^{-1}$  give the limits of the domain of adjustable properties that can be covered by formulating with epoxidized polybutadiene. By comparison to other reactive prepolymers, medium hardness and good flexibility are the dominant fea-

tures of the systems.<sup>16</sup> Solvent resistance is poor for some tested compositions. Intermediate values can be obtained by reducing the amount of added vinyl ether.

## CONCLUSION

Epoxidized polybutadiene is a reactive prepolymer with excellent curing capability. The family of cationically curable systems is extended to polymers with high functionality on flexible chains. A wide range of reactivity and properties can be obtained by blending the prepolymer with reactive viscosity reducers. The choice of the sulfonium salt initiator and the thermal treatment condition provides some control for the properties of the cured films. These must be evaluated after making sure that acceptable cure is achieved. The adhesion of reactive polybutadiene is high, and formulations gave excellent results on glass, aluminum, and steel. Among the few limitations that should be considered is the limited solvent resistance of compositions rich in vinyl ether diluents. A second drawback is expected moderate stability upon aging,

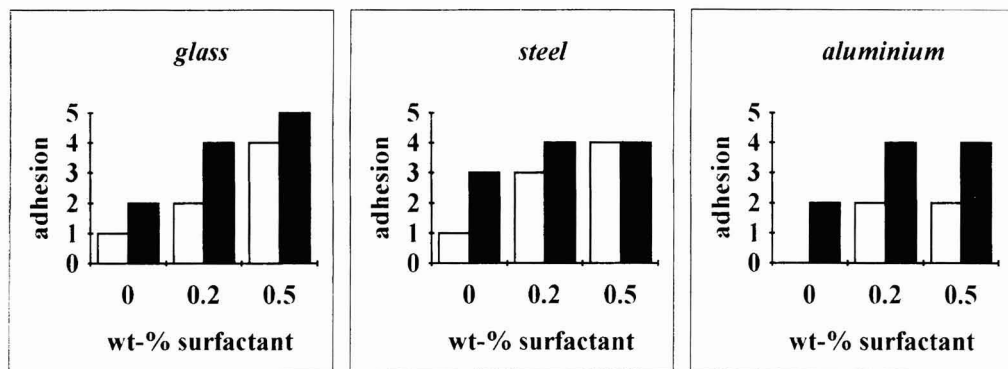


Figure 9—Effects of FC430 surfactant concentration without (white bars) and after thermal treatment (dark bars after 20 min at 73°C) on adhesion on glass, steel and aluminium plates. Coating composition: PEBD 65.5 wt%/EPO2 34.5 wt%/FX512 4 phr/ surfactant as indicated. Wet thickness 25  $\mu\text{m}$

essentially due to thermal<sup>14</sup> and photochemical<sup>17</sup> degradation of the chemically sensitive polymer backbone. These features are balanced by interesting reactivity at interfaces. Associated with high flexibility and good adhesion, they are actually recommended properties for a primer. Further developments of undercoats including epoxidized polybutadienes are currently under investigation.

## SUMMARY

Epoxidized polybutadiene was tested as a reactive prepolymer for 100% solids UV-curable formulations. The presence of triarylsulfonium salts that generate strong acids upon UV-irradiation permits initiation at will of the cationic copolymerization of the epoxy functions of the polymers together with the epoxy or vinyl ether functions of the diluents. Special attention was paid to the control of the initiator photolysis and to the monomer polymerization by UV and/or IR spectroscopy. The basic properties of the cured coatings were evaluated. Adhesion to various substrates was improved by combining the effects of a surfactant additive and a thermal post-treatment to achieve sufficient crosslinking.

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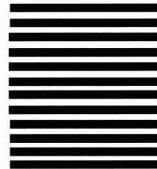
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# Mechanical Stability of Alkyd Emulsions— Part 2: Influence of Alkyd Properties

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The development of waterborne alkyd paints based on alkyd emulsions has been stimulated by more stringent health and environmental regulations. In addition, alkyd paints may have better performance in some applications compared to other types of waterborne paints such as latex dispersions. One problem that needs to be overcome is the limited colloidal stability of alkyd emulsions. It is important that the emulsions are not destabilized when exposed to shear forces, such as those produced by pumps used in the paint industry. An alkyd emulsion consists of alkyd oil droplets dispersed in water and stabilized by a surface active agent (emulsifier). Part 1 of this series described the influence of emulsion droplet size, type, and concentration of the emulsifier on the shear stability. The present study is an additional investigation of the influence of the alkyd properties on shear stability. Increasing the acid value of the alkyd increases the stability of emulsions stabilized with nonionic emulsifiers. Emulsions made using alkyds with shorter oil lengths and higher viscosities were less stable to shear compared to alkyds with higher oil lengths and lower viscosities.

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## INTRODUCTION

For health and environmental reasons, there is an increasing need for waterborne alkyd paints. Although large amounts of waterborne latex paints are used, these paints do not give satisfactory results in all applications. Alkyd emulsions are an alternative to both latex dispersions and white spirit-based alkyds, since they usually do not contain any organic solvents or coalescing agents.<sup>1,2</sup> In an alkyd emulsion, the alkyd oil is dispersed in water and stabilized by a surface active agent (an emulsifier). So far, the main application for alkyd emulsions is in air drying consumer paints. A drawback with alkyd emulsions compared to white spirit-based alkyds is the

longer drying time<sup>3</sup> and the limited colloidal stability. The emulsion should have a storage stability of at least one year, and a mechanical stability that can withstand the pumps used in the paint industry. The emulsions have to be stable at reasonable surfactant concentrations, since large concentrations result in poor film properties, such as high water sensitivity and soft films.

The most important factor for the colloidal stability of an emulsion is that the emulsion droplets are small.<sup>4,5</sup> With small droplets, the coalescence (when two droplets merge into one) rate and sedimentation is reduced. Both coalescence and sedimentation are important destabilizing mechanisms in the emulsion breakdown process. The droplet size obtained during emulsification depends on several factors. Most important are the emulsification method, i.e., the amount of energy used to create the droplets, and the efficiency of the emulsifier.<sup>6,7</sup> The emulsifier should facilitate droplet formation and protect the droplets from re-coalescing.

Part 1 described the influence of emulsion droplet size, type and concentration of emulsifier on the shear stability of alkyd emulsions.<sup>8</sup> The theory for shear-induced flocculation (two droplets in adhesive contact) was also discussed. This study showed that there was a critical apparent shear rate that had to be overcome before the droplets started to flocculate or coalesce. The critical apparent shear rate increased with decreasing emulsion droplet size. When the critical shear rate was reached, the relative coalescence rate seemed to be independent of the initial droplet size. Anionic emulsifiers were more effective in protecting droplets under shear compared to nonionic emulsifiers. Large amounts of ethoxylated nonionic emulsifiers were needed to obtain the same stability achieved with anionic emulsifiers. However, an increased degree of ethoxylation of the nonionic emulsifiers improved their performance.

Anionic emulsifiers are also more effective in the formation of alkyd emulsions compared to nonionic emulsifiers.<sup>7</sup> With ethoxylated nonionic emulsifiers, a close packing of the surfactant on the droplet surface is needed during emulsi-

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Table 1—Properties of Alkyds

Alkyd	Oil Length (as % Triglyceride)	Acid Value (mg KOH/g Alkyd)	Hydroxyl Number (mg KOH/g Alkyd)	Viscosity (cp, 50% in Toluene 20°C)	Molecular Weight (g/mol)
S-68 <sup>a</sup> .....	68	11	32	680 <sup>b</sup>	—
OI 65.....	65	9	81	30.4	20800
OI 74.....	74	8	41	11.8	11900
OI 82.....	82	9	44	7.4	4300
AV 14.....	78	14	47	8.4	4400
AV 11.....	78	11	46	7.7	5600
AV 6.....	78	6	44	9.2	8700
OHN 70.....	78	9	70	8.6	5800
OHN 40.....	78	10	40	8.8	6800

(a) Manufactured by Bergvik Kemi AB, Sweden. The rest of the alkyds are manufactured by Veitsilvoto Oy, Finland.  
 (b) mPa s at 100°C (100% alkyd).

fication to obtain stable emulsions. With anionic emulsifiers, stable emulsions are obtained at a very low packing density.

Another factor that can influence the colloidal stability of an emulsion are the properties of the dispersed (alkyd) phase. Alkyds used in paint have large variations in their properties. In this paper, the influence of alkyd properties, such as acid value, hydroxyl number, and oil length on the shear stability of alkyd emulsions are reported.

EXPERIMENTAL

Materials

The alkyd emulsions contained 50% (w/w) alkyd in water. One commercial and a range of model alkyds with varying acid values, hydroxyl values, and oil lengths were used (Table 1). All alkyds were based on tall oil fatty acid, orthophthalic acid, and pentaerythritol. The white spirit used to dilute the alkyd in some of the experiments was Nysolvin 75A with an aromatic concentration of about 0.5% (w/w) and Nysolvin ws with an aromatic concentration of about 17% (w/w). Both were supplied by Statoil, Sweden.

The emulsifier used was a fatty acid monoethanolamide ethoxylate with 13 ethylene oxide units. The fatty acid is produced from linseed oil. A constant concentration of emul-

sifier, 10% (w/w), on the dispersed (alkyd) phase was used. The pH was adjusted to 7 with a solution of potassium hydroxide before emulsification.

Methods

**EMULSIFICATION:** The emulsions was prepared in a high pressure homogenizer, Microfluidizer TM-110 (Microfluidics, Newton, MA). The emulsifier was dissolved in the aqueous phase which was added to the alkyd. The emulsion was premixed using a magnetic stirrer. Different emulsification temperatures were used for different alkyds in order to obtain comparable droplet sizes. Emulsification temperature and initial droplet sizes are given in Table 2.

**DETERMINATION OF DROPLET SIZE:** The droplet size distribution of the emulsions was measured using a laser diffraction instrument, Mastersizer (Malvern Instruments, England). The instrument uses an approximation of the Mie scattering theory based on the refractive index of the dispersed phase and its absorption. The relative refractive index,  $n_{alkyd}/n_{water}$ , was 1.12 for all alkyds except S-68 diluted to 60% (w/w) in white spirit which was 1.11. The relative refractive index for S-68 diluted to 95% (w/w) in white spirit was 1.13. The absorption value was estimated to be 0.1. The results are recorded as  $D(v,0.5)$  which is the volume-weighted median.

**MECHANICAL STABILITY:** In this study, the increase in droplet size (the coalescence), as a function of the apparent shear rate has been taken as a measure of the mechanical

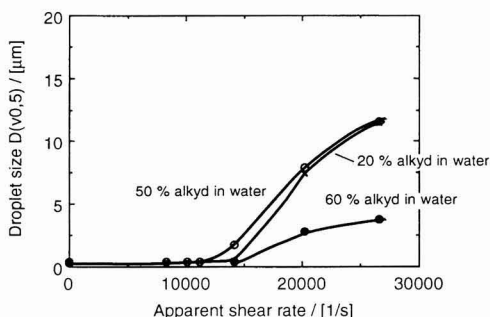


Figure 1—The influence of the concentration of the alkyd (dispersed phase) on the shear stability of alkyd emulsions. The emulsions contain alkyd S-68 and 10% emulsifier on alkyd weight

Table 2—Emulsification Temperatures and Initial Droplet Sizes for Model Alkyds

Model Alkyd	Emulsification Temperature (°C)	Initial Droplet Size D(v,0.5) (µm)
AV 14.....	70	0.22
AV 11.....	70	0.21
AV 6.....	70	0.32
O 182.....	80	0.21
O 174.....	70	0.26
O 165.....	60	0.62
OHN 70.....	80	0.21
OHN 40.....	80	0.25

stability of an alkyd emulsion. The samples were exposed to shear in an Ultra Turrax (Janke & Kunkel, IKA, GmbH, Germany). An apparent shear rate was calculated from the number of revolutions per minute. A more detailed description of the method used is described in reference (8).

**RESULTS AND DISCUSSION**

The influence of the concentration of the dispersed phase (the alkyd phase) on the mechanical stability of emulsions is shown in Figure 1. It can be seen that a concentration of alkyd from 20 to 50% (w/w) in water does not change the mechanical stability of the emulsion. When the concentration is increased to 60% (w/w), a more stable emulsion is obtained. The critical shear rate when the coalescence/flocculation starts is about the same but the coalescence rate (the increase in droplet size) is less for the higher concentration.

The rate of flocculation, expressed as the rate of increase in particle radius in a turbulent flow field should be<sup>8</sup>:

$$\frac{dr}{dt} = c \frac{\hat{s}^{\frac{3}{2}}}{\sqrt{\eta}} \phi r \frac{1}{Wt} \quad (1)$$

where r is the radius of the droplet assuming that flocculation always leads to coalescence, η is the viscosity of the emulsion, φ is the volume fraction of the dispersed phase,  $\hat{s}$  is the apparent shear rate (defined as the shear rate estimated for a laminar flow field), c is the constant, and wt is a stability factor for flocculation in a turbulent flow.

In a concentration emulsion, the viscosity is strongly dependent on the concentration of the dispersed phase. The effects can be estimated by the Krieger-Dougherty equation<sup>9</sup>:

$$\frac{\eta}{\eta_0} = (1 - k\phi)^{\frac{2.5}{k}} \quad (2)$$

where η<sub>0</sub> is the viscosity of the continuous phase and k is an empirical constant close to 1/φ<sub>close packing</sub>. The maximal φ<sub>close packing</sub> for monodispers spheres is 0.74.<sup>10</sup> The shear-induced flocculation should then be dependent on the volume fraction:

$$\frac{dr}{dt}(\phi) \propto \phi(1 - k\phi)^{\frac{1.25}{k}} \quad (3)$$

Figure 2 shows the relative flocculation rate as a function of the volume fraction of the dispersed phase calculated from equation (3). The flocculation rate passes through a maximum at about 30-40% and is significantly reduced at 60% (V/V) dispersed phase. The observed experimental results agree with this, which suggests that the reduced flocculation rate at 60% (w/w) dispersed phase is due to the increased viscosity.

Figure 3 shows the mechanical stability of emulsions of alkyds with varying acid values and constant oil lengths and hydroxyl number. The emulsion using the alkyd with the lowest acid value was less stable to shear than those with higher values. The measured increase in droplet size (the coalescence), is significant but not very large. The increased stability against coalescence with increasing acid value is explained by a contribution to the steric stabilization from

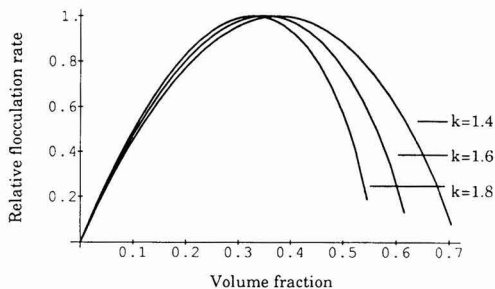


Figure 2—The relative flocculation rate as a function of the volume fraction of the dispersed phase [according to equation (3)]. The calculations have been made with three different values of k. k is an empirical constant close to 1/φ<sub>close packing</sub>

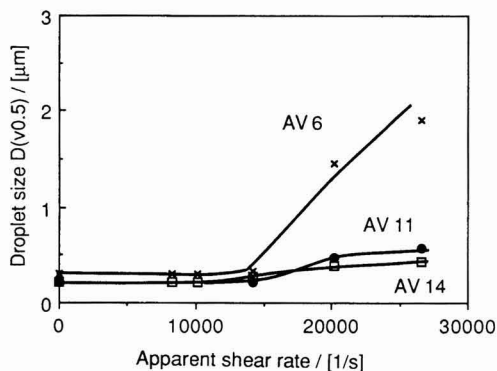


Figure 3—Mechanical stability of emulsions of alkyd with varying acid values (AV), and constant hydroxyl numbers and oil lengths. The acid values are in mg KOH/g alkyd. The emulsions contain alkyd S-68 and 10% emulsifier on alkyd weight

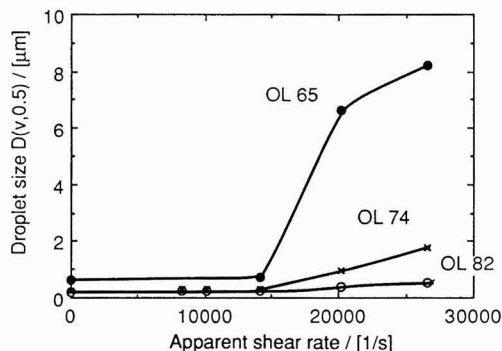
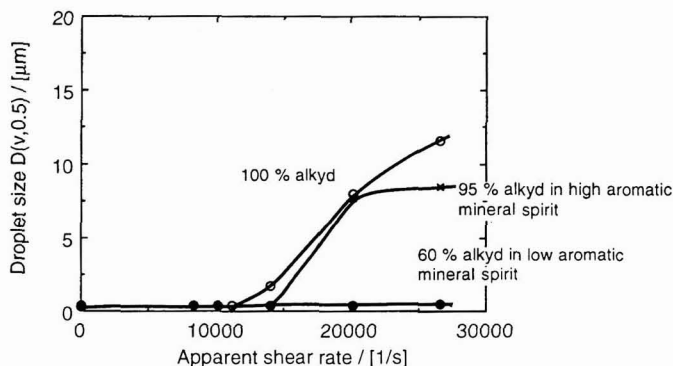


Figure 4—The influence of alkyd oil length (OL) at constant acid values on the mechanical stability of alkyd emulsions. The oil length is calculated as triglyceride. The emulsions contain alkyd S-68 and 10% emulsifier on alkyd weight



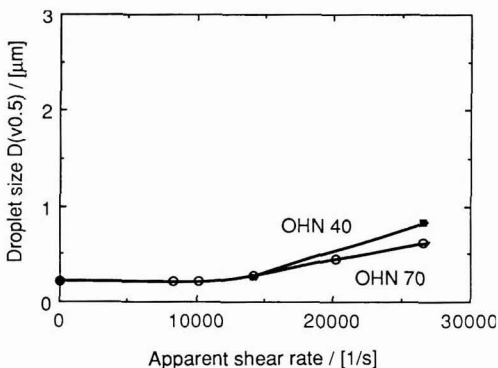
**Figure 5**—The influence of dilution of the alkyd with mineral spirit on the mechanical stability of alkyd emulsion. The emulsions contain 100% alkyd S-68, alkyd S-68 diluted to 60% (w/w) alkyd in a low aromatic mineral spirit and alkyd S-68 diluted to 95% (w/w) alkyd in a mineral spirit, with high aromatic concentration. All emulsions contain 10% emulsifier calculated on the dispersed phase

the nonionic emulsifier by electrostatic stabilization obtained by the higher density of ionic groups in the alkyd.

A change in oil length of the alkyd changes both the polarity and the viscosity of the alkyd. As seen in *Figure 4*, an increased oil length of the alkyd increases the mechanical stability of the emulsion. The alkyd with the shortest oil length has slightly larger initial droplets (0.6 μm) compared to the others. In Part I of this paper, it was found that for emulsions with droplet sizes below about 0.6 μm, the droplet size did not affect the critical shear rate for flocculation or the coalescence rate.

The observed effect is, therefore, believed to be caused by the different viscosities of the alkyds and/or due to different solubilities of the emulsifiers in the alkyds since they have different polarities.

During shear-induced flocculation, the hydrodynamic protection of the droplets is important. Zeichner and Schowalter<sup>11</sup> have shown that the probability for collision



**Figure 6**—Mechanical stability of emulsions of alkyds with varying hydroxyl numbers (OHN), and constant acid values and oil lengths. The hydroxyl numbers are in mg KOH/g alkyd. The emulsions contain 10% (w/w) emulsifier on alkyd weight

between solid spheres in a shear field is reduced by a factor of up to about 100 due to hydrodynamic protection. Hydrodynamic protection is the increase in flow resistance when two droplets approach each other. Ivanov and Dimitrov<sup>12</sup> have shown that the rate of drainage in the film between two approaching droplets is very sensitive to the properties of the emulsifier film at the water-oil interface. If the film is absent, or if the emulsifier is highly soluble in the oil phase, the coalescence rate is an order of magnitude faster. It could be expected that the emulsion from the more polar alkyd (OLGS) would have a higher concentration of this relatively hydrophilic emulsifier in the alkyd phase than the more nonpolar alkyds. Therefore, it will get less hydrodynamic protection compared to the other emulsions.

Another possible explanation is that the effect is due to the different viscosities of the alkyds. The alkyd with an oil length\* of 65% has a viscosity that is about three times as high as one with an oil length of 74%. Emulsion droplets from the alkyds with lower viscosities may be deformed easier when another droplet approaches and, thereby, the coalescence rate may be lower.

*Figure 5* shows the shear-induced instability in emulsions where the alkyd is diluted with mineral spirit before emulsification. The emulsifier concentration is similar for all samples (calculated on the dispersed phase). The pure alkyd and the one diluted to 95% (w/w) alkyd in mineral spirit give a less stable emulsion compared to the one diluted to 60% (w/w) alkyd in mineral spirit. This result may also be due to a change in viscosity and/or polarity of the alkyd as discussed. Dilution of the alkyd with a mineral spirit with a low aromatic concentration makes the dispersed phase less polar; therefore, the solubility of the emulsifier in the phases will be different, thus providing different degrees of dynamic protection. However, the most pronounced effects are due to the viscosity, which changes drastically from very high to low when the alkyd is diluted with mineral spirit. It, therefore, seems most likely that the viscosity of the internal phase is a major factor contributing to the shear sensitivity of alkyd emulsions.

An increased hydroxyl value of the alkyd from 40 to 80 mg KOH/g alkyd, at constant acid value, does not influence the mechanical stability of the emulsion as seen in *Figure 6*. Obviously, the change in polarity of the resin when the hydroxyl number is increased is too small to change the solubility of the emulsifiers in the phases to affect the stability.

**CONCLUSION**

The properties and concentration of the dispersed phase affects the mechanical stability of alkyd emulsions. An increased concentration of the dispersed phase from 50 to 60%

\*Oil length is the fatty acid content in the alkyd calculated as triglyceride.



(w/w) increases the shear stability of an alkyd emulsion. The stability can also be improved by increasing the acid value of the alkyd. However, an increased acid value may also result in negative effects such as a more water-sensitive paint film. A low viscosity alkyd favors the shear stability of the emulsion.

## ACKNOWLEDGMENT

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# Interfaces of Polyphenyletheretherketone (PEEK) and Polyphenylene Sulfide (PPS) Coated Zinc Phosphated Steels after Heating-Cooling Cycles in a Wet, Harsh Environment

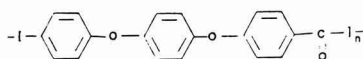
T. Sugama and N. Carciello  
Brookhaven National Laboratory\*

To evaluate the ability of polyphenyletheretherketone (PEEK) and polyphenylene sulfide (PPS) thermoplastic coatings that protect zinc phosphate (Zn•Ph)-treated steels from corrosion in a wet, harsh environment (1.0 wt% H<sub>2</sub>SO<sub>4</sub>, 3.0 wt% NaCl, and 96.0% wt% water at temperatures from 25° to 200°C), we exposed them in autoclave to attempt heating-cooling cyclic fatigue tests (1 cycle - 12 hr at 200°C + 12 hr at 25°C) up to 90 times. Although no changes in appearance were seen in the PEEK specimens after 60 cycles, extending the cycles to 90 caused the delamination of the coating film from the Zn•Ph. The major reason for this delamination was the degradation of the PEEK polymer caused by its hydrothermal-catalyzed esterification. In the PPS-coating systems, chemical reactions at the interfaces be-

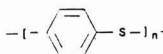
tween the PPS and Zn and Fe in the Zn•Ph layer during cycling led to the formation of ZnS and FeS reaction products, which enhanced the Zn•Ph-to-PPS adhesive bond; correspondingly, there were no signs of peeling and separation of the coating after 90 cycles. In addition, because these intermediate reaction products are insoluble at high pH, they minimized the rate of delamination of the PPS coating caused by the cathodic reaction, H<sub>2</sub>O + 1/2O<sub>2</sub> + 2e<sup>-</sup> → 2OH<sup>-</sup>, at the corrosion side of a defect in the film. In contrast, PEEK coatings containing non-reactive Zn•Ph underwent cathodic delamination, because of the susceptibility of Zn•Ph to alkali dissolution. Thus, we believe that combined layers of PPS and Zn•Ph provide great protection to steels in a harsh environment with temperatures up to 200°C.

## INTRODUCTION

Crystalline polyphenyletheretherketone (PEEK)



and polyphenylene sulphide (PPS)



thermoplastics are very attractive as corrosion-protective coatings for cold-rolled steels in harsh environments at hydro-

thermal temperatures up to 200°C. The major characteristic of these linear polyaryls is their molecular orientation caused by chain extension at a melting point of ≈ 290°C for PPS<sup>1,2</sup> and of ≈ 340°C for PEEK.<sup>3,4</sup> This orientation causes the crystallization of these polyaryls during cooling. Such melt-crystallization behaviors of these polyaryls give them specific, desirable characteristics as coatings, such as high-temperature stability, chemical and hydrothermal resistance, and good mechanical properties.

In our previous studies on the adhesive properties of polyaryls to steel surfaces, we found that the bond strength of the PPS-to-steel joints prepared in an air or oxygen-free N<sub>2</sub> gas environments depends on the species of sulphur-related iron compounds formed by the chemical reaction between the SO<sub>2</sub> gases emitted from PPS and Fe<sub>2</sub>O<sub>3</sub> at the

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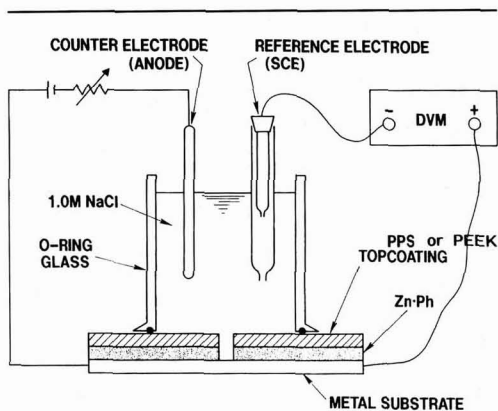


Figure 1—Schematic diagram of the cathodic delamination test

outermost surface sides of the steel, but is independent of the degree of crystallinity of PPS adjacent to steel.<sup>5</sup> This finding suggested that the formation of oxygen-catalyzed reaction compounds resulting from the introduction of oxygen into the interfacial regions gave better adhesion than bond structures assembled in N<sub>2</sub> gas. In contrast, in PEEK/steel joints, the incorporation of oxygen into the interfacial boundary regions affects the oxygen-catalyzed decomposition of PEEK, thereby resulting in its separation from the steel.<sup>6</sup> When this system was prepared in N<sub>2</sub> gas, it exhibited good bonding, because of the charge transfer reaction between PEEK and steel, and the formation of a well-crystallized PEEK.

On the other hand, an ideal polymeric coating that will protect steel from corrosion is one that acts as a barrier against the attack of corrosive reactants, such as oxygen, water, electrolyte species (e.g., H<sup>+</sup>, Na<sup>+</sup>, Ca<sup>2+</sup>, NH<sub>4</sub><sup>+</sup>, Cl<sup>-</sup>,

SO<sub>4</sub><sup>2-</sup>, NO<sub>2</sub><sup>-</sup>), and gases (e.g., O<sub>3</sub>, SO<sub>2</sub>, NO<sub>x</sub>), in natural or artificial environments. Unfortunately, all polymeric coatings are permeable to these reactants in some different degrees. Thus, an important consideration is that when the reactants reach the steel surfaces beneath the coating layers, corrosion occurs at the interfaces between polymer and steel. Once corrosion is initiated, the growth of corrosion products at the interfaces promotes wedging and blistering, which puts tremendous stress on the interfacial side of coating film; consequently, there is localized delamination and buckling of the stressed coating layers.<sup>7</sup> In addition, if the coating films become blemished, the underlying steels react with the reactants that have penetrated through the defect. Such a reaction is commonly called the two-step corrosion reaction: a cathodic reaction, H<sub>2</sub>O + 1/2O<sub>2</sub> + 2e<sup>-</sup> → 2OH<sup>-</sup>, which occurs under the coating adjacent to the defect, and an anodic reaction, Fe - 2e<sup>-</sup> → Fe<sup>2+</sup>, which occurs at a defect in the coating.<sup>8,9</sup> Considerable attention in this corrosion process has been given to the high pH environment created at the cathodic sites; namely, the alkali generated beneath the coating not only causes the formation of corrosion products, but also promotes the saponification of the polymer adjacent to the steel.<sup>10-12</sup> Subsequently, these phenomena occurring at the interfaces between the polymeric coatings and the steels lead to the cathodic delamination of the coatings from the steels. Thus, it is very important to tailor and modify the interface to inhibit the onset of the cathodic reaction.

For such tailoring of material systems in automotive applications, zinc or zinc alloy and zinc phosphate (Zn•Ph) conversion coatings are often introduced into the intermediate layers as a post-treatment of the steel surfaces.<sup>13</sup> We investigated the effect of poly(acrylic acid), p(AA)-modified Zn•Ph coatings on the cathodic delamination of high-temperature performance PPS and PEEK topcoatings in a NaCl solutions.<sup>14,15</sup> Although the rate of delamination of the polyaryls/Zn•Ph/steel systems was considerably lower than that of polyaryls/steel systems in the absence of Zn•Ph, our data indicated that attack by NaOH fluids formed by the charge

valence between OH<sup>-</sup> ions from the cathodic reaction generated at the corrosion side near defects, and Na<sup>+</sup> in the NaCl electrolyte leads to the delamination of the polyaryl film from the phosphated steel. The major reason for this cathodic delamination was an undermining caused by alkali-induced dissolution of anhydrous Zn•Ph layers [α- and γ-Zn<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>] derived from the dehydration of Zn•Ph hydrate [Zn<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>•2H<sub>2</sub>O] during the melt-crystallization of polyaryls at temperatures ranging from 350 to 400°C.<sup>16,17</sup>

On the basis of this information, we emphasized the exploration of the changes in chemistry and morphology occurring at the interfaces between PPS or PEEK coatings and phosphated steels after heating-cooling fatigue tests of defect-free polyaryl topcoat/Zn•Ph/steel in an accelerated corrosion environment at 25 to 200°C. The exploration involves the visual and metallographic observations, and the changes in chemical composition, states, and profiles.

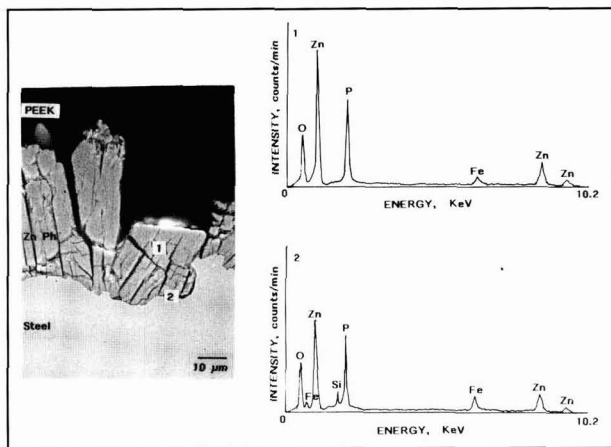


Figure 2—SEM-EDX analysis of cross-section for 60-cycled PEEK/Zn•Ph/steel joint specimens

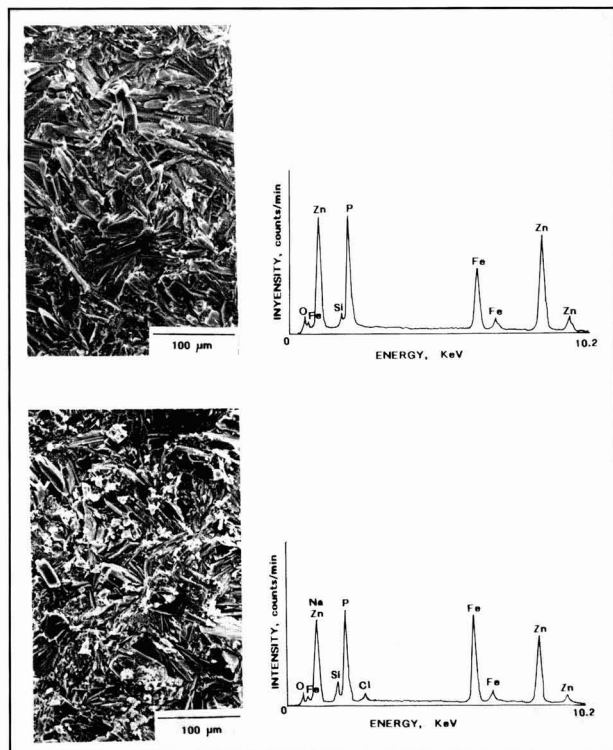


Figure 3—SEM images and EDX analyses for the interfacial Zn-Ph side after 60 (top) and 90 (bottom) cycles

All the information obtained was correlated directly with the results from cathodic delamination tests for defective coating systems.

## EXPERIMENTAL METHODS

### Materials

The metal substrate used as AISI 1010 cold-rolled steel containing 0.08-0.13 wt% C, 0.3-0.6 wt% Mn, 0.04 wt% P, and 0.05 wt% S. The formulation of the zinc phosphate liquid was 4.6 wt% zinc orthophosphate dihydrate [ $Zn_3(PO_4)_2 \cdot 2H_2O$ ], 9.2 wt%  $H_3PO_4$ , 2.0 wt% [25% poly(acrylic acid, p(AA)], 1.0 wt%  $Co(NO_3)_2 \cdot 6H_2O$ , and 83.2 wt% water. The average molecular weight of p(AA) polyelectrolyte (supplied by Rohm and Haas Company) was  $\approx 60,000$ .

In preparing the zinc phosphate (Zn•Ph) samples, the steel surfaces were wiped with acetone-soaked tissues to remove any surface contamination from mill oil. The steel was then immersed for 20 min in the Zn•Ph conversion solutions at 80°C. Finally, the deposited Zn•Ph layers were dried in an oven at 100°C for 2 hr. The morphological feature of Zn•Ph layers covered over the steel surfaces was characterized by an interlocking structure of approximately 20  $\mu m$  long plate-like crystals. PPS and PEEK powders for the

slurry coating were supplied by the Phillips 66 Company and the Imperial Chemical Industry (ICI), respectively. The “as-received” PPS and PEEK were finely divided, tan- and gray-colored powders having a high melt flow with a melting point of  $\approx 290$  and  $\approx 350^\circ C$ , respectively. These polyaryl films were deposited in the surfaces of Zn•Ph-treated steel substrate in the following way. First, the substrates were dipped into a polyaryl slurry consisting of 45 wt% polyaryl and 55 wt% isopropylalcohol at 25°C. The polyaryl slurry coated substrates were heated in air for 2 hr at 350°C for PPS coating, and in  $N_2$  for 2 hr at 400°C for PEEK coating, and then cooled to room temperature at the rate of  $-10^\circ C/min$ . The thickness of the polyaryl coating films for all specimens ranged from 0.075 to 0.1 mm.

### Measurements

The cyclic fatigue tests for the PPS and PEEK-coated Zn•Ph panel specimens (7.5 cm  $\times$  7.5 cm) were accomplished as follows: the PPS or PEEK covered phosphated steel specimens were directly soaked in a corrosive solution consisting of 1.0 wt%  $H_2SO_4$ , 3.0 wt% NaCl, and 96.0 wt% water, and then the heating-cooling cycle (one cycle = 12 hr at 200°C + 12 hr at 25°C) was repeated up to 90 times. The appearance of the specimens and its interfaces were examined after 20, 30, 60, and 90 cycles to gain the information on the hydrothermal durability or decomposition of coatings,



**Table 1—Chemical Composition of Interfacial PEEK and Zn•Ph Sides for PEEK/Zn•Ph/Steel Systems after 0, 30, 60, and 90 Cycles**

Cycle	Failure Side	Atomic Compositions, %						
		P	C	O	Fe	Zn	Cl	Na
0	PEEK	5.94	59.04	30.59	0.59	3.84	—	—
0	Zn•Ph	14.91	36.36	40.56	0.84	7.33	—	—
30	PEEK	6.28	63.82	25.75	0.69	3.46	—	—
30	Zn•Ph	13.40	40.37	38.30	0.98	6.95	—	—
60	PEEK	5.81	65.41	23.84	1.11	3.83	—	—
60	Zn•Ph	13.33	43.13	35.89	1.22	6.43	—	—
90	PEEK	8.95	48.04	34.11	1.41	3.97	2.80	0.72
90	Zn•Ph	7.55	48.33	33.70	3.85	3.47	1.97	1.13

and the changes in chemistry occurring at the polymer-substrate interfaces.

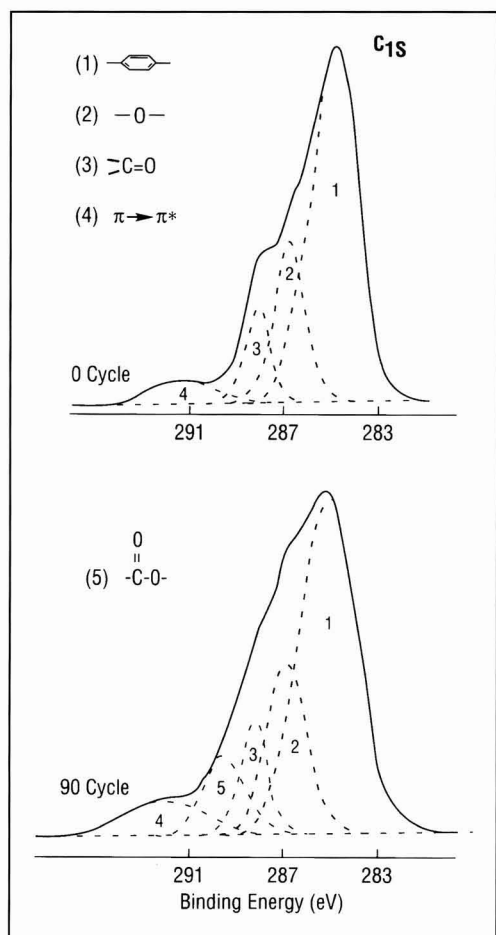
Morphological and chemical alterations were explored in cross-sections of the specimens, and on the interfacial side of substrates separated physically from the coatings after the cyclic tests using scanning electron microscopy (SEM) and energy-dispersion X-ray spectrometry (EDX). X-ray photoelectron spectroscopy (XPS) was used to detect the chemical composition of both the physically induced and naturally failed coating and substrate interface sides and to identify the chemical factors, which act in accelerating the degradation of interfacial structure and in promoting the delamination of coatings. The XPS used as a V.G. Scientific ESCA 3MK II. The excitation radiation was provided by an Al K $\alpha$  (1486.6 eV) X-ray source, operated at a constant power of 200 w. The vacuum in the analyzer chamber of the instrument was maintained at 10<sup>-9</sup> Torr. The atomic concentrations for the respective chemical elements were determined by comparing the XPS peak areas, which were obtained from differential cross-sections for core-level excitation. To set a scale in all the high-resolution XPS spectra, the binding peak was calibrated with C<sub>1s</sub> of the principal hydrocarbon-type carbon peak fixed at 285.0 eV as the internal reference. A curve-deconvolution technique, in conjunction with DuPont curve resolver, was employed to find the individual chemical states from the high-resolution spectra of each element.

The cathodic delamination tests for the polyaryl-coated Zn•Ph specimens after cycling were conducted in an air-covered 1.0 M NaCl solution using an applied potential of -1.5 V vs SCE for 8 days (see Figure 1). Although such a high potential may cause the formation of corrosion products, it was employed to accelerate the rate of cathodic reaction. The thickness of the Zn•Ph layer deposited on the steel substrates (100 × 100 mm square) was an approximately 30  $\mu$ m, as determined with a surface profile measuring system. The total area of film that comes into contact with the NaCl solution was 6.0 × 10<sup>3</sup> mm<sup>2</sup>. A defect was made using a 1-mm diameter drill-bit. After exposure, specimens were removed from the cell and allowed to dry. The polyaryl coating was removed by cutting, revealing a light gray, delaminated region adjacent to the defect.

**RESULTS AND DISCUSSION**

**PEEK Coating Systems**

No blistering or lifting of the film was observed in the PEEK-coated specimens after 60 cycles. This fact strongly



**Figure 4—C<sub>1s</sub> core-level spectra for interfacial PEEK sides before (0 cycles) and after 90 cyclic tests**

suggested that the durability of the adhesion between the PEEK and Zn•Ph is good, and that the PEEK itself is strongly resistant to such a harsh environment. However, an increase to 90 cycles caused the delamination of the film from the Zn•Ph. To visualize the good adhesion in PEEK after 60 cycles, specimens were inspected using SEM coupled with EDX. The SEM micrograph of a cross-sectional area of a specimen is shown in *Figure 2*, in which three regions of the cross-section are highlighted. The EDX spectra (not shown) of the top layer in the SEM micrograph showed the presence of two peaks, an intensive line for C and the weak line for O, revealing the PEEK coating. The bottom layer corresponded to the underlying steel, because there was only one signal of Fe on the EDX spectrum (not shown). Thus, the regions are composed of PEEK (top of figure), Zn•Ph (middle), and the underlying steel (bottom). The EDX spectrum of the area denoted as location 1, corresponding to the Zn•Ph layer, not only has a prominent line of Zn, P, and O signals as the major components of Zn•Ph, but also shows a moderate line of Fe, reflecting the incorporation of some Fe-based compounds into the Zn•Ph layers. Spectral features similar to those of Zn•Ph were identified in a critical interfacial boundary region (location 2) between Zn•Ph and steel. The only difference is the presence of a weak Si signal, revealing contamination of the steel surfaces. Assuming that this interpretation is correct, the O element also may belong to the Si oxide contaminant. As we deduced from the SEM image, the Zn•Ph layers have an open-surface structure with some microfissures and cracks. It is possible that the PEEK, possessing high melt flow characteristics, fully fills in these open spaces and microfissures. We believe that such a PEEK penetrated Zn•Ph composite structure forms a mechanically strong intermediate layer. Furthermore, the image represents good linkages between Zn•Ph and underlying steel, and there were no signs of any corrosion-induced damage nor any separation between the layers after 60 cycles.

To support this information, the failed surfaces on the substrate sides for 60- and 90-cycled PEEK specimens were searched using SEM-EDX analytical tools; the results are shown in *Figure 3*. After 60 cycles, the typical SEM topographical features of Zn•Ph coatings (top photograph) can be seen on the substrate surfaces that were separated physically from the PEEK coating; namely, a characteristic of interlocking texture of rectangular-shaped Zn•Ph crystals which are identical from the EDX spectrum, with Zn and P peaks as the major components. By comparison, a different morphology was observed in images of the substrate surface that separated naturally from the PEEK after 90 cycles. The difference lay in the presence of a large number of granular particles distributed throughout the Zn•Ph layers. The EDX spectrum showed a strong Fe signal and a weak Cl peak, while retaining the Zn and P lines as principal components. Relating to the appearance of brown rust colors on the specimens, the excitation of a pronounced Fe signal suggested that the particles may be associated with Fe-related corrosion products. However, there was no evidence to show whether such corrosion products appear before or after the delamination of coating films.

The changes in chemical composition of the PEEK and Zn•Ph interfaces as a function of the number of cycles were investigated using XPS (*Table 1*). For control specimens at 0 cycle, the composition of the PEEK side removed from the

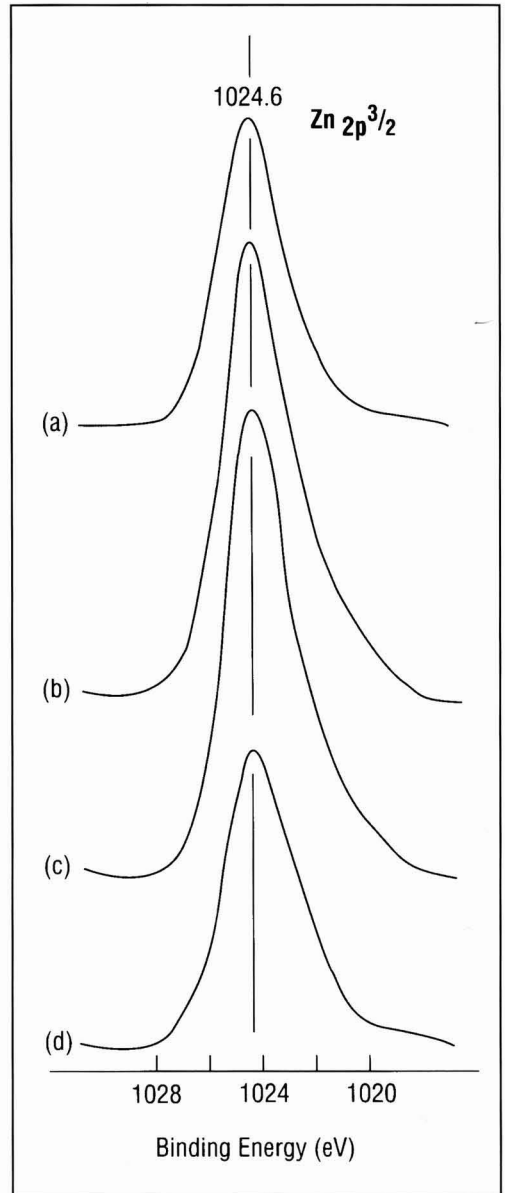


Figure 5—XPS spectra of Zn<sub>2p<sub>3/2</sub></sub> region for the substrate interface of (a) 0, (b) 30, (c) 60, and (d) 90 cycled specimens

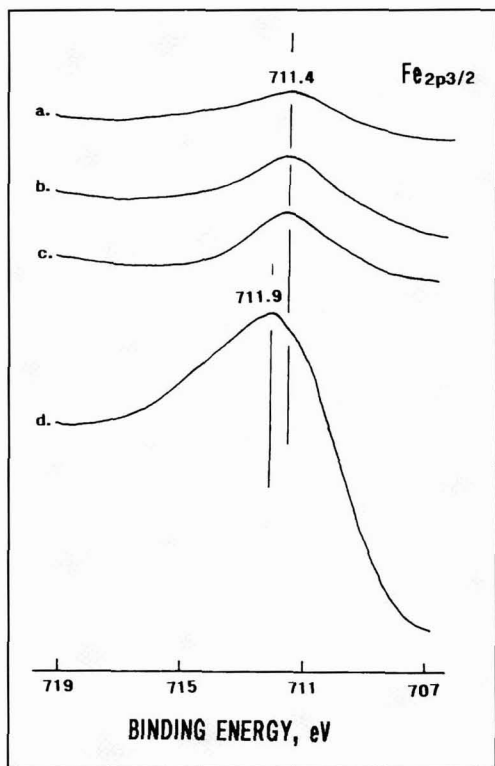


Figure 6—Fe<sub>2p3/2</sub> region for the interfacial substrate side of (a) 0, (b) 30, (c) 60, and (d) 90 cycled specimens

substrate was comprised of 5.94% P, 59.04% C, 30.59% O, 0.59% Fe, and 3.84% Zn. The P, O, Fe, and Zn refer to the Zn•Ph; C and O as the major constituents may be due to the PEEK. To substantiate this speculation, we examined the C<sub>1s</sub> core-level spectrum on the PEEK side by XPS. The resulting C<sub>1s</sub> region (Figure 4, top) can be deconvoluted by the four resolvable Gaussian components at BE of 285.0 eV, 286.5 eV, 288.0 eV, and 291.5 eV. As mentioned in our previous study,<sup>6</sup> the principal component at 285.0 eV as the curve 1, reflects carbon in the aryl groups. The assignments of the

curves 2 and 3 as minor components are attributable to C in ether (C–O–C) and in ketone (>C=O), respectively; the component of curve 4 emerging with the highest BE belongs to the π\* ← π shake-up satellite of conjugated C=C bonds in the phenyl rings. Because all of these components are correlated directly to the structure of PEEK, the C detected at the PEEK interfaces appears to have originated from the PEEK itself.

Table 1 shows that the failed substrate interface had high concentration of P, O, Fe, and Zn atoms and considerably fewer C atoms, compared with the PEEK side. This C is more likely to reflect the p(AA) and contamination in the Zn•Ph layers, rather than C from the PEEK. In fact, the C<sub>1s</sub> region (not shown) indicated excitation of a peak near 289.5 eV, belonging to the C of the carboxylic acid (–COOH) groups in the p(AA), but did not show any signals corresponding to the ether group and the π\* ← π shake-up satellite in PEEK. Consequently, the loss of adhesion is believed to be a mixed mode of cohesive failure, in which the locus of breakage occurs through the Zn•Ph layers and the adhesive one, occurring at the interfaces between the PEEK and the Zn•Ph. In comparisons with uncycled control specimens, our attention was drawn to the following changes in the cycled specimens: the first change was a decrease in the concentrations of P, O, and Zn at the interfacial substrate side with an increase in cycle number, which was inconsistent with the changes in concentration of these elements at the PEEK side; the second change was a slight increase in Fe content as a function of cycle number on both sides; and the third change was that no traces of Cl and Na were found at the failed interfaces of the 30- and 60-cycled specimens. There is a connection between the first and second changes which we interpret as follows: when this coated specimen was exposed in the autoclave at 200°C, penetration of some of the hot solution into the PEEK and Zn•Ph layers promoted a corrosion reaction in the underlying steel. Next, this reaction led to the formation of Fe-related corrosion products at the interfaces between Zn•Ph and steel. Thus, the reason for the decrease in the percentage of P, O, and Zn may be due to the incorporation of Fe corrosion products into the Zn•Ph layer. The third difference signifies that the PEEK coating serves as a barrier to the transportation of the ionic electrolyte species, such as Na<sup>+</sup> and Cl<sup>-</sup>, into the coating layer during the 60 cycles.

Figure 5 illustrates the XPS high-resolution Zn<sub>2p3/2</sub> core-level spectra of the substrate interfacial side for 0-, 30-, 60-, and 90-cycled specimens. The spectral features of all of them

Table 2—Chemical Composition of Interfacial Failure Sides for PPS/Zn•Ph/Steel Systems after 0, 30, 60, and 90 Cycles

Cycle	Failure Side	Atomic Compositions, %						
		S	P	C	O	Fe	Zn	Si
0	PPS	2.83	11.86	37.91	39.75	3.17	4.48	0.00
0	Zn•Ph	1.77	9.90	39.72	39.76	4.15	4.70	0.00
30	PPS	4.41	12.34	36.62	38.03	3.25	5.35	0.00
30	Zn•Ph	4.80	8.09	38.90	39.04	4.39	4.44	0.34
60	PPS	3.91	13.11	36.15	35.77	4.50	6.56	0.00
60	Zn•Ph	5.07	7.42	38.25	40.30	4.71	3.73	0.52
90	PPS	2.76	14.52	35.55	35.15	4.96	7.06	0.00
90	Zn•Ph	5.67	5.95	39.92	39.98	4.83	2.99	0.66

were similar to that of the control (a) which has a single peak at 1024.6 eV, originating from zinc in the Zn•Ph. This finding strongly suggested that hydrothermal and chemical decomposition of Zn•Ph itself does not occur during 90 cycles. In the  $Fe_{2p_{3/2}}$  region (Figure 6), the control (a) displays a very weak signal emerging at the BE position near 711.4 eV; the intensity of this signal slowly increases with increasing cycles up to 60. The delaminated surfaces of the 90-cycle specimens showed an overall excitation of the  $Fe_{2p_{3/2}}$  signal, with two resolvable components at 711.9 and 711.4 eV. To assign the peaks, we investigated several reference Fe oxide compounds, such as iron (II) oxide (FeO), iron (III) oxide ( $Fe_2O_3$ ), and iron (III) hydroxide [FeO(OH)]. The position of the principal peak from the  $Fe_{2p_{3/2}}$  spectrum of these reference compounds was as follows: 709.6 eV for FeO, 711.5 eV for  $Fe_2O_3$  and 711.7 eV for FeO(OH). From such comparison, we judged that the major contributor to the 711.4 and 711.9 eV peaks is Fe in the  $Fe_2O_3$  and the FeO(OH) which is one of the ferric rust compounds, respectively. In fact, we visually observed the appearance of a rusty color on the separated Zn•Ph side after 90 cycles. Figure 4 illustrates the  $C_{1s}$  region of PEEK interfacial side for the 90-cycled specimens. As mentioned, the spectral features of uncycled specimens (0) corresponded to the original PEEK structure. A striking difference in spectral features was observed from the PEEK interface after 90 cycles; the width of the overall peak from 282 to 290 eV was more extensive, suggesting that an additional chemical component was introduced into the interfacial PEEK side during the fatigue tests. The new component, which is reflected in peak No. 5 at 289.4 eV, can be ascribed to the formation of -COO- (carboxylic acid or ester) groups.<sup>18</sup> Such an addition component in the PEEK structure clearly verified that the PEEK coating itself undergoes hydrothermal-catalyzed esterification. However, there is no evidence on how the esterifying process occurs during the long-term cyclic test.

Based upon information obtained from the cyclic tests of PEEK/Zn•Ph/steel joint systems, the following statements can be conclusively drawn. Even through the *in-situ* growth of Fe-related corrosion products at interfaces promotes wedging action which gives a compressive stress to the interfacial coating film side, its effect on the buckling delamination of stressed coating may be negligible. Rather, considerably more attention should be given to the susceptibility of PEEK to hydrothermal-catalyzed esterification, which degrades the polymer film, thereby resulting in its delamination.

### PPS Coating System

An encouraging result was obtained from the cyclic tests of the PPS-coated specimens; namely, there was no sign of delamination and decomposition of PPS coating films after 90 cycles. This fact proved that the bond durability at the

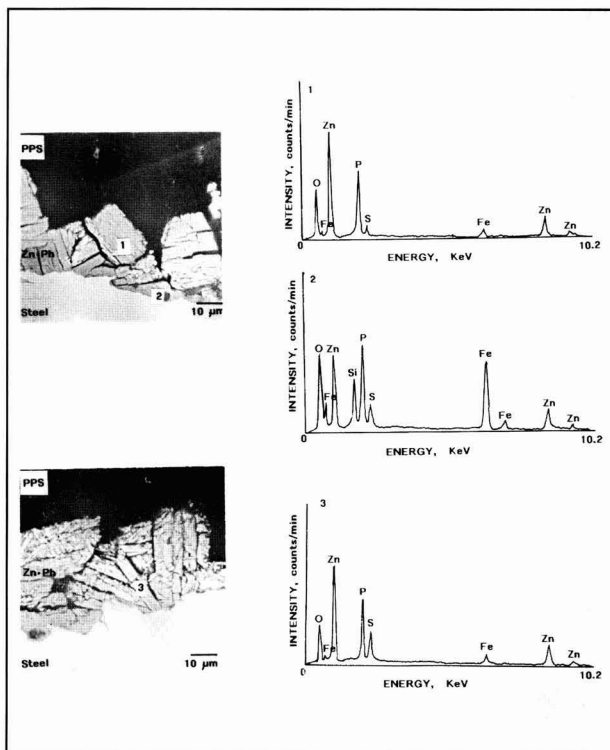


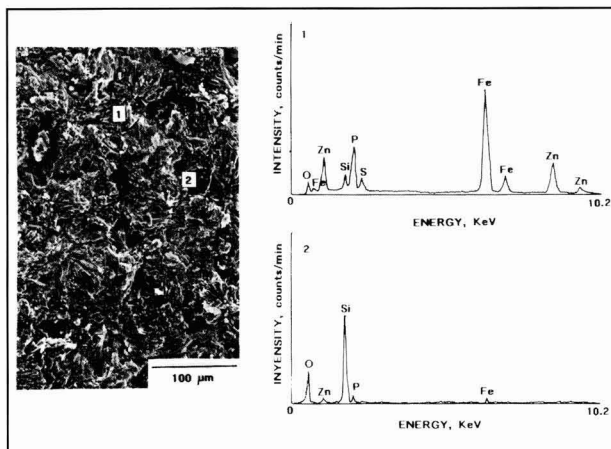
Figure 7—SEM-EDX examinations for the cross-section regions of the 30-cycled (top) and 90-cycled (bottom) PPS/Zn•Ph/steel joints

interfaces between PPS and Zn•Ph is outstanding in such a harsh environment.

To investigate the structure and chemistry of the interfacial bonds that confer such excellent durability, we again examined cross-sections of specimens and of both the interfacial PPS and substrate sides by SEM-EDX and XPS.

Figure 7 shows the SEM images and EDX spectra for the cross-sectional areas of 30 (top) and 90 (bottom) cycled PPS/Zn•Ph/steel joints. These SEM images clearly demonstrated that the cross-section regions have three different layers. The EDX spectrum (not shown) of the top layer in the SEM micrographs indicated that only two elements, C and S, were present, reflecting the PPS topcoating. No traces of Cl and Na were detected in the PPS layers. From the EDX pattern (not shown) containing a single element of Fe, the bottom layer in the micrograph corresponds to the underlying steel. An image similar to that obtained from the 60-cycled PEEK/Zn•Ph/steel joints (Figure 2) can be seen on those exposed to 30 cycles; namely, melted PPS penetrates into the open-surface microstructure and microfissures of Zn•Ph layer. Such filling of the spaces in the Zn•Ph layers by PPS may increase the extent of mechanical bonding between PPS and Zn•Ph layers. Emphasis was focused on the EDX spectra at the locations 1 in the Zn•Ph layer, and 2 in the critical

Figure 8—SEM-EDX data of the substrate interface side removed from the PPS film for the 30-cycled specimens



boundary region between the Zn•Ph and steel. Although we reported that SO<sub>2</sub> gases are emitted from oxidized PPS at high temperatures,<sup>5</sup> a surprising fact here was that the S species from PPS was present throughout the Zn•Ph layers. The detection of S in the Zn•Ph steel interfacial areas intimates that S invaded deeply into the Zn•Ph layers causing the formation of S-related Fe compounds by reacting with the underlying steel. We paid considerable attention to the striking change in the image of the Zn•Ph layer for the 90-cycle specimens. A noteworthy difference from that of the 30-cycle specimens was the development of numerous small cracks. Such a damaged and porous state of the Zn•Ph layer was implicated by the increased intensity of S in the EDX spectrum at a location 3, suggesting that the Zn•Ph layers react with the S species from PPS.

To further substantiate the results from this cross-sectional examination, we studied the chemical composition and development of the microstructure of the failure surfaces on the substrate side removed physically from the PPS film by SEM-EDX. The result from 30-cycled PPS/Zn•Ph specimen is given in Figure 8. Compared to the 60-cycled PEEK/Zn•Ph specimens (see Figure 3, top), a very different image can be seen on the interfacial substrate side of the 30-cycled specimens; namely, no complete covering of Zn•Ph remains on the steel surfaces; therefore, most of Zn•Ph layer must have been transferred to the PPS side when the loss of adhesion occurred. The EDX location 1 had highly intense Fe signal, moderately intense signals from Zn and P, while O, Si, and S were minor components. The detection of Zn and P suggests that some residual Zn•Ph remained on the failed substrate side. Location 2 showed a different morphology from that of location 1: the EDX pattern on this structure with its sharp edge showed two intense signals of Si and O, revealing the formation of Si oxides as the contaminant of the steel surfaces. We infer from this result that the loss of adhesion occurs in a boundary region between Zn•Ph and underlying steel, suggesting that Zn•Ph adheres more strongly to the PPS coatings. The microtexture of specimens after 60 cycles (not shown) was similar to that of the 30-cycled specimens. However, the EDX spectrum had a more intense

Fe signal and less intense Zn•Ph-related P and Zn lines, with S, Si, and O present as minor elements. Further decay of the Zn and P line intensities was recorded at the EDX of the 90-cycled specimens, reflecting the elimination of the Zn•Ph layer adhering to the steel. This SEM (not shown) image represented the appearance of a scroll-tubular cluster. EDX data showed that this cluster consisted of Fe and S, reflecting the formation of Fe-enriched S reaction products beneath the Zn•Ph layer. Thus, increasing the number of cycles not only enhances the rate of reaction of S with Zn and Fe in the Zn•Ph layer, but also leads to the formation of Fe-S reaction compounds from the chemical reaction between S and the underlying steel. However, no experiment on when the propagation of such interfacial interactions is terminated was made in this paper.

Table 2 gives the XPS elemental compositions for both interfacial failure sides in the physically separated PPS/Zn•Ph joint systems before and after 90 cycles. The composition of the interfacial PPS surface of control specimens (0 cycles) closely resembled that of the Zn•Ph interface. The detection of a certain amount of P, Fe, and Zn on the PPS interface verified that these elements migrate from the Zn•Ph-covered steel substrate to the coating sides during the failure of the bond. By comparison with the control in the PEEK/Zn•Ph joint system (see Table 1), two interesting results were found: one was that more Fe is taken up by the PPS, suggesting that the Fe species in the Zn•Ph is attracted to the PPS; the second was that the C content of PPS side is much lower. No Si was detected on both failure sides. Thus, it is reasonable to suppose that bond failure occurs through the Zn•Ph layers. When the specimens were cycled, a conspicuous amount of S was incorporated in the Zn•Ph layers in the first 30 cycles, resulting in a high concentration of S of 4.14 and 4.80% on the failure PPS and Zn•Ph sides, respectively. A further increase in S to 5.67% was reported on the Zn•Ph side after 90 cycles. In contrast, the amount of the PPS side tended to decrease with more cycles. The data also expressed that the high number of cycles causes an increase in P, Fe, and Zn content at the PPS interface, while the amount of O and C falls from 39.75 to 35.15% and 37.91 to 35.55%, respec-



tively. Surprisingly, the amount of P and Zn at the Zn•Ph interface seems to be reduced with increasing number of cycles. This reduction was related directly to the increase in Fe and Si content on the Zn•Ph side; namely, much of the Zn•Ph layer was transferred to the PPS side during bond failure, disclosing the underlying steel surfaces. The information obtained strongly supported our hypothesis that S from PPS interacts with the Zn•Ph layer and plays an important role in developing the Zn•Ph-to-PPS adhesive bonds. The increase in the rate of such an interaction resulted in a locus of bond failure occurring through the mixed layers of non-reactive Zn•Ph and reaction products adjacent to the underlying steel.

Next, our emphasis focused on identifying the reaction products at the interfaces between the PPS and Zn•Ph, and the changes in phase compositions as a function of the number of cycles. This information was obtained by inspecting the XPS  $Zn_{2p_{3/2}}$ ,  $Fe_{2p_{3/2}}$ , and  $S_{2p}$  regions on the failure surface of Zn•Ph side. We already knew that the interaction of PPS with the Zn•Ph generates S-related Zn and Fe compounds as reaction products. Thus, we used four compounds, zinc sulfide (ZnS), zinc sulfate ( $ZnSO_4 \cdot H_2O$ ), iron (II) sulfide (FeS), and iron (II) sulfate ( $FeSO_4 \cdot 7H_2O$ ), as reference samples to identify the BE of the precise peak position in the  $Zn_{2p_{3/2}}$  and  $S_{2p}$  regions. The peak positions from these core-level regions of the reference compounds were: 1022.7 eV in  $Zn_{2p_{3/2}}$  and 162.5 eV  $S_{2p}$  for ZnS; 1024.0 eV in  $Zn_{2p_{3/2}}$  and 170.2 eV in  $S_{2p}$  for  $ZnSO_4 \cdot H_2O$ ; 711.4 eV in  $Fe_{2p_{3/2}}$ , and 161.7 eV in  $S_{2p}$  for FeS; and 712.7 eV in  $Fe_{2p_{3/2}}$  and 170.5 eV in  $S_{2p}$  for  $FeSO_4 \cdot 7H_2O$ .

Figure 9 shows the changes in spectral features in the  $Zn_{2p_{3/2}}$  region, obtained by varying the cycle number. The spectrum of the control (a) reveals only a single symmetric peak at 1024.6 eV, caused by Zn originating from the Zn•Ph. A noticeable change in the shape of the  $Zn_{2p_{3/2}}$  signal from the 30-cycled specimen (b) was the emergence of a new signal at 1022.7 eV. According to the reference peaks, this new peak conceivably could be the Zn in the ZnS reaction product derived from the interaction between PPS and Zn•Ph; the intensity of this peak grows with more cycles. After 90 cycles (d), the peak at 1022.7 eV becomes a dominant component, while the Zn•Ph-related peak at 1024.6 eV considerably decays. Relating this result to the SEM cross-section image of 90-cycled specimens (see Figure 7, bottom), the development of numerous microfissures in the Zn•Ph layer may be due to degradation of the Zn•Ph layers caused by the *in-situ* conversion of Zn•Ph into the ZnS reaction product. The S species from the PPS not only have strong affinity with the Zn in the Zn•Ph, but also presumably react with the Fe in either the Zn•Ph layer or the underlying steel. Thus, the  $Fe_{2p_{3/2}}$  region (Figure 10) of specimens before and after 90 cycles was also inspected to identify the formation of the Fe-related S reaction products. The spectrum from the control had an intense signal at 711.4 eV. Although the peak position of  $Fe_2O_3$  in BE is very close to that of FeS, we believed that the assignment of this peak as the main component is more likely to be Fe originating from the FeS compounds formed by chemical reactions between PPS and the  $Fe_2O_3$  in the Zn•Ph layer, rather than that in the  $Fe_2O_3$ . A similar spectral feature was seen in the 90-cycled specimens. Relating these findings to the SEM-EDX examinations, we suggest that the formation

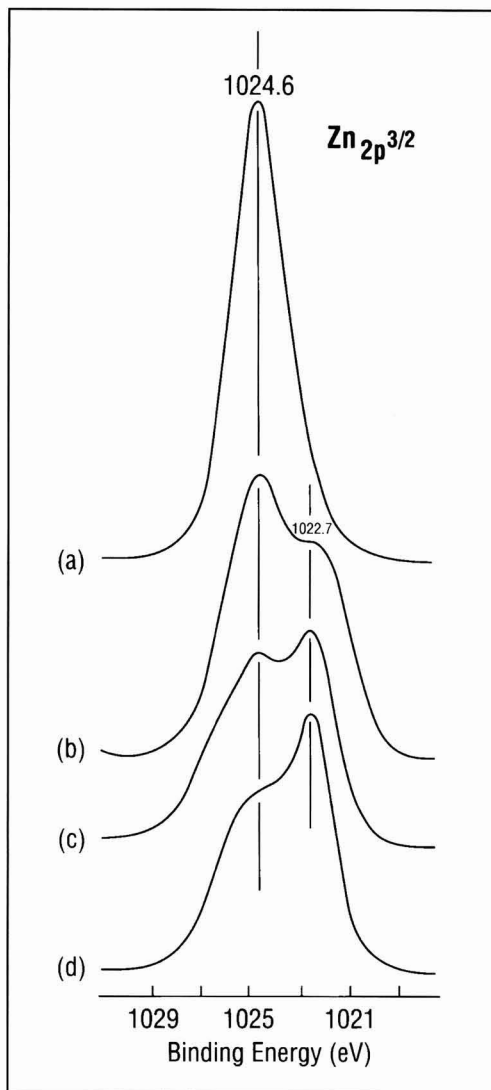


Figure 9— $Zn_{2p_{3/2}}$  region for the interfacial substrate side of (a) 0, (b) 30, (c) 60, and (d) 90 cycled PPS/Zn•Ph/steel joints

of FeS identified from the 90-cycled specimen was not only produced by the interaction of PPS with  $Fe_2O_3$  in the Zn•Ph, but also may have been derived from the reaction between S and the underlying steel. We note that there were no signals reflecting any sulfate compounds, such as  $ZnSO_4$  and  $FeSO_4$ . Inspection of the  $S_{2p}$  region strongly supported the previously mentioned information on the reaction products. Figure 11 shows the  $S_{2p}$  spectra on the interfacial PPS side removed from the Zn•Ph before and after 30 and 90 cycles. The  $S_{2p}$  region for the control showed two major

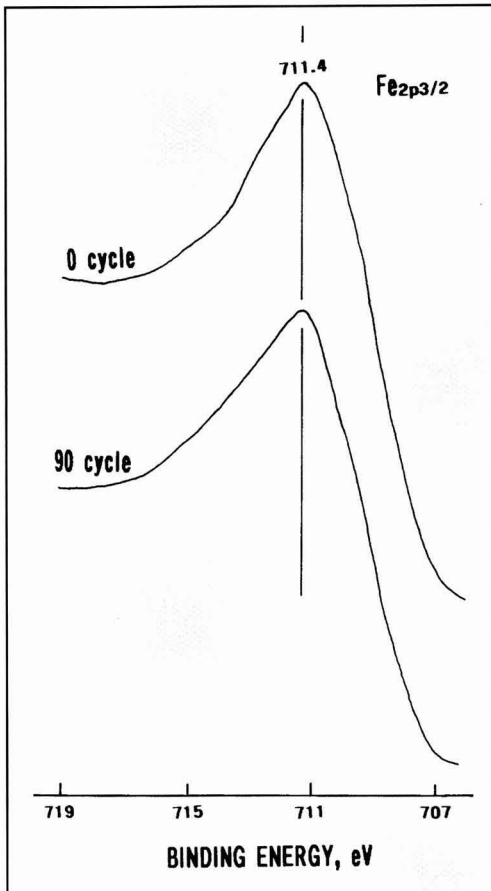


Figure 10—Fe<sub>2p3/2</sub> inspection on substrate sides for the 0- and 90-cycled specimens

components at 163.8 eV and 162.4 eV. The former peak is attributable to the S in the PPS,<sup>5</sup> and the latter reflects S in the FeS. Although no depiction of the spectra was made on the high BE sides (167.0 to 171.0 eV), there were no signals from peaks corresponding to the formation of ZnSO<sub>4</sub> and FeSO<sub>4</sub>. An additional S-compound is clearly introduced into the PPS-Zn•Ph interfacial regions after 30 cycles. From the reference peaks, we judged that this new peak at 161.5 eV is due to the S in the ZnS formation. This spectrum also showed that the signal intensity of S in the PPS at 163.8 eV was relatively attenuated, while an intense FeS signal at 162.4 eV was maintained. A possible interpretation for such changes in spectral features was that the interfacial PPS side is covered by these S-related reaction products. A spectrum similar to that of the 30-cycled specimen was obtained from the 90-cycled one; namely, it showed two reaction products, FeS and ZnS, generated in the interfacial boundary regions between the PPS and Zn•Ph. We believe that such sulfide-

based reaction products formed at the PPS-Zn•Ph contact zones play a major role in forming the intermediate crosslinking structure, which improves the Zn•Ph-to-PPS adhesive forces.

### Cathodic Delamination

An important question that still remains concerns the ability of such sulfide reaction products to reduce the rate of cathodic delamination of the coating films from the Zn•Ph. Because the FeS and ZnS compounds are substantially insoluble in the water and alkaline solution, we might expect that these reaction products would suppress the delamination of polymer film caused by alkali-catalyzed dissolution of Zn•Ph layers occurring at the cathodic reaction side,  $H_2O + 1/2O_2 + 2e^- \rightarrow 2OH^-$ . Although a high potential of -1.5 v versus SCE employed in this test may cause the formation of unwanted corrosion products, the delaminated areas of PEEK and PPS films from the Zn•Ph were measured for 0-, 20-, 30-, 60-, and 90-cycled specimens after eight days of cathodic tests (Figure 12). For PEEK/Zn•Ph/steel joint systems, the curve indicates that there is a low rate of delamination of PEEK in the first 30 cycles. However, an increase to 60 cycles resulted in the increase in the rate of delamination. As mentioned earlier, further extension to 90 cycles led to the delamination of film caused by esterification-induced decomposition of the PEEK structure at the interfaces. Thus, no cathodic test was carried out on the 90-cycled specimens. In contrast, the rate of delamination for the PPS/Zn•Ph/steel joint systems was progressively reduced as a function of cyclic number. The delaminated value of  $\approx 1.8 \text{ mm}^2$  for the 90-cycled specimens was  $\approx 20$  times lower than that for the control specimens. Such a dramatic reduction was due mainly to the rate of *in-situ* conversion of the Zn•Ph layers into FeS and ZnS reaction products in the PPS-Zn•Ph interfacial regions; that

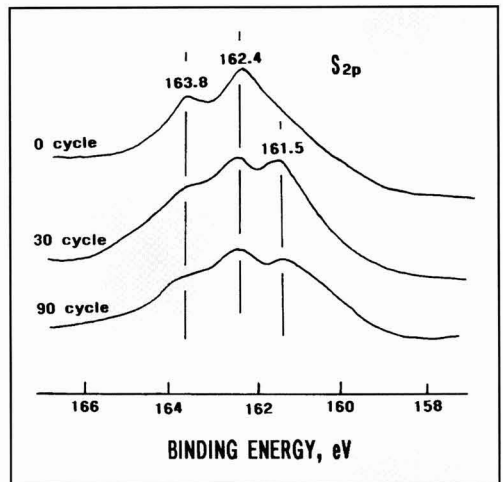


Figure 11—Changes in S<sub>2p</sub> spectrum feature on interfacial PPS side removed from the substrate as a function of cycles

is, a high conversion rate of Zn•Ph as the cycles increased caused the formation of large amounts of the FeS and ZnS reactions compounds which have no effect on the cathodic reaction-caused decomposition of the interfacial intermediate layers. Because the cathodic reaction which occurs under the coating adjacent to the defect creates a high pH environment at the interfacial boundary, the magnitude of susceptibility of the interfacial intermediate layers to alkali-induced decomposition plays a key role in suppressing the cathodic failure. Thus, it is reasonable to assume that the intermediate FeS and ZnS formations, which are insoluble in alkali solution at pH  $\approx$  13, have a high resistivity to cathodic reaction-created alkaline environments, thereby resulting in a minimum rate of delamination. This concept directly reflects why the rate of delamination for the control is higher than that of specimens after the cycles, because of the presence of a large amount of non-reactive Zn•Ph layers which are essentially vulnerable to the alkali-catalyzed dissolution.

## CONCLUSION

We can make the following generalizations from the results of the heating-cooling cyclic tests of polyphenyletheretherketone (PEEK)- and polyphenylene sulfide (PPS)-coated zinc phosphated steel specimens exposed to a harsh environment. Although no cosmetic damages, such as blisters, cracking, and peeling, were seen in PEEK coated specimens after 60 cycles (1 cycle = 12 hr at 200°C and 12 hr at 25°C), an increase to 90 caused the delamination of PEEK film from the phosphated steel. Metallographic observations suggested that the melted PEEK thermoplastic penetrates into open-surface microstructure and microfissures in the crystalline zinc phosphate (Zn•Ph) layers, thereby contributing to making a good bond at the PEEK-to-Zn•Ph interfaces, by the mechanically anchoring PEEK polymer. Despite some penetration of water into the PEEK and Zn•Ph layers, there was no evidence for delamination caused by the wedging action of the corrosion products that grew underneath the coatings. However, a shortcoming of PEEK coating was its susceptibility to hydrothermal-catalyzed esterification. Thus, esterification-induced degradation of PEEK structure after 90 cycles caused the buckling delamination of film.

In contrast, the bond structure for the PPS/phosphated steel joint systems had both mechanical interlocking and chemical bonds. The latter were characterized by representative of the formation of iron sulfide (FeS) and zinc sulfide (ZnS) reaction products yielded by chemical interactions between PPS and the Zn and Fe in the Zn•Ph layer during exposure in a wet, harsh environment. The amount of ZnS compound progressively increased with an increased number of cycles. Although such an interfacial chemical reaction led to the development of numerous microfissures in the Zn•Ph crystal layers, the combined bond structures from mechanical and chemical linkages significantly enhanced the extent of the Zn•Ph-to-PPS adhesive force, thereby resulting in a failure mode in which the loss of adhesion occurs through the mixed layers of Zn•Ph and reaction products. This finding was reflected in the fact that there was no visible damage in the specimens after 90 cycles. Also, S species from the PPS readily penetrates into the Zn•Ph layers to

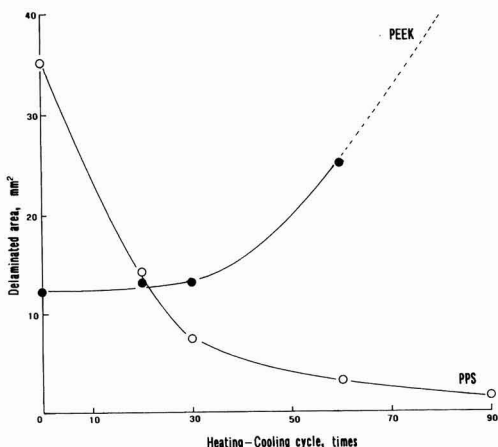


Figure 12—Rate of delamination of PEEK and PPS coating films from the substrate after cathodic tests for eight days for the polyaryl/Zn•Ph/steel joint systems as a function of heating-cooling cycles

reach the underlying steel in the first 30 cycles, and then directly reacts with the steel to form FeS. These reaction products at interfaces played a major role in inhibiting the alkali-induced delamination of PPS film from the substrates that was caused by cathodic reaction,  $H_2O + 1/2O_2 + 2e^- \rightarrow 2OH^-$ , occurring at corrosion sites in the steel, because of their insolubility in the alkali solution at pH  $\approx$  13. Therefore, such reaction products must prevent the alkali dissolution of the intermediate layers. As expected, PEEK coating systems which do not have any alkali-insoluble reaction products underwent the delamination because they were undermined by the alkali dissolution of Zn•Ph layers. Nevertheless, we believe that the combined layers of PPS and Zn•Ph significantly protect the steels from corrosion in a harsh environment.

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*These subcommittee reports are for the use of the membership of ASTM Committee D01 in continuing its work and for the interest in the activities of ASTM Committee D01. These reports are not official actions of ASTM and may not be quoted as representing any action of the American Society for Testing and Materials.*

## June 1994 Subcommittee Reports of ASTM Committee D01

The June 1994 meeting of ASTM D01 on Paint and Related Coatings, Materials, and Applications was held June 26-29, 1994 at the Sheraton Hotel, Raleigh, NC. During this time, 180 members and guests met in about 170 scheduled meetings of D01 and working task groups. The current membership of D01 is 587.

### Highlights

D01 members were pleased to have several Environmental Protection Agency (EPA) personnel attend meetings, answer questions, and provide additional information. A special joint symposium on volatile organic compound regulations and measurements, and hazardous air pollutants was held Tuesday afternoon. Both EPA personnel and industry personnel presented talks.

*D01.06*—ISO TC 35 on Paints and Varnishes has expressed interest in meeting with D01 in 1996, possibly in San Francisco in June. Increased interest in cooperation is expected due to the commonality of members, especially among global corporations.

*D01.21*—Because of EPA's urgent need for methods of measure "Hazardous Air Pollutants" (HAPs) in paints, D01.21 is establishing a new task group (D01.21.52A) to develop methods to identify and quantitate HAPs in paints with the use of GC/MS or HS/GC/MS. The task group is requesting that interested people contact S. Ramesh at 313-948-2156.

*D01.23*—A standard for measurement of contact angles of coated surfaces using a goniometer is being initiated, led by V. Scarborough.

*D01.42*—D01.42.32 was formed to develop a specification for whole paint. This discussion followed reconsideration of D 4717, a

paint specification for interior flat wall paint, developed by D01.41, but not approved by the society several years ago. Consensus specifications for whole paint would be helpful to many groups including consumers, specifiers, and regulators who need measures of paint performance.

D01.53—D01.53.03, chaired by D.A. Cocuzzi, has prepared more than 20 sets of panels which will soon be sent to exterior weathering sites. Laboratory accelerated weathering will be initiated at a later time. Gloss, color, and chalk will be monitored as a function of time.

### Awards

The following awards were presented:  
CERTIFICATES OF HONORARY MEMBERSHIP IN D01—John C. Weaver and Harry E. Ashton  
HENRY A. GARDNER AWARD—Francis Gale  
OUTSTANDING TASK GROUP CHAIRMAN—Thomas Vonderbrink  
CERTIFICATES OF APPRECIATION—Douglas Grossman, Mark Gottsegen, and Robert Morrison

### Dates and Locations of Future Meetings

January 22-25, 1995—San Antonio, TX.  
June 25-28, 1995—Atlanta, GA.  
January 21-24, 1996—Fort Lauderdale, FL.  
June 23-26, 1996—San Francisco, CA.  
January 26-29, 1997—Fort Lauderdale, FL.  
June 22-25, 1997—Toronto, Ontario, Canada.

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### DIVISION 1 ADMINISTRATIVE

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#### SUBCOMMITTEE D01.94 AWARDS AND MEMORIALS

**T.J. Sliva, Chairman**

The chairman reported that the Paul N. Gardner Co. had donated \$5,000 for an award to be presented to a member of D01 who has developed an innovative test device or technique which would lead to the improvement and reproducibility of methods currently under the jurisdiction of Committee D01. This award is dedicated to the

memory of Maynard Euverard, a long-time active member of D01 who passed away last year.

Memorial Resolutions were prepared for Leon Birnbaum, Fred Schwab, and Mark Morse.

#### SUBCOMMITTEE D01.06 INTERNATIONAL COORDINATION

**J.C. Weaver, Chairman**

ISO TC 35, Technical Committee 35 on Paints and Varnishes, expressed interest via minutes of its April 1994 meeting in Tokyo, of meeting with D01 in 1996, after technical committee 35's next meeting in the United

Kingdom in 1995. D01.06 has begun negotiations to accomplish this, possibly in San Francisco in June 1996.

Commonality of members, especially from global corporations, in their working groups and in our task groups may evolve as a practical means of achieving commonality of technical details in respective formal standards. These pairs of corporate representatives can adapt these details to the two, different editorial formats, e.g., ASTM's *Blue Book*, and balloting protocols, e.g., ASTM's *Green Book*.

"Paints and Varnishes, Annual Review, 1993" is a first ever, eight page, joint report of ISO/Technical Committee 35, CEN/Technical Committee 139, and CEN/Technical Committee 298. It includes an introduction



by Technical Committee 35 chair Prof. E.L.J. Banchen of Akzo and an insert list of 14 standards in 36 parts published in 1993. Copies of the report are available.

### **SUBCOMMITTEE D01.08 ENVIRONMENTAL CONCERNS**

**E. Praschan, Chairman**

Chairman E. Praschan called the meeting to order. Three members and 10 visitors were present. The group reviewed the existing scope and membership of this administrative committee. They agreed that the current scope adequately describes the role of D01.08 in providing information, coordination, and recommendations on environmental matters and test methods.

D01.08.01 on Durability and VOC discussed the Steel Structures Painting Council study and tentative conclusions, which indicate there is no universal relationship between durability and paint VOC content. Some of the paint types did show poorer performance at lower VOC content.

D. Leggett advised of new government proposals to further reduce the lead content of coatings. Other information concerning state regulatory activity and lead methods was made available to the group.

The group reviewed the significance and status of federal and state regulations requiring operating permits for major emissions sources. A primary requirement is developing an accurate emissions inventory for each facility, which will demand good paint material information.

Concerns were discussed about repeatability of low VOC/high water content in coatings, which is being addressed by D01.21. Methods for analysis of volatile hazardous air pollutants are also needed.

Information and magazine articles pertaining to air sampling, pollution prevention, and international environmental standards activity were distributed.

### **SUBCOMMITTEE D01.13 CONSUMER AFFAIRS**

**T.J. Sliva, Chairman**

The chairman reported that contacts had been established with representatives from the Consumer Safety Products Commission, Good Housekeeping Institute, and Consumers Union. These organizations expressed a willingness to maintain a liaison with Committee D01 and felt that the introduction of new low VOC coatings may necessitate the development of new consumer related standards. It is hoped that future contacts will be established with ASTM Committee F-15 on Consumer Products and ISO/Technical Committee 35 by the time for our joint meeting in 1996.

### **SUBCOMMITTEE D01.15 LECTURES AND SYMPOSIA**

**T.J. Sliva, Chairman**

The chairman reported that a mini-symposium would be held at the January 1995 meeting on rheology. R. Watkins, of Bohlin Reologi, will present a talk on high shear viscometers, their abilities to measure pseudoplasticity at varying temperatures, and shear rates and the use of this type of instrumentation to predict flow, leveling, and sag properties. J. Peters, of BYK-Gardner, will present a talk on low shear viscometers, their proper use, interpretation of test results, and calibration procedures.

The chairman reported that the mini-symposium on Paint Applicator Tools scheduled for the January 1996 meeting may have new presenters. A task group was set up under D01.61 to discuss alternate talks. G. Harsch, of the Wooster Brush Co., agreed to present a talk on paint brushes. C. Martin, of T.S. Simms Co., or J. Price, of EZ Paint, will present a talk on rollers. F. Burns, of EZ Paint, will serve as moderator.

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## **DIVISION 20 RESEARCH**

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### **SUBCOMMITTEE D01.20 QUALITY ASSURANCE AND STATISTICS**

**R.K. Morrison, Chairman**

*D01.20.01—Interlaboratory Testing—* An interesting discussion was held on the various merits and problems of D 3980 and E 691, both standard practices for interlaboratory studies. This group is charged with the responsibility of comparing the two practices, in (1) the ease of understanding and using the practice, and (2) the validity of the results obtained. To make these evaluations possible, each cooperator has a copy of the software programs involved with each method, and will be given data sheets to use with the software.

*D01.20—*A discussion was held on the direction that ISO 9000 is moving with the new revisions. The new documents will have numerous requirements for the implementation of statistical techniques, and the possible ramifications that this will have on D01 members was debated. Another topic discussed was the need to quantify the capability of a method, which by its nature, yields attributes data (such things as counts, pass/fail, etc.). Normal process capability methods cannot be used with this type of data, but several members of D01 have expressed a need to have a way to know how good this type of method may be.

### **SUBCOMMITTEE D01.22 HEALTH AND SAFETY**

**J. Benga, Chairman**

*D01.22.01—Flammability and Combustibility—*R. Osterman, Chairman, lead the discussion related to the use of "Setaflash" in the title of D 3278, "Flash Point of Liquids by Setaflash Closed Cup Apparatus." In general, the ASTM Form and Style Guide does not allow the use of trademarks in the title of methods, unless the trademark could be considered to be in the public domain. Since there is still some confusion about this, D01.22.01 agreed to withdraw the title change to D 3278 from ballot and deal with this issue at a later date. We will acknowledge the holder of the trademark as an editorial change to the method. The status of the round-robin study is placed on hold due to lack of qualified participants and equipment at this time.

R. Montemayor gave a brief overview of some D02 activities, especially as they are related to the measurement of flash point of viscous fluids.

*D01.22—*R. Osterman volunteered to be Secretary for D01.22. Discussion focused on the lack of active members in D01.22 with specific expertise in the area of flash point and combustibility. In light of this, it was suggested that D01.22 turns over jurisdiction of the flash point methods to D02. After considerable discussion, it was decided to keep these methods under the jurisdiction of D01.22. Even though D02 has the expertise and the willingness to oversee these methods, the lack of control over the methods and moving them to a volume other than the ones dealing with paints, would not be in the best interest of our membership. The flash point and flammability methods will continue being overseen by D01.22, but we do need active participation to ensure that these methods are technically sound and up to date. The subcommittee reaffirmed the recommendation of D01.22.01 that the revision to D 3278 be withdrawn from the ballot.

*D01.21.10—Lead Paint Policies—*J. Weaver, Chairman, welcomed 12 members and three guests to a wide ranging discussion of the current proliferation of lead-based paint abatement politics and enterprises. Especially welcomed was guest Mike Beard, of U.S. EPA, AREAL, who is Vice Chairman of Committee D22 on Sampling and Analysis of Atmospheres. Mr. Beard was the principle organizer of the July 1993 Boulder, CO conference on "Lead in Paint, Soil and Dust," sponsored by D22 along with D01.D18, and E06. In addition, he is the principle editor of *ASTM STP 1226* to be published soon which contains most of the 30 plus papers presented at the conference.

ASTM E06.23 on "Abatement of Hazards from Lead in Structures" may have in

print by autumn 1994 a special compilation of the 20 or more standards it has developed. It may include relevant standards from other committees such as D22 on Atmospheres and D01.

A new ASTM lead hazard activity is the offer by E50.03 on environmental assessment to supplement the E06.23 program with legalistic standards on real estate liability and management. Repartee on this *SN Letters*, July, pp 10-11, by P.R. Erville and T.J. Schruben, focuses on CARE of public and private housing and liability insurance thereon. Dr. Weaver views with alarm this narrow focus on care of a structure while continuing to neglect the care of the small child at risk under the dust and dirt continuously tracked and wafted into a home from soil long contaminated by the 50-years accumulation of seven million tons of lead from gasoline across the United States. This lead in dust is far more ingestible by a child than is adherent wall paint containing little if any measurable and soluble lead.

Dust origin: a new current study in Cleveland, OH, begins to reveal by mineralogy that lead bearing dust associated directly with high blood levels in children shows patterns of silicate and other minerals like those in nearby soil. This is consistent with a City of Cleveland, May 1994 seminar on current programs featuring: (1) Thomas Splitter, of EPA, on "Lead in Soil and Dirt" and (2) Shirley Scott, Licensed Practical Nurse, on "Educating Parents in Prevention of Lead Poisoning."

Cleveland's \$3.8 million grant from the HUD Title X, \$44 million largess on nationwide lead-hazard abatement can pioneer a shift in HUD emphasis toward constant care of the child at risk by frequent washing of the child and its immediate surrounding of dust and dirt, and away from the 1970's dogma of "lead-based paint," politically correct while technically false. Competition for those \$44 million will be intense between abatement contractors, lawyers, inspectors, certified analytical laboratories toxicologists, and hygienists. Better that most of it go to LPNs who visit homes of children at risk and teach hands-on hygiene.

CDC's imminent new report is rumored to show a continuing decline in blood lead levels in children, consistent with the J.L. Annett plot of the close correlation in 1976-1980 between declines in blood lead levels and lead use in gasoline, and this task group chairman's optimistic forecast that by the year 2000 the average blood lead in children will have dropped too low to interest federal and state legislators in further proliferation of lead laws, while those seven million tons of gasoline lead naturally dissipate slowly from dust and dirt.

Speciation and bioavailability of the various chemical and physical forms of lead have been a glaring lack from 1970 or earlier in the U.S. federal and state laws and regulations of lead in dwellings. They con-

tinue to ignore Peter Buseck's 1980 detailed speciation of lead compounds in atmospheric samples in Phoenix, AZ. Now, wider studies of speciation build up more evidence to refute the exaggerations in the 1970's lead-based paint dogma.

CPSC has formally recognized ASTM D01.57's D 4236, "Standard Practice of Labeling Art Materials for Chronic Health Hazards," published currently on noncompliance of imported crayons. D 4236's subsection on bioavailability will be enhanced by the new ASTM standard D 5517.

In nature, July 6, 1994, a renowned French wine made from grapes grown near heavy traffic highways is reported to contain trimethyl lead and triethyl lead from incidence in 1950 to maximums in the 1970s to almost none in 1991 in proportion to the use of lead in gasoline over those 40 years.

Lead immobilization in soils by application of hydroxyapatite is reported in two recent papers from Ohio State University. This implies use of commercial phosphate fertilizers as another remedy against exposure of lead to small children in dwelling yards and in playgrounds.

Industrial hazards of lead are recognized from ancient Rome to the present and will continue to be of high concern. K.A. Trimmer's massive tome, available from the SSPC, covers most all aspects of the industrial paint segment of lead hazards.

Lead contents, requiring remedial action in paints, dusts, soils, atmospheres, etc. need reassessment from 1971 to now. The 600 ppm (0.06%) limit of total lead in paint solids was recommended in 1971 by Barry G. King and his committee of toxicologists, was endorsed in 1972 to ANSI re its Standard Z66.1 by Paul F. Wehrle and his committee of pediatricians, and thereafter was adopted in various lead laws. Their myopic focus on only leaded paint is now discredited by wide ranging studies of lead in atmospheres, dusts, soils, etc. The high costs of chemical and physical analyses of paint versus that 600 ppm limit of lead mandates a concerted campaign by the coatings industry, including ASTM D01.21, to achieve a critical review of that 600 ppm toward more realistic limits, probably by CDC in concert with EPA in joint advice to federal and state legislatures.

*D01.21.13—Coordination of VOC Standards & Information*—K.H. Fujimoto, Acting Chairman. The task group was fortunate to have Jim Berry and Dave Salmon, and Madeline Strum, who represented the U.S. EPA, present at this meeting. Their comments, elucidations, and statements were of great help in answering the many questions raised by the attendees. The representatives of the paint industry are complaining about the many goals they have to meet to reduce their use of VOCs, but its burden isn't so bad when we look at the chemical industry. This industry has received the mandate for the next three years to have 370 plants in 38

states reduce emissions of 111 toxic substances, 88% from the 1990 levels. The requirement to cut toxic emissions by about 510,000 tons a year is part of the 1990 CAAA. Indirectly, it will trickle down to the paint industry.

In answer to a question about the test parameters used in ASTM D 2369, Mr. Berry discussed how the EPA came to accept the one hour bake at 100°C for volatiles as specified in this method. At the time the test was developed, the compromised and specific test parameters used in D 2369 gave the best precision. Perhaps for today's new paint technologies, these test parameters may not be a good compromise. The results from the round-robin on low VOC and high water containing paints contemplated by D01.21.24 will answer this question.

Complaints were heard that EPA's Reference Method 24 does not give reliable results on some specific paint products. It was suggested that the best way to approach this problem is to gather enough information to prove the point, and then approach the EPA for acceptance of a variance for Reference Method 24. In the past, these type of complaints were based on the differences between the theoretical VOCs vs. Reference Method 24 results. Our answer is that theoretical values are just that, theoretical.

The question was raised as to what method to use to determine the VOCs of inks and ink products. EPA's Reference Method 24 does not actually describe how the various types of inks are to be tested for their VOC content. It was felt that this test method was too severe a test for some inks such as newspaper inks. There are other VOC test methods such as those developed by D01.56 on Inks, but they have not been accepted by the EPA. Therefore, it was suggested that reference to ASTM D 4713 in Para. 1.4 of D 2369 be eliminated because it gives, to the reader, the concept that it is accepted by the U.S. EPA.

J. Berry was also concerned by Note 3 in D 2369 which states "If decomposition or degradation occurs, etc." He thought this appeared to be a repetition of the first sentence in Paragraph 4, until it was pointed out that someone running the test must know what to do if his product decomposes. Based on D. Salmon's comments, the last sentence in "Note 3" will be studied and rewritten, if necessary.

It was pointed out that the EPA is a public health organization under the 1970 Clean Air Act. To ensure the public's safety and decrease pollution, 189 Hazardous Air Pollutants (HAPs) have been identified. Less than 200 industries emitting 10 tons of one or 25 tons of a combination of HAPs have also been identified. After this affects the VOC goals for the paint industry.

*D01.21.14—New Publication and VOC Workshops*—K.H. Fujimoto, Chairman, announced that the second edition of ASTM

technical manual, MNL-4, "Manual on Determination of Volatile Organic Compounds (VOC) Content in Paints, Inks, and Related Coating Products," has been published and it is available from ASTM. J.J. Brezinski, Editor, has done an excellent job in bringing all the latest VOC information together.

The new manual brings us up-to-date on the:

(1) Applicability and limitation of ASTM standards approved by the EPA for the determination of VOCs in paints plus a historical perspective from the EPA's view of factors which influenced development of coating regulations;

(2) Most recently developed or revised ASTM standards and experimental procedures under development for the determination of VOCs;

(3) The Federal Reference Method 24 and Method 24A; and

(4) Tabulation of VOC emission limits for surface coating operations from EPA's Control Technique Guidelines and New Source Performance Standards.

Nineteen persons attended the May 4-5, 1994, ASTM Paint VOC Measurement Workshop in Chicago. The successful laboratory demonstrations were held at the Rust-Oleum Analytical Laboratory, Pleasant Prairie, WI. The new workshop will be held November 2-3, 1994, in San Francisco, CA, with R. Zerrudo and the BAAQMD Laboratories acting as our host for the ASTM test method demonstration. This will be followed on May 3-4, 1995, with a Paint VOC Workshop in Raleigh, NC, with J. Lamberton and the RCI laboratory acting as our host for the ASTM test method demonstrations.

*D01.21.24—Revision of D 2369, Volatile Content of Coatings*—M. Sites, Chairman. J. Benga, PPG Industries, presented data on problems encountered with high water content, low VOC coatings, especially those with low solids. Problems with high-solids coatings and powder coatings were mentioned and D 2369 may not be appropriate for measuring percent volatiles of these coatings. R.J. Wherley, of the Glidden Co., issued a document in 1981 in which he used a computer program to determine the effect of the precision limits (i.e., VOC confidence limits), of each of the separate test methods used to determine VOCs. He concluded that, in general, precision decreases with increasing coating density, increasing volatile content and/or increasing water content. Mr. Benga will give a presentation on this topic at the EPA/ASTM Inter-Face Symposium.

As a result of a discussion in D01.21.80 last year, it was pointed out that although D 2369 (and other methods used to determine VOCs) has been criticized as being inadequate for today's low VOC paints and coatings, no data has been submitted to D01.21 to verify these complaints. D01.21.24 decided to take action and find out whether D

2369 can accurately measure the low VOCs and high water containing paint products on the market today. There was a good response when collaborators and samples were requested. Eight to 10 laboratories expressed interest in participating in the round-robin testing. D01.21.24 decided this round-robin will include only coatings which are low in VOCs and those containing a high percentage of water. In addition to determining percent volatiles using D 2369, participants will be asked to determine density, D 1475, and percent water with D 4017 and/or D 3792, and calculate the VOCs. This information will be helpful in determining the efficacy of EPA's Reference Method 24.

The question was raised whether multi-component systems should be included in this round-robin. The task group decided to postpone this problem until a later date. Persons interested in this subject, especially extending the induction period beyond one hour, should contact Mary E. Sites at 617-861-6600, ext. 2360.

The latest revision of D 2369, which will be balloted in 1994, includes the removal of any reference to D 4713, "Test Method for Nonvolatile Content of Heat Set and Liquid Printing Ink Systems." The final editing of Para. 1.4, Note 3 and Para. 2 is not finalized. Reference to 2-ethoxyethyl acetate in Para. 6.4 will be removed since this solvent is no longer used and it is classified as a health hazard.

J. Berry and D. Salmon participated in the discussions of (1) the basis for the "minus water" concept in comparing solvent- and water-reducible paint systems, (2) the difficulty in obtaining consistent results with low VOC paints, and (3) at what level of VOC should the "minus water" concept be dropped.

*D01.21.24A—Ion Chromatography, Electrocoat Bath Samples*—K.H. Fujimoto presided at the meeting in the absence of Chairman J. Furar. The "New Standard Guide for Determination of Anions" received a negative from P. Guevin on a D01 ballot. The negative, which had five parts, was found not persuasive. Each part was discussed separately as summarized below. Mr. Guevin stated that the guide lacks guidance and does not provide a method for measuring nitrate anions. The task group agreed this is not necessary since this is a guide and not a method or practice. He noted that terms such as permeate and ion chromatography are lacking, but the task group agreed they were not necessary. Persons consulting this "guide" would be familiar with these terms. Mr. Guevin stated that the types of apparatuses should be added to educate the reader on which ones are applicable. The task group agreed this is not necessary since this is a "guide" not a test method. He noted that the form and style of ASTM standards required footnotes to inform the reader where referenced documents can be found. The task group stated these will be inserted,

where necessary, by the editor. And finally, he wondered whether there were technical articles which could be referenced. The task group had found none. (However, the subcommittee decided that the guide should be reviewed before being balloted.)

*D01.21.26—Review of D 2697, Volume Nonvolatile Matter in Clear or Pigmented Coatings*—In the absence of the Chairman, K.H. Fujimoto acted as temporary Chairman. Copies of the test method for "Percent Volume Nonvolatile Matter in Clear or Pigmented Coatings Using a Helium Gas Pycnometer" were distributed for information and comments. This latest revision of the test method has been submitted for concurrent D01.21 and D01 Ballot.

Copies of the data from the last round-robin, which was received and reviewed by the temporary chairman, were distributed. Two sets of data had been tabulated: the paint film density, and the %VNV values calculated from the paint density. Full ASTM E 691 precision data could not be presented since the work was still incomplete. From the data, it appears the precision of the paint-film measurements is about six times better than of the calculated %VNV values. This discrepancy appears to be caused by either the wet paint density measurements or the percent nonvolatiles measurements, or both. Both values are needed to calculate the %VNV. It will be recommended to the chairman that he review all the data used to calculate the %VNV.

J. Berry stated he is anxious for the development of the helium gas pycnometer method for determining the %VNV of all paints. Once a reliable test method is developed, he would like to have all VOCs reported as mass VOC/unit volume applied solids.

*D01.21.27B—New Approach to Determine VOCs*—R.K.M. Jayanty, Fujimoto acted as temporary chairman in the absence of Mr. Jayanty. To understand the status of this task group, the minutes of January 25, 1994 were reviewed. In summary, a proposed method received nine negatives in a 1992 D01.21 ballot. After no apparent action was taken on the method over a one-year span and, Mr. Jayanty informed the task group that (1) EPA has provided funds for RTI to develop a simplified volatilization chamber, and (2) Midwest Research Institute is to conduct an interlaboratory study of the method and he would report the results at a meeting.

Although there were several technical problems with the proposed method, the need for an improved method is so great that the task group decided to: (1) resurrect the nine negatives and study them to ascertain the bases for the objections, and (2) see what can be done to improve the method since it does show merit.

*D01.21.51—Determination of Formaldehyde in Paints*—D. McCuum, Chairman,

reviewed the January minutes. The results of experiments measuring evolved formaldehyde by the colorimetric acetylacetone approach and/or liquid chromatography (LC) of the processed solutions were discussed. The formaldehyde was collected in tandem impingers by passing nitrogen over aqueous standards or paint samples in test tubes. The limited experiments indicate that the extra steps of using the LC does not provide any significant advantage over the different colorimetric measurement. Potential interferences were discussed. Acetaldehyde was reported to produce false positive color, but it required a lot more acetaldehyde than formaldehyde to produce the same absorbance. Some general discussion included: reported contamination of "blank" sampling tubes used in air sampling using OSHA Method #52, apparent decrease in formaldehyde levels in the headspace of paint samples with time, and the difficulty of measuring free formaldehyde due to various equilibria.

A preview of determining free formaldehyde in latex by the emulsion polymer roundtable (see D01.33.26) was presented by J. Lamberton. They have developed an LC post-column derivatization method that appears to be better than the LC pre-column derivatization method discussed at the last meeting.

The final item was a discussion on initial plans for a mini-round-robin using in-house samples. Four labs (PPG, Reichhold, Hüls, and Glidden) have agreed to test the evolved formaldehyde/acetylacetone colorimetric approach by generating data for standards, blank and "spiked" paint samples made at each location. Results obtained will be reported at the next meeting.

*D01.21.52—Paint Solvent Analysis by Gas Chromatography*—J. Benga, Chairman, reviewed the minutes from the January 1994 meeting, and noted the lack of activity by the task group. The general agreement was that we need to be more diligent in our efforts. A technical discussion related to modifications of the proposed capillary gas chromatographic (GC) procedure followed. It was suggested that the head space/GC/MS and headspace/GC method development be run in parallel with the direct injection method. It was agreed that both options would be pursued. The need still exists for collaborators to help decide what method should be worked on first, headspace/GC or capillary/direct injection. The chair is soliciting interested parties to participate in a round-robin and to develop a test method for the solvent analysis of paint and coatings. Interested parties should contact Mr. Benga at 412-492-5511 or K.H. Fujimoto at 313-788-9707 immediately.

*D01.21.54—Revision of D 4017, Water in Paints and Paint Materials by Karl Fischer Titration*—R. Osterman, Chairman, reviewed the decision made at the January meeting to include the methanol extraction

method which uses Hydranal<sup>®</sup> reagents to determine the percentage of water present. This method was developed by W.C. Golton, of E.I. DuPont, as an alternate method to handle samples which do not yield reproducible results.

The precision statement of the current method will be revised to reflect the excellent results obtained from a previously run round-robin on four water reducible paints. This data has been published in the second edition of ASTM's Manual on Determination of Volatile Organic Compounds (VOC) in Paints, Inks, and Related Products. The precision statement developed from another round-robin conducted by J. Benga on paints containing 50-75% water, and using Hydranal reagents, will be incorporated into the alternate method section. The revision will be submitted for concurrent D01 and D01.21 Ballot.

A letter received from T. Sliva was discussed. It concerned ways to buffer materials possessing a pH above eight before determining their water content. It was the consensus of the group that a reduction in sample size may solve the problem. However, the concept will be examined to ascertain if it has merit.

The Cal Poly Method for determining the water content of a paint product was discussed. Prof. Max T. Willis has developed, what he claims is a more accurate method than D 4017, to measure water in paints. In the proposed method, 1-methoxy-2-propanol (MPA) is mixed into the paint specimen, thoroughly dispersed, and the water/solvent/MPA is distilled/extracted. Aliquots of the distillate are tested for water content using the Hydranal Karl Fischer Titration Method. However, the new precision statement, which is to be placed in D 4017, for results obtained from the round-robin using Hydranal, surpasses the results reported in the Cal Poly Method.

*D01.21.56—Revision of D 3960, Practice for Determining Volatile Organic Compounds (VOC) of Paints and Related Coatings*—M.E. Sites, Chairman, reviewed the minutes of the last meeting. Copies of the latest revision, D 3960-93, which has been published in the *ASTM 1994 Book of Standards*, were distributed to the attendees. The development of D 3960 and the constant need to bring it up-to-date were discussed. The 1991 revisions incorporated various comments and suggestions from EPA personnel. These include changes in the symbols used, definitions of terms and modifications made on the equations for calculating VOCs so that they were, essentially, identical to those adopted by the EPA and those used in the federal regulations.

It was recommended that Method D 5403, "Standard Test Method for Volatile Content of Radiation Curable Materials," be referenced in D 3960. While this standard is not referenced in Method 24, the

EPA has given it special approval until it adopts a method.

A discussion followed concerning the possibility of referencing D 3960 in the EPA's Reference Method 24. This practice was not adopted until after Reference Method 24 was issued. J. Berry stated that one of the reasons D 3960 has not been referenced is the constant revisions D 3960 has undergone in the last few years. It is still the hope of this task group that D 3960 will be referenced in a future revision of Reference Method 24.

*D01.21.08—Exploratory Analytical Chemistry*—K.H. Fujimoto, Chairman, reported that D01.21.24 has not started its round-robin to evaluate today's low VOC and high water content paints and coatings. Samples and collaborators are being solicited. One of the questions asked was how low in VOC and how high in water content the test paint samples should be. The task group could not give a definite answer. It will be left up to the chairman.

At the January meeting, the task group decided that since D01.21.52 was developing a GC test method to quantitate solvents in paints an additional task group to develop test methods for the identity and quantitation of HAPs present in paints by use of headspace (HS)/gas chromatograph (GC)/mass Spec (MS) or GC/MS should not be formed. However, from the chairman's conversations with the EPA and state regulatory bodies, we do not have the luxury of plodding along concentrating only on the use of the GC as planned in D01.21.52. Unless more active steps are taken to run a parallel group to study and to develop test methods for identifying HAPs in a paint matrix, outside parties will develop the tests for us. Time is of the essence. Under Title 5 and Title 3 of the 1990 CAA Amendments, HAPs must be identified and quantitated. Thus, D01.21.80 voted to form a new D01.21.52A to develop test methods to identify and to quantitate HAPs in paints with the use of GC/MS or HS/GC/MS. Due to the lack of a volunteer to chair this group, Mr. Fujimoto proposed to approach S. Ramesh, of BASF-Southfield, to take over this assignment with Mr. Fujimoto acting as Vice-Chairman. Recommendations to form D01.21 will be made to D01.21. Those laboratories interested in participating in this very interesting and challenging endeavor should contact S. Ramesh at 313-948-2156. The chairman has been informed that the EPA wants all of the HAPs present in a paint to be extracted and identified with the use of a GC, that is, just identify the peaks and quantitate them. No one knew how this could be done without also getting degradation products and extraneous reaction products.

K.H. Fujimoto also reported that S. R. George, who was working for Prof. Frank N. Jones, of Eastern Michigan University, has developed a "so-called" improved ver-



sion of ASTM D 3792, "Test Method for Water Content of Water-Reducible Paints by Direct Injection into a Gas Chromatograph," which she would like D01.21 to evaluate and to comment on. As agreed with Prof. Jones, copies of this method were distributed to the attendees for comments and evaluations. It was the general consensus that (1) the use of methanol for an internal standard is poor since most automotive and some industrial finishes contain methanol, (2) the use of the correct amount of internal standard to match the water content of the test specimen is good routine GC technique and it is used constantly by those using D 3792 even though it is not spelled out by the method. [This technique is stressed at the ASTM Paint VOC Measurement Workshop held bi-annually by D01.21 and perhaps it should be incorporated into the method] and (3) the proposed column may give better results than the Porapak Q, packed column, used in the present method. This should be checked out. The group extended its appreciation to Prof. Jones for the many suggestions on improving D 3792. At this time, D01.21 is studying the proposed changes and will decide whether there is enough interest to activate the task group.

It has been almost three years since D01.21 held its successful "Symposium on Analysis of Paints and Related Materials: Current Techniques for Solving Coating Problems" in Pittsburgh, PA. Considering it will take us about two years to prepare for the symposium, we should get started as soon as possible for another one. The steps necessary to have a successful symposium were discussed such as finance, getting the speakers, sending out flyers, publicity, and hotel preparations. No positive action was taken at this meeting.

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

**P.R. Guevin, Jr., Chairman**

*D01.23.10—Adhesion*—G.L. Nelson, Chairman, reported the revision of D 3359, "Test Methods for Measuring Adhesion by Tape Test," successfully completed D01.23 ballot. Several editorial suggestions were reviewed and will be included before the item proceeds to D01 ballot. The chairman identified three items that should be included in the next revision of the method; namely, a revised Figure 1, showing loss of adhesion of a coating, a table to show performance properties for commonly used tapes as a second appendix and a footnote to Para 12.2.2. to advise that some work has been successfully done using a 5 mm grid to scribe organic coatings with dry film thicknesses greater than 5 mils.

H.L. Novak, of United Technologies USBA, Kennedy Space Center, submitted a

report on grit blasting as a means of scribing coatings for evaluation of adhesion using D 3359. The report was distributed. A presentation of the paper is expected at the next meeting.

*D01.23.12—Dry Film Thickness*—S.K. Boocock, Chairman. The meeting was chaired by R.F. Weaver, of SSPC, in the absence of the chairman. Mr. Weaver reported the revision to D 1400, "Test Method for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Metal Base," completed D01 ballot and will now proceed to society ballot.

In other business, the task group discussed the parameters of the round-robin required to update the reproducibility and repeatability (precision and bias) statements in both D 1186 and various instrumentation used for slip resistance testing. J. Cockrell, of SKL Enterprises, had agreed to evaluate three porch and deck enamels using the Mark II instrument and report his results by the January meeting. These coatings were previously tested under both wet and dry conditions using the Ergodyne Slip Resistance Tester. Dr. Cockrell also gave an overview of the meetings of Committee F-13 on Safety and Traction of Footwear held the previous week.

*D01.23.16—Water Vapor Transmission*—T.J. Sliva, Chairman, reported revisions to D 1653, "Test Method for Water Vapor Transmission of Organic Coatings Films," successfully completed society ballot. The chairman led a discussion on two possible directions the task group might investigate; namely, incorporation of an inverted cup method as outlined in ASTM E 96 and development of a method to compare moisture vapor transmission rate using the Mocon Tester vs D 1653. There was no interest by the task group at this time.

H. Ashton reported on the *Journal of Testing and Evaluation* article, "Infrared Technique as a Research Tool for Measuring Water Vapor Transmission of Roofing Membranes," where moisture vapor transmission was measured using an infrared detector.

It was the decision of the task group, as no further work was anticipated on the method at this time, to temporarily declare the task group inactive.

*D01.23.19—Drying Time*—T.J. Sliva, Chairman, distributed Draft #2 of the proposed "Standard Test Method for Drying, Curing, or Film Formation of Organic Coatings using Mechanical Drying Time Recorders," for review. Draft #1 received two negatives from the recently completed D01.23 ballot. The negatives were reviewed by the task group and found to be persuasive. The method was revised by the chairman to include their comments and suggestions and will be resubmitted to D01.23 ballot before the January meeting. The re-

sults from the first round-robin using the straight line drying time recorders were distributed and reviewed. The cooperators evaluated four coatings, including an architectural latex flat and alkyd paint, alkyd flat floor finish, and an air dry white acrylic lacquer, each of varying drying times. The cooperators also ran drying time in accordance with procedures outlined in D 1640 at the same time. While the results from three of the four cooperators looked promising, it was the decision of the task group to run an additional round-robin. The chairman will report the results of this testing at the January meeting.

*D01.23.20—Exploratory Research*—P.R. Guevin, Jr., Chairman, discussed the round-robin conducted by D. Beamish, of DeFelsko, using the PosiTector<sup>®</sup> 100 C2 and described the work done with this ultrasonic thickness instrument to measure the thickness of three different organic coatings at three different thicknesses on concrete non-destructively. Five laboratories participated in the round-robin. The initial data looks very promising. The chairman will analyze the data and will prepare a draft of a test method to be submitted to the task group members for review by the January meeting.

Contact angle measurement was then discussed. The chairman reviewed input from NIST and PPG Industries pertaining to contact angle measurement to ascertain interest in the task group in writing up a method whereby contact angle measurements would be used to determine the extent of surface preparation prior to painting. V. Scarborough, of Thompson & Formby, has agreed to chair D01.23.21 on Contact Angle Measurement. P. Guevin will provide technical assistance and guidance in preparing a test method for review.

## **SUBCOMMITTEE D01.26 OPTICAL PROPERTIES**

**R. Kumar, Chairman**

*D01.26.02—Color Measurement*—R. Kumar, Acting Chairman, reported that D 5531-94, "Guide for the Preparation, Maintenance and Distribution of Physical Product Standards for Color and Geometric Appearance of Coatings," passed the society ballot. Former task group chairman R.T. Marcus has resigned and a new chairman will be elected before the January 1995 meeting.

D 1544, "Test Method for Color of Transparent Liquids," needs revision. J. Peters will work with others to establish round-robin experiments to arrive at numerical data for glass filters. Anyone interested in this project, please contact Mr. Peters, at BYK Gardner Inc., 800-343-7721.

*D01.26.11—Gloss and Goniophotometry*—A.F. Rutkiewicz, Chairman, reported that a pro-



posed new standard, "Test Method for Instrumental Measurement of Distinctness of Image Gloss of Coating Surfaces," passed D01.26 ballot. However, there were several comments. One comment required a technical change of the angular values of Table 1, Method A, "Angles and Dimensions of Source Image and Receptors." The test method will also be revised to include references to the round-robin report prepared by M.P. Morse. A copy of this report will be forwarded to ASTM for cataloging. These changes will be balloted concurrently in D01.26 and D01.

Methods D 523, "The Method for Specular Gloss," and D 4449, "Test Method for Visual Evaluation of Gloss Differences between Surfaces of Similar Appearance," will be submitted for reapproval.

**D01.26.24—Tinting Strength**—R. Morrison, Chairman, reviewed the ballot returns on D 387, "Test Method for Color and Strength of Color Pigments with a Mechanical Muller." Although a negative was found persuasive, the standard is about to be dropped from the *ASTM Book of Standards* because revision or reapproval is overdue. The negative voter will be contacted to see if he will withdraw his negative, with the assurance that the points raised will be addressed as soon as possible.

The new test method for "Evaluating the Relative Tint Undertone of Titanium Dioxide Pigment" received four negatives and several comments on D01 ballot. Some of the negatives were found persuasive, and the method has been withdrawn from ballot, in order to make the required corrections.

**D01.26**—The subcommittee invites new members to work on revision of old standards and some new standards related to optical properties. Many of the standards need to be updated due to changes in technology. Anyone interested in active involvement in this subcommittee, please contact Romesh Kumar, at Hoechst Celanese, (401) 823-2161.

## **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," which will include a new step gauge for cleanliness. The preliminary diagram of a lighting box for improved viewing of the dispersion pattern was discussed and will be revised for inclusion in the method. Also to be included are some suggested techniques for reading the gauge in order to improve the precision of the method. The significance and use section will point out some of the limitations of the method.

**D01.24.33—Odor Evaluation**—D.E. Durr, Chairman, discussed the first draft of the proposed "Standard Guide to the Detection, Identification and Characterization of the Odors of Paints, Inks and Related Materials." Both A.F. Rutkiewicz and D.E. Durr, will prepare a more detailed version of the draft for discussion at the next D01 meeting.

## **SUBCOMMITTEE D01.27 ACCELERATED TESTS FOR PROTECTIVE COATINGS**

### **D. Grossman, Chairman**

**D01.27.02—Water Tests**—D. Grossman, Chairman. A D01 ballot received no negatives on a new Method B for D 2247, "100% Humidity." The draft will go on to society ballot. This new procedure gives instructions for generating humidity by bubbling compressed air through an unheated water bath, while maintaining temperature by a heated water jacket around the walls.

The chairman contacted G. Gardner of D01.46 Industrial Paints about possible changes to D 870, "Immersion," but D01.46 plans to write their own separate document pertaining to chemical immersion, not pure water immersion as found in D 870. This task group will now become inactive until further work is required.

**D01.27.04—Light and Water Exposure Apparatus**—L.E. Thieben, Chairman, reported that the new standard for "Xenon Exposure Apparatus" will be submitted for D01.27 ballot incorporating "relative spectral irradiance" data for indoor and outdoor exposure at various bandpass wavelengths.

W. Ketola drafted revisions for D 822, "Open Flame Carbon Arc," and D 5031 "Enclosed Carbon Arc," with sections on scope, significance, and precision conforming to related exposure standards. J. Evans suggested adding a safety caveat about wearing a respirator when changing carbons, and he will send appropriate wording to W. Ketola to add to the draft, which will then go to D01.27 ballot.

**D01.27.10—Accelerated Outdoor Weathering**—J. Robbins III, Chairman, led a discussion of the D01.27 ballot returns of D 4141, "Standard Practice for Conducting Accelerated Outdoor Exposure Tests of Coatings." One item on this ballot was to change Section 5.3, which would make the use of reference materials optional instead of mandatory. This proposed change received three negative votes which were found persuasive. One additional negative vote and three comments were received on other areas of the document. This document will be revised and sent to concurrent D01.27/D01 ballot.

**D01.27.17—Evaluation of Weathering Effects**—M. Crewdson, Chairman, noted that

D 714, "Evaluation of Blistering," is overdue for reapproval, and in danger of automatic withdrawal. It will be submitted for concurrent D01.27/D01 ballot for reapproval. After reapproval, a draft with necessary revisions will be prepared for balloting.

D 4214, "Evaluating Degree of Chalking of Exterior Paint," is due for major revisions. R. Morrison will draft a revision for D01.27 ballot.

The task group decided to draft a new combined evaluation standard encompassing D 660, "Evaluation of Checking," D 661, "Cracking," D 662, "Erosion," and D 772 "Flaking." M. Crewdson and J. Martin will collaborate on the draft.

The second instant round-robin on evaluation of blistering and flaking was postponed. It will be discussed at the next meeting.

**D01.27.18—Outdoor Exposure Tests**—J. Martin, Chairman, reported that D 1014, "Outdoor Exposures of Paints on Steel," received no negatives on D01.27 ballot. Comments from H. Ashton were reviewed. The draft will automatically proceed to D01 ballot. Upon further discussion it was decided to rewrite D 1014 to include exposure on metals in general, not just limited to steel. J. Martin, B. Boni, and L. Thieben will redraft and it will be balloted at D01.27 level, after the current draft finishes D01 ballot.

**D01.27.29—Test Substrates**—D. Grossman, Chairman, drafted a revision of D 609, "Steel Test Panels," to address storage of bare steel panels with VCI will be sent to D01.27 ballot. A revision to D 3891, "Glass Test Panels," was drafted by L. Pattison and will be sent to D01.27 ballot. G. Rommal will draft a revision to D 2201, "Zinc Coated Steel Panels for Paint Tests," that updates Appendix XI. Characterization of Zinc Coatings, to show many of the newer metric designations used in the auto industry. The draft will go to concurrent D01.27/D01 ballot.

**D01.27.30—Corrosion Tests—Automotive**—W. Van der Linde, Chairman, assumed leadership of D01.27.20 upon F. Lutze's resignation. The proposed new standard, "Outdoor Salt Accelerated Corrosion Test," needs to be revised by W. Van der Linde for rebalot. A round-robin will be organized by W. Van der Linde, using auto refinishing systems. Six cooperators have agreed to perform exposures, which are estimated to last three months.

The new Guide to Laboratory Cyclic Corrosion Tests (CCT) will be revised by P. Brennan for D01.27 ballot. Data from the SAE corrosion round-robin was reported by S. Grossman. Several CCT show reasonable correlation with on-vehicle exposure. Those interested in copies of the data can contact S. Grossman at (216) 835-8700.

*D01.27.31—Corrosion Tests—Non Automotive*—S. Boocock, Chairman. R. Weaver chaired in S. Boocock's absence. He reported progress on sample preparation for the huge round-robin exposure test for both industrial maintenance paints and coil coatings. For the industrial maintenance paints, SSPC has prepared about half of the 2,000 panels. Ten different paint systems will be used. Two hundred replicates of each system will be prepared, but some of these will inevitably be rejected for high or low coating thickness. Five replicates will be used for each exposure. Test panels are 4 in. x 6 in. x 3/16 in., hot rolled steel (rust grade A & B), blast cleaned to SSPC-SP5 white metal, profile of 1.5 mils. The panels will have a 4 in. vertical scribe. Panels edges will be dipped in a suitable coating. Panels should be ready for exposure in September 1994.

For the coil coating samples, D. Grossman reported that considerable experimentation has been done in conjunction with G. Rommal on impact and scribing procedures to get repeatable dimples and scribes. A suitable impact height and die has been identified for each of the eight coil coating systems. Also, a scribing apparatus that allows a calibrated pressure and stylus angle has been investigated, to produce repeatable scribes that go through the paint, but not through the galvanizing. Several hundred replicates will be scribed and dimpled by August. G. Rommal pointed out the need to keep the shearing burrs consistent, with burr up always on one side, and burr down always on the other.

The following sites will be used for atmospheric exposures of both the industrial and the coil coatings: Kure Beach, 25 m lot; Kure Beach, 250m lot; Coke Works near Pittsburgh (moderate industrial); Neville Island near Pittsburgh (mild industrial); Mellon Institute Roof, Pittsburgh (urban); and South Florida Testing Labs, Naples, FL. (a duplicate marine site).

A number of round-robin issues must be decided at the next meeting. G. Rommal will write a proposal for evaluating the coil coatings. Also, accelerated exposure candidates need to be evaluated and allocated samples.

D. Grossman reported that the "Prohesion" cycle has been approved by Committee G01 aa Annex A5 to G 85, "Modified Salt Spray Tests." This cycle consists of one hour at room temperature and one hour dry at 35°C, with a dilute electrolyte of 0.05% CaCl and 0.35% ammonium sulfate.

A revised draft of the new standard for "Cyclic Salt Fog, Dry Off, and UV/Condensation" will be sent to D01.27 ballot. This practice calls for one week in a chamber running the new G 85 Annex A5 followed by one week in a G 53, "Fluorescent UV/Condensation" chamber, for a total of six weeks.

W. Van der Linde discussed the mini round-robin results of the prohesion test using five automotive paints. Three tables of data showing scrobe undercutting, standard deviation, coefficient of variation, etc., were distributed. Copies can be obtained from D. Grossman at (216) 835-8700. L. Pattison will perform statistical analysis of the data per E 691. The task group is looking for suggestions on how to assess and address the variability between different manufacturers of prohesion-type cabinets.

## SUBCOMMITTEE D01.28 BIODETERIORATION

**M.C. McLaurin, Chairman**

*D01.28.01—Package Stability*—M.C. McLaurin, Chairman. The minor revision to D 2574-93 was balloted and approved with no negatives. Three comments were made with various editorial corrections, including SI unit consistency and conversion. After the meeting, these corrections were reviewed with L. Gutman and will be incorporated with the revision at the next printing. The task group will review Section 5.3 which contains some confusing text and does not explicitly delineate how to do the *Enterobacter* sp. broth culture in January.

*D01.28.02—Rapid Determination of Enzymes*—C.W. Vanderslice, Chairman, updated the group on work done in his lab using both an enzyme-susceptible (CMC) and a more resistant polymer (HEC) for enzyme detection. Extremely low (<1ppb) enzyme levels can be detected if suspect materials can be referenced to an enzyme free control. The 1991 draft of the test method will be amended to include an enzyme resistant HEC, along with the special type of CMC described. A round-robin will be run using vinyl-acrylic latex paints to which cooperators will add pre-determined amounts of cellulase and t-butylhydroperoxide to include polymer degradation and detection. Four cooperators will participate.

*D01.28.03—Microbial Quality of Raw Materials*—J. Hinkle, Chairman. The proposed new method was balloted with the note added to address the previous negative. This version was approved with no negatives and only two editorial comments. The corrections were made on the proofs of the new method, designated as method D 5588. This method contains a reference to the enzyme method (D01.28.02) still under development. When that method is completed, the new method number must be included in D 5588 (Appendix XI.I).

*D01.28.04—Resistance of Paint Films to Algae Attack*—J. Hinkle, Chairman. The proposed new method D 5589, was balloted

and approved with only one comment relating to SI unit conversion. Comments were received from K. Roberts and P. Kappock that they were able to obtain and reproduce the Singapore test method (shake flash). They had obtained the species *Trentapohlia odorata* from a culture collection in Great Britain, and indicated they could provide additional information to the group prior to our January meeting.

*D01.28.05—Revision of ASTM D 3273 (Mildew Chamber)*—J. Hinkle, Chairman. The revisions of D 3273-86 were balloted and approved. This revision should appear after the next printing. This task group will be continued to address the possibility of using D 3273 for exterior coatings (which most companies already do anyway). Some items that will need consideration include light sources to duplicate exterior exposures, effects of moisture on *Aureobasidium pullulans* (A.p.), etc., to address the issue that A.p. does not thrive inside in the presence of other fungi (others take over). It was noted that it would be impossible to maintain pure A.p. cultures in a chamber due to airborne contamination. The fungal agar plate assay requires A.p. to be run separately from all other fungi D 5590.

*D01.28.06—Resistance of Paint Films to Fungal Attack*—J. Hinkle, Chairman. The proposed new method was balloted and approved with only the comment on correcting SI unit conversions. K. Roberts indicated they see occasional noncorrelation to outside exposures when a fungicide is very water soluble. This type of problem should be examined in a proposed round-robin. Several companies have agreed to participate with these pre-prepared paint systems loaded with several biocides to be sent out by the chairman (PVC, acrylic, and alkyd modified).

*D01.28.07—Revision of D 3274 (Defacement Ratings)*—B. Matta, Chairman. Proposed revisions to the method were discussed by task group members with minor alternations suggested. Because D 3274 is overdue for approval, it was decided to ballot proposed revisions prior to the development of computer imaged photographic plates.

The chairman reported that seven software companies were consulted to determine the feasibility of converting the defacement ratings currently contained within the method to computer imaged failures. However, it will require additional time to acquire necessary hardware to run imaging software. D01.28.07 will remain an open task group to further improve this method.

*D01.28—Biodeterioration*—M.C. McLaurin, Chairman. The minutes from January's meeting were approved with one correction—in the D01.28.05 notes, the fourth line should read "... other strains of

fungi as agreed . . ." (fungi not algae). It was noted that we have made fantastic progress on many of our methods with three new methods finally approved. We currently have only one standard requiring ballot action (D 3274) which should be at least partially revised by January's meeting.

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## DIVISION 30 PAINT MATERIALS

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### SUBCOMMITTEE D01.31 PIGMENT SPECIFICATIONS

#### D.E. Kesatie, Chairman

*D01.31.08—Titanium Dioxide*—L. Williams, Acting Chairman, reported that it was agreed to place D 476, "Standard Specification for Titanium Dioxide Pigments," on the D01 ballot for reapproval. It is recognized that the method needs to be rewritten in its entirety. This had to be done before 1999. Recommendations and suggestions will be solicited from titanium dioxide manufacturers and users. Ultrafine titanium dioxide may be included in the new method.

*D01.31.12—Zinc Pigments*—D. Leggett, Chairman, reported that a method for zinc phosphate pigments is still under discussion. W. Spangenberg and M. Denesha will collaborate on a method regarding the two types of zinc phosphate based on water of hydration.

The method D 520, "Standard Specification for Zinc Dust Pigment," was reviewed. Discussion focused on the lead content in the zinc dust pigment. D 520 will be on the D01 ballot for reapproval with no revisions. The task group will revise the specification by the next meeting. The type I will be reviewed to establish a maximum at 0.25%, for lead content. Type II will be reviewed to see if the existing 0.01% lead content is realistic or if it should be revised.

Under old business, nine standards on the D01 ballot for reapproval or withdrawal were reviewed. Three standards: D 480, "Method for Sampling and Testing of Flaked Aluminum Powders and Pastes," D 912, "Cuprous Oxide for Use in Antifouling Paints," and D 962, "Aluminum Powder and Paste Pigments for Paints," were reapproved and have been placed on the society ballot for reapproval.

Negative votes were received on the withdrawal of: D 212, "Chrome Green Pigments"; D 768, "Yellow Iron Oxide, Hydrated"; D 911, "Mercuric Oxide for Use in Antifouling Paints"; D 1649, "Strontium Chromate Pigment"; D 3021, "Phthalocyanine Green Pigments"; and D 3724, "Synthetic Brown Iron Oxide Pigments." The negative votes on D 212 and D 911 were found to be not persuasive and were passed to D01 for a vote. The negatives on with-

drawal of: D 480, D 769, D 1469, D 3021, and D 3724 were found to be persuasive and the standards will be balloted for reapproval. D 1649 will be on the D01 ballot as a revised specification.

Under new business, 17 standards were reviewed. Sixteen standards: D 083, "Red Lead Pigment"; D 153, "Specific Gravity of Pigments"; D 209, "Lampblack Pigment"; D 211, "Chrome Yellow and Chrome Orange Pigments"; D 561, "Carbon Black Pigment for Paints"; D 603, "Aluminum Silicate Pigments (Hydrous)"; D 604, "Diatomaceous Silica Pigment"; D 605, "Magnesium Silicate Pigment (Talc)"; D 763, "Raw and Burnt Umber Pigments"; D 964, "Cooper Powder for Use in Antifouling Paints"; D 1208, "Common Properties of Certain Pigments"; D 2218, "Molybdate Orange Pigments"; D 2448, "Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment"; D 3360, "Particle Size Distribution by Hydrometer of the Common White Extender Pigments"; D 3619, "Aluminum Silicate Pigments (Anhydrous)"; and D 4288, "Calcium Borosilicate Pigments"; will be placed on the D01 ballot for reapproval. One standard, D 475, "Pure Para Red Toner Pigments," will be placed on the D01 ballot for withdrawal.

### SUBCOMMITTEE D01.33 POLYMERS AND RESINS

#### J. Lamberton, Chairman

*D01.33.14—Alkyd*—J.G. Lamberton, Chairman, discussed the status of the standards currently requiring ballot action. Several negative votes were received on the balloted standards. Negatives concerning D 1312 and D 1615 were found persuasive. Changes regarding the negative comments were made to these standards. These will be resubmitted to D01.33 ballot. Changes to D 1639, "Standard Test Method for Acid Value of Organic Coating Materials," were approved with the addition of keywords, bias statement, and correction of the potassium hydroxide reagent preparation.

*D01.33.26—Water Reducible Polymers Including Atex*—J.G. Lamberton, Chairman, reactivated this task group. A new formaldehyde in latex method was proposed due to the urgency of better test method uniformity within the industry. An HPLC procedure with post column derivatization using Nash's reagent was proposed. Four round-robin tests had been completed by the group. Precision and bias statistics for the written method were provided for the task group's information and approval. Based on the completeness of the information provided concerning the method and the urgency for publishing the method, it was recommended that the method be published as a provisional standard.

*D01.33—J.G. Lamberton*, reported the status of the standards and noted those requiring ballot action. Two standards, D 0207, "Specification for Dry Bleached Lac," and D 0360, "Specification for Shellac Varnishes," submitted for withdrawal received negatives. A possible steward for these standards could not be contacted and as a result, the standards will be balloted for withdrawal due to obsolescence and lack of interest. The change of jurisdiction for D 1546, "Standard Method for Performance Tests of Clear Floor Sealers," to D01.42 was noted. It was requested and approved that D01.33 accept responsibility for D 2354, "Test Method for Minimum Film Formation Temperature (MFT) of Emulsion Vehicles," Standard D 1641, "Test Method for Exterior Durability of Varnishes," received two negatives and two comments on the main ballot. Since no one was present at the meeting with the expertise to address the issues raised, it was voted that D 1641 be balloted for withdrawal due to obsolescence and lack of interest. The action of D01.33.26 concerning provisional publication of the new formaldehyde in latex standard method was affirmed. The subcommittee further voted its appreciation to J. Alexander and the industry group for the completeness of the work done on the formaldehyde in latex standard method.

### SUBCOMMITTEE D01.34 NAVAL STORES

#### J. Russell, Chairman

*D01.34.01—Capillary Gas Chromatography of Rosin and Fatty Acids*—W. Trainor, Chairman, announced that there were no negative D01.34 votes, and the method will be sent to D01 ballot. In response to a comment that "adjustments in temperature and flow rate may be necessary to maintain optimum performance of the equipment during aging of the column," the subcommittee agreed to ballot the modified text in a joint D01.34/D01 ballot this fall.

The chairman also announced that the results of the round-robin carried out on the samples of rosin and fatty acid had been very encouraging and no changes in procedure were necessary. It was agreed that a second round-robin, this time using the internal standard method, would be carried out. The same rosin and fatty acid samples would be used and the chairman will send out a letter with detailed instructions on the procedure to follow.

*D01.34.04—Iodine Value of Tall Oil Fatty Acids Using Isooctane as a Solvent*—J. Bowers, Chairman, reported that no negative votes had been received in the subcommittee ballot and so the method automatically passes on to main committee ballot. However, one affirmative vote with com-

ment was received. That comment suggested that the solvent should not be restricted to isooctane. It was agreed that the chairman would write a short paragraph indicating that other solvents such as cyclohexane could be used. This will be added to the text and the September ballot will then become a joint D01.34/D01 ballot.

**D01.34.05—Softening Point of Rosin and Rosin Derivatives**—P. Zawislak, Chairman, reported on the status of this project. Various laboratories have now worked with the silicone oil DC200 as a heat transfer agent and the results are very encouraging. Although DC200 looks very promising for the manual method it remains questionable for the Herzog apparatus. Currently, the apparatus only has settings for glycerol and water. It may be necessary to add an additional setting before the Herzog can be specified in method E 28. The chairman will also amend the text of E 28 to include the use of DC200 and this change will be discussed at the next subcommittee meeting.

In regard to the automatic Mettler cup and ball instrument, J. Russell reported that Mettler had agreed to stock the stainless steel ball recommended by the subcommittee. W. Trainor reviewed some of his recent results on the influence of heating rates on softening point using the Mettler equipment. These results showed that the indicated softening point of a resin varies with the type of ball used and the type of resin being tested as well as the rate of temperature rise.

**D01.34.06—Color Measurement of Rosin and Fatty Acids**—W. Mark, Chairman, reported that the aim of this project was to develop a standard method that is more accurate and reliable than the current ASTM Method D 1544. Representatives from BYK-Gardner and HunterLab made presentations regarding possible approaches and members of the industry discussed techniques that they are currently using. The most attractive approach at this time appeared to be the use of moderately priced spectrophotometer/colorimeter. Representatives of manufacturers of such instruments will be invited to make presentations at the next scheduled subcommittee meeting to more thoroughly assess their instruments. It was also agreed to carry out an informal round-robin experiment using a stable fatty acid. B. Grady will distribute samples of fatty acid and participants will measure the color using any or all of the methods they have available. The results should indicate how the level of precision varies with the degree of sophistication of the test and so indicate the type of instrument that will meet the needs of the subcommittee.

It was reported that no response has been received from D01.26 regarding the modification of D 1544, "Color of Transparent Liquids" (Gardner Color Scale), to include a paragraph on the limited accuracy of the method. P. Zawislak will attend the June

meeting of D01.26 to learn the current status of the method.

**D01.34**—J. Russell, Chairman, reported that all current standard methods for which D01.34 is responsible are up-to-date. However, D 3008, "Resin Acids in Rosin by Gas Liquid Chromatography," will require ballot action shortly. It was agreed to change the title to state that this method is based on the use of packed columns in order to avoid confusion with the capillary column method currently being balloted. Also, it was agreed to add the TMPAH method for methylation to the text prior to balloting.

Precision and bias sections will be added to the standard methods for which research reports are available and those methods will be submitted for reapproval later in 1994. The next meeting of the subcommittee will be Atlanta, GA, on October 7 and the following meeting will be San Antonio, TX, with D01 in January 1995.

## SUBCOMMITTEE D01.35 SOLVENT, PLASTICIZERS, & CHEMICAL INTERMEDIATES

### R.G. Montemayor, Chairman

**D01.35.10—Hydrocarbon Solvents and Ketones**—S.A. Yuhas and R.L. Hinrichs, Co-Chairmen. This task group is on schedule with the review/reapproval of the 24 standards for which it is responsible. D 235, "Specification for Mineral Spirits," received a late negative from P. Hudson. Because the negative was not received in time, the standard will proceed to D01 ballot. Based on Mr. Hudson's comments, the task group decided to revise D 235 to clarify the statement on "isoparaffinic hydrocarbons" fitting the Type III "Odorless" category. A footnote will be added to the statement on Type III Odorless in the table of physical and chemical properties. The proposed footnote is: "Only products which have a very high isoparaffinic hydrocarbon content, i.e., approaching 100% are considered to fit the "odorless" category." The standard with the new footnote will be sent to D01.35 ballot. Seven standards passed society ballot, one standard passed D01 ballot, and four standards passed subcommittee ballots. A draft of the Capillary GC Method for measuring total aromatics in mineral spirits was incorporated into the existing D 3257, "Total Aromatics in Mineral Spirits by GC." After minor corrections to the proposed draft are made, this revised standard will be submitted for subcommittee ballot. A request was made to Mr. In-Sik Rhee, for help in revising the Federal Specification PD680 for Mineral Spirits.

**D01.35.20—Reactive Monomers**—J.E. Frugé, Chairman. The task group's standard are on schedule for review. Two standards, D 2192 and D 3845, passed subcommittee

ballot and will proceed to D01 ballot. D 3547 and D 3451 were reviewed and will proceed to concurrent D01 and society ballots.

**D01.35.30—Chemical Intermediates**—J. Morrison, Chairman, reported that all standards are current. Two standards, D 3546 and D 3620, passed D01 ballot and will proceed to society ballot. Eleven standards passed subcommittee ballot and will go on to D01 ballot. D 1719, "Specification for Isobutyl Alcohol," received a negative from P. Guevin regarding a technical error in a footnote. The negative was found to be persuasive and the standard will be rebalotted at the D01.35 level with the corrected footnote. D 770, "Specification for Isopropyl Alcohol," received a negative from S. Yuhas, Jr., regarding the odor testing requirement. The negative was found to be persuasive and the standard will be rebalotted at the subcommittee level with the proposed changes.

**D01.35.40—Plasticizers and Ester Solvents**—J.E. Lawniczak, Chairman. All standards are up to date and on schedule for review. Two standards passed society ballot and will be published in 1995. Five standards were approved on a D01.35 ballot and will proceed to D01 ballot. Two standards will be on a fall D01.35 ballot.

**D01.35.50—Coordination**—L. Forrest, Chairman, led a discussion on standards from D02 and D 16. From D02, D 3278 was revised and a precision and bias statement was added. Its new title is "Standard Test Method for Flash Point by the Setflash (Closed Cup)." D 86, "Distillation of Petroleum Products," was revised to make the thermometer centering device mandatory. D 93, "Flash Point by Pensky-Martens Closed Tester," was revised to include automatic instruments. It received an extensive negative from the IP which was resolved after very lively intellectual discussion. The IP withdrew its negative with editorial changes and further revision in the future. The round-robin concerning D 93 flash point by procedure A and procedure B as a function of the sample viscosity was completed. The data showed that if the sample kinematic viscosity is greater than 5 cSt at 40°C, procedure B should be used. Using procedure A on these samples will give an artificially low flash point.

From D 16, D 841, "Nitration Grade Toluene," will be sent to ballot. D 2360, "Trace Impurities in Monocyclic Aromatic Hydrocarbons," was revised to use a capillary method. D 848, "Acid Wash Color of Industrial Aromatic Hydrocarbons," was extensively reviewed with many aspects such as effect of overmixing and shear being looked at carefully.

**D01.35.60—Method Development**—R. Bartram, Chairman, noted that six methods are being developed or reviewed:

(1) Acetone in Diacetone Alcohol—The status of the robustness test conducted was discussed. Some problems still remain with the method. It was decided to poll the members to see if there is still a desire for the method before proceeding with further studies.

(2) Capillary GC Method for Acrylate Esters—A recommended GC method is going to be the subject of a robustness test. Rohm and Haas and BASF will be asked to see if they are willing to participate in the study.

(3) A revision of D 3257 to include capillary GC and the precision values obtained from the D 3257 interlaboratory program has been made. The revised method will have three procedures. The revised standard will be balloted at the D01.35 level.

(4) Capillary GC method for the purity of MAK/MIBK—A robustness test is planned for this method during the summer.

(5) Capillary GC method for D 3545, "Alcohol Content and Purity of Acetate Esters"—Eastman Chemical has provided a method of the review.

*D01.35*—Three new members were added to the D01.35 roster: K.D. Drakash, of Detroit Edison; W. Spitzley, of Total Petroleum; and F. Young, of Sherwin-Williams. R. Scott resigned his membership in the subcommittee.

In old business, the members of D01.35 were asked how they feel about the role that HAPs might play with ASTM specifications and test methods under the subcommittee's responsibility. The following statement represents a consensus of the inputs received as well as the discussions held during the meeting.

"ASTM standards are the most respected and used standards worldwide. They are concerned largely with quality and performance of materials based on their compositions and/or their physical and chemical properties, not their hazards. The EPA and HAPs are specific only to the United States. The subcommittee's unanimous consensus is that it would be unwise to include the ASTM standards reference to a material's toxicity and regulatory status. The MSDSS are where this information belongs. Formation of a task force is premature at this time."

### **SUBCOMMITTEE D01.36 CELLULOSE AND CELLULOSE DERIVATIVES**

**G.Y. Moore, Chairman**

The subcommittee welcomed two new members, J. Morton from Buckeye Cellu-

lose Corp., and D. Kiesel, from The Dow Chemical Co. D01.36 maintains 26 standards, of which 11 need action. D. Kiesel will become steward of D 1347, "Standard Test Methods for Methylcellulose," and D 2362, "Standard Test Methods for Hydroxypropyl Methylcellulose," and J. Morton will tend to D 1926, "Standard Test Methods for Carboxyl Content of Cellulose."

The following standards received subcommittee negatives: D 1787, "Test Method for Pentosans in Cellulose," and D 2438, "Test Method for Silica in Cellulose." The negatives were based on withdrawal without replacements, but no suggestions were offered. The negatives were found not persuasive, and later upheld by main committee vote. The intent was to bring the withdrawal to main committee to obtain suggestions or alternations to make the method useful, or to find out that the method is no longer being used.

The following three standards received D01 negatives: D 1348, "Test Method for Moisture in Cellulose," D 1439, "Test Method for Sodium Carboxymethylcellulose," and D 2364, "Method for Testing Hydroxyethylcellulose." All three methods received negatives from R. Montemayor on the basis that the precision and bias statements were not clear. In the methods, the term intralaboratory and interlaboratory precision statements were used, where R. Montemayor preferred repeatability and reproducibility. The decision was made to add repeatability and reproducibility in parentheses behind intralaboratory and interlaboratory (editorial change) after which R. Montemayor withdrew his negatives. In D 2364, a change was made in the drying time (from three hours to two hours), with no clear evidence that this reduction in time did not affect the precision. G. Moore will generate a research report from data within his company to support the statement. In addition, negatives were received on D 1439 and D 2364 by Mr. Edwards, who felt it was not clear if round or square stainless steel rods were used in the construction of an agitator. The negative was found not persuasive, with the intent that when the method comes up for ballot again, this clarification will be made.

The following standards will be revised and submitted for D01.36 ballot this fall. D 0301, "Test Method for Soluble Cellulose Nitrate," D 0914, "Methods for Testing Ethylcellulose," and D 1695, "Terminology of Cellulose and Cellulose Derivatives" will be revised by G. Moore. Standards D 2929, "Test Method for Sulfur Content of Cellulosic Materials by X-Ray Fluorescence," D 3516, "Practices for Ashing Cellulose," and D 3971, "Test Method for Dichloromethane Soluble Matter in Cellulose," will be provided with keywords by J. de Wit.

The following standards will undergo major revision or will be balloted for with-

drawal with replacement. D 1915, "Method for Chromatographic Analysis of Chemically Refined Cellulose," D 2641, "Test Method for Chlorine in Cellulose," by J. Morton, and D 3876, "Test Method for Methoxyl and Hydroxypropyl Substitution in Cellulose Ether Products by Gas Chromatography," by D. Kiesel.

### **SUBCOMMITTEE D01.37 INK VEHICLES**

**A.N. Scarlatti, Chairman**

*D01.37.01—Resin Solutions*—A.N. Scarlatti, Chairman, reported that the "Standard Practice for the Preparation of Oil-Based Resin Dispersions Using a Hot Oil Bath" has been approved for publication. Negative votes on the three other recommended practices for resin dispersion preparation submitted to D01 ballot were reviewed. Those negatives were discussed and voted persuasive.

J. Fetsko questioned why four separate methods for preparing resin dispersions are needed since someone wanting a method for preparing resin dispersions would have to order four methods from ASTM. She suggested that these practices be incorporated into one practice for four sections.

The committee voted to accept this proposal. A.N. Scarlatti will address all the negatives received and rewrite one "Standard Practice for the Preparation of Oil-Based Resin Dispersions" and present it at the January 1995 meeting.

*D01.37.02—Resin Dilutability*—J.W. Daugherty, Chairman, stated that this task group will be incorporated into D01.37.07 on Resin/Solvent Compatibility. A. Mercado stated that he felt there was a need to have a method to determine alkyd compatibility of resins. A.N. Scarlatti expressed the need for a standard alkyd. M. Mercado volunteered to chair a task group (D01.37.09) on alkyd compatibility of ink resins.

*D01.37.04—Standard Ink Oil*—E.W. Casserly, Chairman, reported that standard ink oils have been established and are available through Exxon and Pennzoil. A.N. Scarlatti stated that he feels standard ARLO (alkali refined linseed oil) and soybean oil is needed for resin testing. However, it was brought up that these products are subject to change on aging and a standard might not be possible.

D. Frisch said that he would contact the Soybean Oil Association to see if they would send a representative to our next meeting. Mr. Frisch will also send a letter to Cargill to get representation for a standard ARLO.

A.N. Scarlatti said that he will remove this subcommittee from next years' agenda unless there is interest in providing a standard ARLO and soya.



*D01.37.05—Quality Control of Ink Oils*—J.B. Sardisco, Chairman, addressed negatives received on the PKP method for the quality control of ink oils. Comments, negatives, and suggestions included: (1) the method should specify 10 point type for the use in cloud point determination, (2) the precision statement should be elaborated to be more specific, (3) pentane should be listed in the reagents, (4) changes need to be made in the title and scope conform with ASTM guidelines, (5) the definition of PKP value should be revised, (6) a summary of the method should be added, and (7) redundancies in the significance and use section should be eliminated. J. Sardisco will make all necessary changes in the test method and check with J. Cichon for a proper precision statement. The method will then be rebalotted.

*D01.37.06—Pigment Dispersibility of Paste Ink Vehicles*—A. Mercado and M. Fuchs, Co-chairman, reported that method screening tests using three varnishes with one pigment on the Hoover Muller did not show any differences in vehicle wettability. It was decided to redo the test with less revolutions and/or less weights.

M. Fuchs submitted a mini-flush procedure to try to determine the flushability of vehicles. But, since it was based on a Hoover Muller, M. Fuchs didn't think it would work.

A. Mercado said that he would submit a Baker Perkins test that may work with presscake. Messrs. Mercado and Fuchs will work on a test method for flushing vehicles based on the Baker Perkins test.

*D01.37.07—Resin/Solvent Compatibility*—D.P. Frisch, Chairman, reported that the proposed round-robin to prepare resin solutions was not necessary since statistical data already existed. The second round-robin to look at the proposed precipitation temperature method was undertaken with good results. A.N. Scarlatti will rewrite the scope of the proposed method and make other editorial changes, then send it to the task group for comment. He will use the aniline point method as a guide. D. Frisch will write a precision statement based on the round-robin results. The revised method will be discussed at the January meeting.

*D01.37.08—New Membership*—D.P. Frisch, Chairman, reported that D01.37 is seeking greater membership from ink vehicle manufacturers and other raw material suppliers. D. Frisch will write a letter and send it to A.N. Scarlatti to use in contacting potential D01.37 members.

*D01.37.09—Alkyd Compatibility of Ink Resins*—A. Mercado, Chairman, stated that he will fax his company's current method for determining alkyd compatibility to A.N. Scarlatti. They will put the method into the ASTM format for presentation at the January 1995 meeting.

*D01.37—Ink Vehicles*—A.N. Scarlatti, Chairman, reported that because of travel conflicts, J. Daust will resign as Secretary of D01.37. A. Mercado has been asked to consider this position unless an enthusiastic volunteer comes forward.

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## DIVISION 40 PAINT PRODUCTS APPLIED ON SITE

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### SUBCOMMITTEE D01.42 ARCHITECTURAL FINISHES

L. Schaeffer, Chairman

*D01.42.03—Porosity of Paint Films*—L. Williams, substituting for C. Tatman, Chairman, reported that test paints and supplies have been shipped to the cooperators for completion of the round-robin by mid-August. A draft of the proposed test method and test data from the chairman's lab were then reviewed. A number of procedural and statistical questions were raised. These will be reviewed and revised as necessary by the chairman and communicated to the cooperators.

*D01.42.04—Wet Adhesion of Latex Paints*—W. Vanderslice, Chairman, reviewed the problem of finding a primer to serve as the substrate in this test. New test data from the chairman's lab using panels coated with a fast-drying industrial gloss alkyd enamel were presented. This enamel produced the same wet adhesion rank order with test paints as had been obtained previously with experimental Leneta panels made using a UV clearcoat. The latter, being non-oxidizing, has the advantage of aging stability, which is important. The consensus was that the next round-robin be conducted using both substrates. L. Schaeffer will produce additional UV-coated panels using a thicker plastic substrate to prevent "cut-through" during scoring of the test paints. Using water as the scrub medium, both "early" (four hours) and "mature" (seven days) wet adhesion will be evaluated. The number of types of test paints will also be expanded to include not only semi-glosses, but also eggshell and satin flats and a house paint, based on a wider variety of latexes possessing wet adhesion. Paints of this description will be supplied by The Sherwin-Williams Co.

*D01.42.05—Adhesion of Latex Paints to Chalky Surfaces*—A. Leman, Chairman, distributed the results of a round-robin on rating the degree of chalking of exterior paints. Six cooperators tested three panels each of a weathered chalky latex paint and rated the degree of chalking according to D 4214-89, procedures 7.1 and 7.2. The data showed

very good interlaboratory reproducibility. Concerns that the cooperators' ratings were lower than those read immediately after outdoor exposure, may be related to loose chalk coming off the panels during transportation and handling. Two paint formulations, one latex and one alkyd/oil-based, which are reported to weather rapidly to a chalky surface, were reviewed. Several suggestions for modifying these formulas will be incorporated by the chairman and he will apply them to primed cedar panels. D. Marshall, of Marshall Laboratories, agreed to expose the panels on their test fences in Florida and weather them to an ASTM chalk rating of five. Once the paints have achieved this degree of chalking, a round-robin will be conducted using these panels to test the adhesion of latex paints applied over them

*D01.42.22—Guides for Testing Architectural Coatings*—H. Ashton, Chairman, reported that the technical revisions to D 3730, "Guide to Testing High-Performance Interior Architectural Coatings," agreed to at the last meeting, had been submitted to D01 ballot. Only one comment was received that suggested modifying the touch-up uniformity clause. It was decided to submit the original document, to society ballot and to consider the suggested wording on the next subcommittee ballot. A revised "washability" clause and review of the work on "accelerated tests for yellow" is in progress. At the January meeting of D01, Method D 1546, "Performance Tests of Clear Floor Sealers," was transferred from D01.33 to D01.42. The method revised by D01.33 appeared on D01.42 letter ballot 94-01. Two negatives were cast. The method has been assigned to D01.42.22. A revision to eliminate the ambiguities between "finish" and "finishing" and between "treated" and in two ways, was circulated to the attendees. This stimulated a discussion on the need for the standard and a revision of the type of sealers that have been used. A few members stated that this type of sealer was still in use by consumers and by floor refinishers for houses and smaller commercial buildings. Since there are no compositional requirements, the item is actually a performance type of standard. It was agreed to include in the scope that the sealers are for use on interior wood floors. The revised method will be submitted to subcommittee letter ballot as a "practice."

*D01.42.25—Scrub Resistance of Latex Paints*—T. Sliva, Chairman, reviewed the status of previous round-robin work. Improved reproducibility has not been shown even when round-robins are conducted using the same scrub medium, calibration panels, wet paint samples, film applicators, and brushes. A discussion followed on the differences in density and stiffness between brushes produced by the two referenced manufacturers. Since it was felt that the

pertinent dimensions and construction of the brushes should be specified, the chairman will work with the brush manufacturers to come to an agreement on the specifications and report his findings at the next meeting. Discussion followed on those sections relating to the moisture content, conditioning, and leveling of the brush. The brush manufacturers will investigate the possibility of supplying brushes pre-leveled. The need to reference calibration panels or provide illustrations of the end points for inclusion in the next revision of the method was discussed. Alternate methods of evaluating wet abrasion resistance were discussed and suggestions were solicited. The chairman distributed copies of the DIN Method 53 778, "Evaluation of Cleansibility and of Wash and Scrub Resistance of Coatings," and asked members to review the document to see if any sections should be included in D 2486. As D 2486 is due for balloting, the chairman will review it, make any needed editorial revisions, and submit it to subcommittee ballot before the January meeting. The chairman will also prepare a draft of the revision of the method for discussion at the next meeting.

**D01.42.26—Burnish Resistance of Latex Paints**—W. Vanderslice, Chairman, updated the task group's progress by reviewing the current proposed test method, using cheesecloth as the abrading medium and the Gardner Straight-Line and Washability Machine fitted with the "sandpaper attachment" holder. In response to the consensus at the last meeting, new data from his lab were introduced showing the change in 85° gloss (burnishing) for three commercial "do-it-yourself" interior wall paints dry-rubbed in 5, 10, 15, 20, 25, and 50 cycles. However, a recommendation was made that the data be subjected to mathematical analysis and an experimental design run to determine if a better maximum existed. Test data after 20 cycles were then shown for four "do-it-yourself" and three "professional painter" quality interior paints supplied by The Sherwin-Williams Co. The results under both dry and wet conditions showed promise in differentiating the burnish resistance of the test paints. Reporting instrumental readings as direct changes in gloss rather than percentage changes was preferred. It was also recommended that the dry and wet test procedures be incorporated into a single method, and that cooperators be asked to rank burnishing visually along with the instrumental assessment.

**D01.42.28—Paint Removers**—V. Scarborough, Chairman, presented the revised method for discussion. The issue of including the specific manufacturer's name of the coating to be removed was discussed at length. The chairman will contact them to determine if there are any objections to this. Four cooperators volunteered to participate in the round-robin.

**D01.42.29—Guide to Testing of Exterior Wood Stains**—A. Leathers, Chairman, reviewed the proposed exterior stain definitions that were previously mailed to the task group members. Following a brief discussion, definitions for "opaque" and "semi-transparent" stains were unanimously adopted. The task group decided that the guide will be confined to methods that are unique to stains. The existing ASTM guides were reviewed by V. Scarborough, and applicable tests for stains were indicated. An immediate need for a review of D 5401, D 3278, and D 5301 was noted. The need to evaluate both the stain's and wood's resistance to UV light was recognized. Information from the EMPA Institute of Switzerland will be sent to the chairman as well as the name of an expert from Clemson University to assist with defining UV tests. Testing ideas will be gathered by the chairman for distribution at the next meeting. The chairman will write a proposed scope and outline for the "guide" to be sent to task members prior to the next meeting.

**D01.42.30—Wet-Edge Time of Latex Paints**—E. Countryman, Chairman, reviewed test methods for this property from a number of companies and other standards organizations. Four test panels prepared by the chairman were then reviewed. Three involved a drawdown-brushout on sealed Leneta charts. Since "open time" had a different meaning to one member, it was decided to refer to this method as "wet-edge" or "lap-in" in the future. A discussion ensued involving other application methods such as rolling and spraying. It was decided for the present to limit the scope of this method to brushing. The Sherwin-Williams test method for lap-in time will be sent to the chairman, who will mail it to the cooperators. Task group members are to study the various methods and mail their recommendations to the chairman who will write up a draft of a proposed method for discussion at the next meeting.

**D01.42.31—Water-Beading on Exterior Coatings**—V. Scarborough, Chairman, presented an outline for discussion. It was decided that the initial scope of the new task group's work would be to first develop a method for measuring contact angle utilizing a goniometer. Since this entails measurement of a physical property, the task group will be transferred to D01.23 to begin this segment. A first draft of the method will be presented at the next meeting. After a reliable method is developed, its use and significance can then be expanded.

**D01.42—Architectural Finishes**—L. Schaeffer, Chairman, reported that revised standards D 3450, D 3730, and D 4828 had passed D01 ballot with no negatives and will proceed to society ballot. Revisions are in progress for overdue standards D 1546, D 1736, and D 1849, which will then be sub-

mitted for subcommittee ballot. Comments and a provisionally withdrawn negative on D 5326 will be discussed at the January meeting in San Antonio. D 2354 on "Minimum Film-Forming Temperature of Emulsion Vehicles" has been transferred from D01.42 to D01.33. T. Sliva initiated discussion on transferring proposed specification D 4717, "Specification for an Interior Latex Flat Wall Paint," from D01.41 to D01.42's jurisdiction. Subcommittee D01.42 rejected this proposal by a vote of 3-5-7. Additional discussion followed on establishing a new task group to investigate the desirability of D01.42 sponsoring a whole paint specification. By a vote 10-1-4, this motion by T. Sliva was carried. The new group will be designated D01.42.32. A chairman is being sought. Two other new task groups have been formed. These are D01.42.30 on "Wet-Edge Time of Latex Paints" (E. Countryman, Chairman) and D01.42.30 "Water-Beading on Exterior Coatings" (V. Scarborough, Chairman). The latter group will temporarily be transferred to D01.23 where a method for instrumental measurements of the contact angle of water on various painted surfaces will be developed. L. Schaeffer announced that he was resigning as D01.42 Chairman and that his successor would be W. Vanderslice, who is presently Secretary of the subcommittee.

## SUBCOMMITTEE D01.46 INDUSTRIAL PROTECTIVE COATINGS

### G.W. Gardner, Chairman

**D01.46.02—Surface Preparation**—K.A. Trimmer, Chairman, reported that SSPC has agreed to allow ASTM to incorporate the new Vis 3, "Power Tool Cleaning" visual standards in D 2200, "Pictorial Surface Preparation Standards for Painting Steel Surfaces." Trimmer will write a Method C for D 2200 to cover power tool cleaning and send it to D01.46 ballot.

G.W. Gardner reported that D 2092, "Practices for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting," has been revised and will be sent out to D01.46 ballot.

K.A. Trimmer is making changes in D 4417, "Test Methods for Measurement of Surface Profile of Blast Cleaned Steel," and it also will be sent out to D01.46 ballot. G.W. Gardner has received information from C. Higgins of ATI Systems, Inc., on Model #2100 Surf Pro surface profiler, with an inquiry as to if D01.46 would find it useful for surface profile measurements. This instrument is used to evaluate surface profiles much, much smaller than needed for evaluating blast profile for industrial protective coatings. Mr. Trimmer will review the information. C. Higgins is willing to bring the instrument to the January 1995 meeting.

The information has also been presented to D01.23 and other subcommittees.

K.A. Trimber has reviewed D 4940, "Test Method for Conductometric Analysis of Water Soluble Ionic Contamination of Blasting Abrasives," and recommends that it be sent to D01 ballot. The task group agreed. He informed the task group that newer, portable conductivity meters seem to give comparable results to those used to develop D 4940. These portable meters would offer considerable advantage for field use. The task group will perform a round-robin on the portable meters. D. Chance, K.A. Trimber, D. Griffin, R. Weaver, and C. Ray all expressed interest in collaborating in this round-robin. Mr. Trimber will work with W.C. Johnson to determine how many meters and labs are needed and coordinate the round-robin testing.

*D01.46.03—Repainting*—G.W. Gardner, Chairman, reported that a D01 ballot to withdraw D 4752, "Test Method for Measuring MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub," received five official voting negatives and four additional non-official negatives. The task group unanimously agreed with these negatives that D 4752 should not be withdrawn. M.E. McKnight analyzed the testing done by D. Chance on three different inorganic zincs at three different times and recommends that precision determination for D 4752 is not possible due to the arbitrary rating scale. The task group unanimously agreed and Gardner will prepare a revised D 4752 with a new precision statement and other previously agreed upon changes to send out to D01.46 ballot. A precision determination for D 5402, "Standard Practice for Assessing the Solvent Resistance of Organic Coating Using Solvent Rubs," is not possible since numerical ratings are not used.

Review of D 610, "Test Method for Evaluating Degree of Rusting and Painted Steel Surfaces," has revealed a considerable discrepancy between photographic standards and pictorial representations. Additional computer-generated pictorial representations of various percentage rusting indicate additional discrepancies. The task group agreed that present standards were not adequate for all field situations and that new standards would improve the capabilities of D 610. To this end, M.E. McKnight will produce computer-generated pictorial representatives for three different dispersions of rusting for each percentage level: (1) a single (or very few spots); (2) a uniform dispersion of spots; and (3) a concentration of spots in part of the total area.

R. Weaver will use the computer-produced representations to locate panels demonstrating each particular level of rusting with the intention of using these panels for producing photographic standards. SSPC, through Mellon Institute, can produce these photographic standards.

*D01.46.07—Inspection*—G.W. Gardner reported that D 3276, "Standard Guide for Painting Inspectors (Metal Substrates)," will be removed from the ASTM standards in 1995 due to the age of the last revision and the task group's inability to make suggested improvements to satisfy all of the D01 members. D 3276 is technically sound and many improvements have been incorporated into the latest revised draft. However, additional improvements can be made. Since time will not allow these improvements, G.W. Gardner will write a letter for concurrent D01/society ballot urging reapproval of the D 3276-86 edition while the task group expedites work on enhancing the current draft.

Draft #4 of the new "Standard Guide for Painting Inspectors (Concrete and Masonry Substrates)," received several comments on the most recent ballot. Negatives by A. Beitelman, D.K. Griffin, and H.R. Stoner were persuasive and this guide is withdrawn from ballot. Changes resulting from these negatives, as well as comments from J.F. Montel, J. Raska, and K.M. Wolma will be incorporated into a Draft #5 and then sent out for D01.46 ballot.

*D01.46.12—Sampling*—K.A. Trimber, Chairman, reported that the new "Practice for Field Sampling of Coating Films for Analysis of Heavy Metals" has been revised and will be sent out to D01.46 ballot.

Mr. Trimber indicated that ASTM E06.23 has been working on a soil sampling standard. M.E. McKnight reported that this standard was in the process of being published and she would see that K.A. Trimber received a copy so D01.46.12 could review.

Mr. Trimber inquired as to the interest in this task group regarding writing a standard for sampling of blast debris. Interest was high, but experience and D01.46 responsibility in this area was questionable. He will check to see if there is an ASTM committee on waste that would be more appropriate and capable of handling this task.

*D01.46.13—Guide for Testing*—M.E. McKnight, Chairman, conducted the first meeting of this new task group. She had prepared a starting document that outlined testing for industrial protective coatings. Discussions centered around the scope and need for this guide. After considerable discussion, the task group decided that it may be necessary to provide several standards. The first should be a listing of applicable tests. M.E. McKnight and J.T. Cheng will put together such a list for our next meeting.

*D01.46.14—Chemical Immersion*—G.W. Gardner reviewed the background behind this new task group. All seemed surprised that there was not applicable ASTM method for chemical immersion testing. This is not intended to be an accelerated test, but instead a test to simply evaluate the chemical immersion resistance characteristics of a coating. The task group unanimously agreed

that it would be very beneficial to have a standard method for performing chemical immersion resistance testing. C. Ray volunteered to be task group chairman. Mr. Ray will research the various methods from NACE, ANSI, ASTM D 33, and manufacturer's procedures to get a starting point standard for the January 1995 meeting.

*D01.46*—G.W. Gardner, Chairman, welcomed four new members to the D01.46 roster: Judy T. Cheng, of Ameron PCS; Kenneth W. Espert, of Center for Applied Engineering; Thomas Manning, of International P.C.; and Kash D. Prakash, of Detroit Edison Co. D01.46 has 80 members (58 official voting members and 22 non-official voting).

Updates from each task group were given and can be found in the preceding minutes. In addition, M.E. McKnight, Chairman of task group D01.46.04, "Pull-Off Adhesion," reported that she had been unable to get ASTM to correct D 4541, "Standard Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers." She will send the revised D 4541 for D01 ballot.

Three standards are due for review for ballot. Ms. McKnight will review D 5043, "Methods for Field Identification of Coatings," and D 5065, "Guide for Assessing the Condition of Aged Coatings on Steel Surfaces," to determine if they are suitable for ballot for reapproval. G.W. Gardner will review D 5064, "Practice for Conducting Patch Test to Access Coating Compatibility," to determine if it is ready for ballot for reapproval.

## **SUBCOMMITTEE D01.47 MASONRY TREATMENTS**

### **E. McGettigan, Chairman**

*D01.47.01—Water Repellency of Treated Masonry*—V. Scarborough, Chairman, lead the group in a discussion of the results of varying the dip treatment times on 2 in. x 2 in. x 2 in. mortar cubes at 10 sec, 30 sec, 1 min, and 3 min. The increased dip times did not show differences in performance of the waterborne or solvent-borne coatings which were included in the test. It was suggested that the two heating cycles of 110°C may be causing microcracks to occur in the cubes and this might be affecting the results. Other methods might include looking at concrete disks or 3 in. x 6 in. x 1/4 in. concrete slabs rather than using cubes. These disks or slabs would probably dry more readily at a lower temperature. The use of a dye in either the sample treatment or the immersion water was suggested to detect microcracks. A Cobb ring was also suggested to eliminate immersion of the samples. All of these suggestions will be considered to develop a modified method. The results will be reported at the next meeting.

*D01.47.05—Water Vapor Transmission of Treated Masonry Substrate*—E. McGettigan, Temporary Chairman, lead a discussion on the direction of the task group. The proposed method has not given acceptable intra- or interlaboratory correlation, despite several revisions and round-robin test studies. It was suggested that for film forming water repellents, method D 1653 should be used. The difficulty is in determining the performance of penetrating sealers. A modification of D 1653 could be used by substituting a suitable medium (e.g., glassfiber fabric or silica impregnated paper). An investigation into the different types of media and their applicability for this method will be completed and reported at the next meeting.

*D01.47.06—Freeze/Thaw Resistance of Treated Masonry*—L. Stark-Kasley, Chairman, distributed copies of the proposed test method, "Evaluation of the Freeze/Thaw Resistance of Mortar Specimens Treated with Water Repellent Treatments." The proposed method was recently submitted for D01.47 ballot, and four negatives were received. Three of the negatives were persuasive and were satisfied based on changes to the method. The title was changed to "Evaluation of the Effect of Water Repellents on Freeze/Thaw Resistance of Hydraulic Cement Mortar Specimens." The title now focuses on the water repellent treatment and freeze/thaw cycling instead of the specimen. The substrate name was changed to reflect the actual substrate used. The scope was also broadened to focus on the water repellent treatment and relation to performance in the field. Lastly, the definitions of complete failure, fine cracks, and heavy cracks were added. This method will be rebalotted at the subcommittee level following this meeting.

*D01.47.07—Alkali Resistance of Masonry Treatments*—B. Berglund, Chairman, distributed the results of the immersion study of treated and untreated substrates in 1N and 10N KOH solutions. Performance differences were observed between two silanes using the 10N solution. Test results which incorporate the following changes in the current draft of the method will be used to understand the alkali resistance of the treatment: (1) a five percent KOH solution by weight, (2) a corresponding KCl solution by weight, and (3) an increase in testing temperature to accelerate the study. In addition, another test method utilizing a chromate paint to evaluate water repellent treatments will be performed. This test should demonstrate differences in coating performance resulting from the alkalinity in the substrate alone. The results of the testing will be reported at the next meeting.

*D01.47.10—Evaluation of Field Applied Treatments*—E. McGettigan, Chairman, distributed the results of the first round-robin

test study. The intralaboratory correlation was consistent for the various specimens tested. The interlaboratory results were within a reasonable range for an initial round-robin. Several suggestions on clarifying the method were offered, and other techniques for performing the test procedure were discussed. Another round-robin is scheduled before the next meeting.

*D01.47.11—Research and Planning*, E. McGettigan, Chairman, lead a discussion on the present technique used to bring test specimens to a constant weight, which involves heating at 110°C. It was brought to the committee's attention that this preparation may have adverse effects on cementitious materials. Doug Hooten, of the University of Toronto, has performed research which demonstrates an increase of gas permeability through concrete specimens heated over 100°C. The reasoning is that the high temperatures cause microcracking of the cement paste. A research project will be developed to determine if this phenomenon is a factor in several of the subcommittee's test methods.

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## DIVISION 50 PAINTS FOR FACTORY APPLICATION

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### SUBCOMMITTEE D01.51 POWDER COATINGS

**N.D. Emily, Chairman**

*D01.51.02—Hiding Power of Powder Coatings*—M.P. Sharma, Chairman, reviewed the results of a round-robin test where four different powders were evaluated. Reproducibility and repeatability were good. Red pigmentation creates a challenge because of the relatively good hiding power of the pigmentation. A draft method will be available for the next semi-annual meeting.

*D01.51.03—Spray Characteristics of Powder*—N.D. Emily acting for C.R. Merritt, Chairman, discussed Draft #4 of a first pass transfer efficiency test procedure for laboratory evaluations. A round-robin will be conducted to provide information on the practicality of this draft method. Five collaborators volunteered to investigate this technique.

*D01.51.05—Specific Gravity of Powder*—D. Schneider, Chairman, reported on the results of another collaborator who ran the pycnometer method where their technique sample measurement was refined. Variation in the data reported indicated retesting with a new set of powder samples was warranted. Retesting will be done as

soon as possible. Metallic powders are still causing measurement problems. A first draft of a new method will be generated before the January 1995 sessions. Metallics will not be included in this draft.

*D01.51.06—Particle Size Distribution*—J.D. Pont, Chairman, presented an initial draft of a "Guide to Particle Size Measurements." This document will be reviewed and comments forwarded to the chairman prior to the next meeting.

*D01.51—New Projects*—N.D. Emily, Chairman, reviewed the status of measuring uncured powder film thickness. A new device will be investigated which may have general industry potential. Other options for new projects included application fluidity of powders and cured film thickness measurement of textured powder and powder coatings over textured substrates.

The Chairman reported on the status of the revision of D 5382, "Guide to Optical Properties of Powder Coatings." This revision is to include the latest published references which were not available in 1993. The new projects task group will meet annually during the winter session.

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## SUBCOMMITTEE D01.52 FACTORY-COATED WOOD BUILDING PRODUCTS

**S.B. Schroeder, Chairman**

*D01.52B—Hardboard*—S.B. Schroeder, Chairman, reported progress on the research report on statistics for the new Cobb Ring test method for Determination of Liquid Water Permeability of Applied Coatings on Hardboard and other Composite Wood Products. This method will be submitted to ballot in 1994 and a new round-robin conducted in 1995.

*D01.52.13—Prefinished Hardboard*—T. Rieth, Chairman, reported that Revision 7 of the proposed "Standard Practice for Performing Accelerated Outdoor Weathering of Factory Coated Embossed Hardboard Using Concentrated Natural Sunlight and Soak-Freeze-Thaw Procedure" had been distributed to D01.52 members for comment prior to the Raleigh meeting. Several technical items and a number of editorial items (identified by ASTM staff) were discussed and resolved. Several technical issues were voted not persuasive. Revision 8 will be generated for concurrent D01.52/D01 ballot.

For various reasons, additional data relating Accelerated vs. Natural Exposures for Alternate Coating(s), Substrate(s), and Methods," were unavailable for this meeting. Expected at the January meeting is the following:

(1) An Akzo study relating correlation of this method with fence exposures for a range of substrate/coating combinations.

(2) An update of Xenon arc/soak/freeze/thaw exposures for a primed specimen set with pass-fail control material.

(3) An evaluation of fence exposures from an extensive AHA primed test series, from which it is hoped additional pass-fail controls will emerge for evaluation.

**D01.52.15—Film Thickness**—R. Matejka, Chairman, reported that discussion centered around a round-robin using smooth surface coated board versus embossed coated board. It was decided to use the embossed coated board. A new technique for measuring film thickness using video microscopy will be incorporated in the revision of D 5325-92. It was also decided to investigate the possible use of a new film thickness system from SJH Designs of Oakville, Ontario.

Stan Schroeder has agreed to be temporary committee chairman of D01.52.15, in order to expedite the proposed film thickness round-robin.

**D01.52—D01.55 on "Factory Applied Coatings for Preformed Products,"** requested that D01.52 adopt four of their methods which were related to wood substrates, since D01.55 did not have adequate membership to review the methods. These four methods are: D 1211, "Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood"; D 3459, "Humid-Dry Cycling for Coatings on Wood and Wood Products"; D 3023, "Determination of Resistance of Factory Applied Coatings on Wood Products to Stains and Reagents"; and D 2571, "Testing Wood Furniture Lacquers."

There was a unanimous vote to accept these methods. However, the acceptance of the four methods, D01.55, required that D01.52 reconsider its committee title and scope.

The new title of D01.52 will be "Factory Coated Wood Products." The new scope of D01.52 will be "to develop coordinated methods of test for factory applied coatings on wood and wood composite products. This scope does not include the wood alone, the paint alone, or methods of application, insofar as these fall under the jurisdiction of other ASTM groups." Hopefully, the expanded scope will increase committee membership.

In new business the American Hardboard Association has requested that ASTM address such technical problems as: (1) surface smoothness; (2) surface toughness, and (3) extension of the soak/freeze/thaw, conc. sunlight test for the weathering acceleration of primed substrates. Several members agreed to review existing ASTM and other methods on these topics for discussion at the winter meeting.

## SUBCOMMITTEE D01.53 COIL COATED METALS

**R.J. Tucker, Chairman**

**D01.53.01—Pretreatment of Substrates**—H.E.G. Rommal and D.C. Steel, Chairman, discussed the rewrite (necessitated due to a persuasive negative) of the balloted document. Revisions will be made and the practice will be reballoted.

**D01.53.03—Task Group on Accelerated Weathering**—D.A. Cocuzzi, Chairman, reviewed 23 panels, each panel representing 50 square feet of prepainted metal, retained at Akzo Nobel, Columbus, OH. Additional colors in coil polyester technology will be sought and included in the study. As soon as this is accomplished, triplicate panels will be sent to for real-time, 45°S, open-backed South Florida exposure. An additional set of panels will be sent for real-time Phoenix exposure. Standard gloss (60° specular reflectance), color (45/0 geometry, Hunter color space), and tape chalk readings will be taken to monitor to degradation. At future meetings, we will begin to discuss the accelerated weathering techniques which will be used to compare against the real-time results.

**D01.53.04—Task Group on Dry Film Thickness Measurement**—G.R. Pilcher, Chairman, discussed the draft method on the drill (boring) dry film thickness measurement device. The entire document was reviewed. Revisions will be made and circulated once again to D01.53 members to solicit comments prior to subcommittee balloting.

**D01.53—Coil Coated Metals Subcommittee Meeting**—R.J. Tucker discussed the one negative on D 3794, "Standard Guide for Testing Coil Coatings." After discussing the matter with Scott Orthey, it was decided to attempt to convince the author of the negative to withdraw it, which was done and the standard will go to society ballot. D 3281, "Test Method for Formability of Attached Organic Coatings with Impact-Wedge Bend Apparatus," D 4145, "Test Method for Coating Flexibility of Prepainted Sheet," and D 4146, "Test Method for Formability of Zinc-Rich Primer/Chromate Complex Coatings on Steel," are all up for review and will be discussed at the next meeting.

## SUBCOMMITTEE D01.55 FACTORY APPLIED COATINGS ON PREFORMED PRODUCTS

**E. Praschan**

**D01.55.06—Coatings on Plastics**—M. Lutterbach, Chairman, reported that the

wrong version of the revision of D 3002 was balloted. Two negative votes and four affirmative votes with comments were received. D 3002 will be revised per the two negative votes and submitted for D01 ballot. The task group determined that the four editorial comments were not persuasive.

**D01.55.11—Mar Resistance of Automotive Clear Coatings**—A. Rutkiewicz, Chairman. The task group recommended that the test method incorporating both dry abrasion and wet abrasion be submitted for D01.55 ballot so that comments would be available for the January 1995 task group meeting. The results of a round-robin test of the dry abrasion and wet abrasion methods identified several issues that need to be resolved.

**D01.55.12—Non-Conductive Coatings for Electrical Protective Equipment**—L. Thieben, Chairman, indicated that this new task group was assigned the number D01.55.12. The chairman will contact the chairmen of Committees D09 and F18 to clarify responsibility for this activity. The chairman will also submit an advertising release, inviting interested parties to attend the January task group meeting.

**D01.55—E. Praschan** announced that this was his last meeting as D01.55 chairman. The new chairman is Richard Diem. In D01.55 action, D 2199 was withdrawn from ballot with three negative votes. Mr. Diem will contact the three voters and see if they have any suggestions for change. D 3002 will be revised per two negative votes and reballoted. The task group determined that the four editorial comments were not persuasive. Standards D 0365, D 3732, D 2198, D 2337, D 2338, and D 3133 will be reviewed and revised as needed prior to balloting. J. Devido reported that responsibility for methods D 1211, D 2751, D 3023, and D 3459 has been transferred to D01.52. In new business, it was agreed that a continuing activity for D01.55.06 would be Exposure Testing of Coated Plastic Parts.

## SUBCOMMITTEE D01.56 PRINTING INKS

**J.M. Fetsko, Chairman**

**D01.56.02—Lightfastness of Prints**—J. Robins, Chairman, reported that the available data are insufficient to develop a precision statement for D 3424, "Test Method for Lightfastness and Weatherability of Printed Matter." It was suggested that the test results be evaluated by a ranking method according to page nine of the *Blue Book*.

**D01.56.12—Viscosity of Liquid Inks**—J. Daugherty, Chairman, reported that he contacted C. Schoff, chairman of D01.24 to include the precision statement from a liquid ink round-robin in D 4212, "Test Method for Viscosity by Dip-Type Viscosity Cups."



*D01.56.14—Setting of Heatset Inks*—D. Ness, Chairman, distributed copies of a proposed test method using the Sinvatrol for evaluating relative setting of heat set inks. Minor suggestions were made regarding the use of printing gages. The precision statement should state that the test samples must be compared to a control.

*D01.56.15—Density (wt/gal) of Paste Inks*—P. Ford, Chairman, distributed copies of a letter requesting that the precision statement from a round-robin on ink densities by weight-per-gallon cup be added to D 1475, "Test Method for Density of Paints, Varnishes, Lacquers, and Related Products."

*D01.56.17—Guide for Printing Inks*—J. Fetsko, Chairman, reported that D 5010, "Guide for Testing Printing Inks and Related Materials," will be balloted to include D 1308, "Test Method for Effect of Household Chemicals on Clear and Pigmented Organic Finishes."

*D01.56.19—Printing Strength*—J. Daugherty, Chairman, distributed an internal method for determining the relative strength of printed ink films. The method will be written according to ASTM protocol and submitted to P. Ford for a pilot study.

*D01.56.20—Technical Coordination*—P. Ford, Chairman, explained that the difficulty of VOC tests for water inks was caused by variability both in the VOC determination and in the water analysis. C. Tebeau offered to serve as liaison with D01.24 Volatile Content of Coatings.

*D01.56.21—Print Abrasion*—G. Vandermeersche, Chairman, distributed revised copies of a proposed standard covering the use of the GV-CAT for testing cans and other metal decorated products. It was suggested that the test method be checked out by a person who does abrasion testing on a regular basis. J. Cichon volunteered his company's services and will also process available round-robin data for a precision statement.

*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, a new round-robin must be run in duplicate. It was also suggested that the manual rub off method be included even though the precision was poor.

*D01.56.23—Degree of Dispersion by Microscopy*—A. Mercado, Chairman, reported that he conducted a pilot study using a Patterson globe reticle and a method for standardizing the preparation of specimen slides. He will distribute photomicrographs to subcommittee members for evaluation and the text of a proposed test method prior to the next meeting.

*D01.56.25—Degree of Radiation Curing*—E. Kobylarz, Chairman, reported that he conducted a test using the Control Cure Analyzer, the Crock Tester, and a Sutherland Rub Tester with a 0.25 pound weight. The wiping solution was MEK for coatings and 30% MEK in IPA for the inks. He will conduct a study of a post cure and further tests to determine repeatability of the three methods.

*D01.56.26—Chemical Resistance*—J. Cichon, Chairman, reported that he met with D01.55, who were receptive to his suggestion that D 1308 (see D01.56.17) be revised to accommodate printing inks. He also distributed copies of internal test methods for alkali resistance and bleed resistance and will put them into ASTM format. Copies of five relevant ISO test methods will be obtained.

*D01.56.27—VOC40 of Non-Heatset Paste Inks*—P. Ford, Chairman, reported that recent round-robin data will be the basis for a revised precision statement for D 5328, "Test Method for VOC40 of Non-Heatset Paste Inks at 40°C." It will include a table with various levels of VOC and the precision obtained at each level.

*D01.56.28—Tinting Strength of Liquid Inks*—D. Ness, reported that a Sherwin-Williams white base showed better repeatability than Porter due to its higher pigment contact and fluid viscosity. It was also found that a larger cuvette did not improve the accuracy of instrumental readings and that Meyer rod draw downs were subject to flotation and changes with time.

*D01.56.29—Membership*—J. Daugherty, Chairman, distributed a list of names, addresses, phone and fax numbers of current members who regularly attend meetings.

*D01.56.30—Instrumental Color Measurement*—J. Cichon, Chairman, distributed copies of D 3134, "Practice for Establishing Color and Gloss Tolerances." He will also obtain ISO 2846, which details a print method for color measurement and transparency on a reference substrate. The subcommittee agreed to defer work on spectrophotometric measurements until the print preparation method (D01.56.32) is completed.

*D01.56.31—Opacity of Printing Inks*—A. Semijaic, Chairman, distributed copies of an NPRI study conducted by Bassemir and Zawacki, which found that the Leneta varnished opacity charts showed opacity differences in printed ink films. The method will be written as per ASTM format and a pilot study conducted with inks at varied opacity levels.

*D01.56.32—Laboratory Printing*—J. Cichon, Chairman, distributed copies of the new NPRI method for the Little Joe proofing press. He will also obtain copies of ISO

Test Method 5737 (1983) entitled "Preparation of Standard Prints for Optical Tests."

*D01.56*—J. Fetsko, reported that three test methods are up to five-year review. The methods (and corresponding shepherds) are: D 4040, Falling Rod Viscometer (J. Fetsko), D 4361, Apparent Tack (G. Carr), and D 4942, Water Pickup (P. Ford). Members were requested to review the methods and send suggested revisions to Mr. Fetsko by December 1994.

## **SUBCOMMITTEE D01.57 ARTISTS' PAINTS AND RE- LATED MATERIALS**

**B. Gavett, Chairman**

*D01.57.02—Lightfastness of Pigments*—T. Vonderbrink, Chairman, reported that the test methods in D 4303 need major revision because the equipment had been changed in the fluorescent box, the exposure levels in the sun test needed to be equated with the other two methods, and the manufactures of the xenon arc equipment needed to reach agreement on the exposure level to equate to the sun level. He said he hoped to have more information for the January 1995 meeting. New members and guests present questioned the reasons for using these three test methods.

*D01.57.04—Specification for Artists' Paints*—B. Gavett reported that the Table I changes had been balloted with no negatives but with one comment that manufacturers use the CI number as well as the color name, since pigments and names vary considerably. After a discussion of the typesize of common names in relation to proprietary names, no action was taken.

The proposal suggested by R. Gamblin to revise Section 5 regarding placement of common and propriety names was discussed in detail. It was agreed to form a study group of interested persons to develop standardized common names for pigments and to have this group meet Saturday night and Sunday morning before the January 1995 meeting in San Antonio.

In answer to a question at the previous meeting, it was reported that Pigment Yellow 1 had been dropped from D 4302 when approving the resin oil standard because of numerous fading discrepancies and that it would have to be retested before being returned to the standard. No action was proposed.

*D01.57.07—Physical Properties*—It was reported that NGA, CCI, and the Smithsonian will develop a four-year plan to study current art materials regarding conservation issues and manufactures were asked for their input as to issues, materials, and their priorities.

It was suggested that there be a separate standard for gessoes that would address issues such as composition, interlamination, application, and adherence of oil paints. It was agreed to start an activity to outline some of the issues and/or problems under the direction of this task group. It was also agreed that persons interested in this new activity should contact R. Gamblin, Chairman, so the process could be started by mail. Mr. Gamblin suggested that the oil paint standard should address the issue of stabilizers. J.T. Luke suggested a test method on the solubility and removal of varnish be considered and said that the noted expert, Robert Feller, had done a considerable amount of research that could be used as a base for such a method. Ms. Luke agreed to serve as a liaison between Mr. Feller and the Physical Properties group.

*D01.57.08 and D01.57.12—Toxicity Labeling and Determination of Toxicity—W. Stopford, Chairman, and C. Jacobson reported that CPSC had found excessive levels of lead in Chinese crayons and were now looking at other children's crayons, chalk, finger paints, and modeling clay. C. Jacobson also reported that CPSC was considering a revision of the scope of the art material definition in LHAMA. W. Stopford said that CPSC's lead testing methodology was not published and had not been validated and that ACMI had tested all its members' crayons using D 5517, which is currently in the D01.57 ballot process. He said that ACMI and CPSC were doing interlab testing comparing ACMI testing with D 5517 and CPSC's method and that ACMI will encourage CPSC to use D 5517.*

Mr. Stopford also reported that CPSC was looking at formulas of children's crayons, chalk, finger paints, and modeling clay because of a concern about dichloro-benzidine dyes, which are metabolized and are carcinogenic, but that only dichloro-benzidine pigments are used in such art materials evaluated by him. He also said that such pigments may leave benzidine-congener dyes, so all such art materials in the ACMI program have been tested favorably using D xxx-94, proposed Standard Test Method for "Determining Extractability of Aromatic Amines from Art Materials."

W. Stopford stated that Section 5 in Draft #6 of D 5517, which was distributed at the meeting, had been revised to show this method's correlation to animal testing in 5.1, that it is applicable in low-soluble metals in 5.4, and that there is not enough data for it to be applicable for inhalation exposure in 5.5

Mr. Stopford distributed Draft #2 of a standard on amines, to which he had added a section on significance and use. He urged that it be submitted to D01.57 ballot so that it could be published for CPSC to use in testing for benzidine-congener dyes and pig-

ments and so that it can be validated by interlab testing.

*D01.57.10—Consumer Evaluation—J.T. Luke, Chair, reported that D 5383 and D 5398 had been accepted but may not yet be in print. She said that she was compiling the research to go to ASTM as support material. She also said that the research report for D 4303 had been submitted to ASTM and would be revised and resubmitted, if necessary, after D 4303 is revised. After discussion, Paula Volent, of the National Gallery of Art, agreed to assist Ms. Luke in writing a grant to get such research reports published. Ms. Luke also reported that the tinting strength standard, D 4838, completed the balloting process for the five-year review.*

*D01.57.11—Gouache Paint—T. Takigawa, Chairman, reported that he had corrected six lightfastness categories in the standard, had changed the manual application of the test panels in 7.5.1, and felt 5.4 on contents was not needed. After discussion, it was agreed to indicate the vacuum and other methods were preferable to taping down the paper in 7.5.1, to add the word "relatively" before the word "opaque" in the definition in 3.2.1 to keep the distinction between gouaches and watercolors but to allow artist gouaches that are not completely opaque, and to change the word "intended" to "formulated" in that same definition. It was noted that this latter change should also be made in the watercolor standard. It was agreed that the gouache standard was ready for ballot.*

*D01.57.14—Colored Pencils—J.W. Beauchene was named Chairman. Mr. Beauchene and L. Armstrong reported on the tests conducted on five products' samples drawn by two groups, artists and non-artists. Mr. Beauchene said they would retest the non-artist samples again to double check variations of results of two samples. They reported that the results showed good repeatability of instrumental readings done on samples of the same colors prepared by several different people. Most artists present felt that pigments should be identified as with paints. J.W. Beauchene said identifying pigments might be a problem since manufacturers do not wish to reveal formulations and sometimes use seven-eight pigments in some pencils. He said he would survey other manufacturers on this issue. Members were asked to forward questions or comments to Mr. Beauchene.*

*D01.57—J.T. Luke requested that the minutes of the January 1994 meeting be changed to clarify her negative regarding common names appearing in type size one-half that of the proprietary name. While she urged that the current requirement of no less than one type size smaller than the proprietary name be retained, she cast her nega-*

tive because it was unclear in the proposed revision whether one-half the measured size or one-half the type size would be allowed. She said he believed that one-half the type size would be illegible in some type fonts.

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## DIVISION 60 PAINT APPLICATION

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### SUBCOMMITTEE D01.61 PAINT APPLICATION TOOLS

#### F.B. Burns, Chairman

*D01.61.01—Paint Brushes—T. Sliva, Chairman, reviewed the subcommittee balloting of the proposed test for "Comparing the Leveling Efficiency of Paint Brushes" and the "Evaluation of the Cleanability of Paint Brushes." Both methods received one negative vote which was found persuasive by the task group.*

The task group carefully reviewed each method. It was the decision of this task group to remove the reference to the ASTM standardized scoring scheme in both methods and to report results only by rank order. Both standards will be sent to D01.61 ballot. "Comparison of the Leveling Efficiency of Paint Brushes" will include a reference for the proper use of angular sash brushes when running the test. The standard "Evaluation of the Cleanability of Paint Brushes," will incorporate an illustration to expand upon the brushout procedure.

*D01.61.02—Paint Rollers—F. Burns, Interim Chairman, reported that J. Price has resigned as chairman of this task group. F. Burns asked for a volunteer to take over this group; on a permanent basis. At this point, the chairmanship remains open.*

*D01.61.03—Woven Paint Applicator Fabrics—M. Murray, Chairman, distributed Draft #1 to the task group for review. Minor word changes were made to the scope to include the industry that deals with paint applicator fabrics. Two definitions will be added, "end" and "pick," and the micrometer definition will be more descriptive and reference brand names. In the Determination of Fabric Construction, pile height apparatus will be better explained and referenced to D 1777, and metric units will be added to parenthesis. The chairman will continue working on the document, and a revision will be sent to the group prior to the January meeting.*

*D01.61.05—Bulk Density of Filaments and Bristle—C.F. Nelson, Chairman, reviewed comments which had been received on the proposed method to measure "bulk density of paintbrush filament." Comments indicate dissatisfaction with the use of an insert to spread filament in tapered bundles because the insert damages the filaments.*

The preferred method is to measure the small end of a tapered bundle with a second or modified measuring device which does not require an insert. There was general agreement to eliminate the insert, and it was decided to revise the proposed method to make it applicable only to level filament. The method was reviewed, striking references to tapered filament. The method will be updated and sent to the D01.61 ballot.

C. Martin, will look at the test apparatus and determine if it can be modified to extend the range so that the small end of the tapered bundle can be measured. When this is resolved, the method on tapered filament will be resumed.

*D01.61.06—Buckling Resistance of Filaments*—T. O'Brien, Chairman, reviewed the clamp slippage problem with the current test device. D. Prins has modified the clamp to eliminate slippage. He will be contacted

and his modifications will be incorporated into a new draft which will be reviewed at the next meeting. Once clamp slippage is corrected, a new round-robin will be initiated.

*D01.61.07—Symposium Planning*—T. Sliva, Chairman, reviewed the objectives for the proposed symposium on paint application tools scheduled for the D01 meeting in January 1996.

Following group discussion, it was concluded that the symposium will define brushes, rollers and pads in terms of materials, construction and performance. The importance of the applications to coating film quality will also be emphasized and demonstrated.

G. Harsch, of Wooster Brush, agreed to prepare and present the section on paint brushes. C. Martin, of T.S. Simms Co., tentatively agreed that his company would take

the section on paint rollers. F. Burns, of EZ Painttr, agreed to serve as moderator, and summarized the activities of the subcommittee in ASTM terms. EZ Painttr will take the section on pads and accessories and develop live demonstrations.

Time allowances were discussed within the one to one-and-one-half hour allotted. It was also concluded that visual aids were essential and need to be developed by each presenter. An abstract is to be developed by the June 1995 meeting.

*D01.61*—Subcommittee balloting of two proposed standards, "Test Method for Comparison of the Leveling Efficiency of Paint Brushes," and "Test Method for Evaluation of the Cleanability of Paint Brushes," was reviewed. Both had 88% return and eight affirmatives, one negative, and one abstention. The negative voter in both cases was the task group chairman.

## June 1994 Subcommittee Reports of ASTM Committee G03

### SUBCOMMITTEE G03.01 EDITORIAL AND DEFINITIONS

#### M. Crewdson, Chairman

There were no negatives on the main G03 ballot for revision of G 113 (definition of black box). W. Ketola distributed Draft #3 of the proposed Standard Practice for Conditioning and Handling of Nonmetallic Materials for Natural and Artificial Weathering Tests. This draft will be revised based on changes agreed to at this meeting and will be submitted G03.01 ballot. R. Fischer distributed drafts of two sections to be included in the appendix to the proposed Standard Guide for Application of Statistics to Weathering Test Results: (1) Analysis of Variance, and (2) Rank Correlation. Mr. Fischer reviewed each draft in detail and it was agreed that these would be excellent additions to the proposed guide. This draft standard will be submitted for G03.01 ballot when drafts for the appendix sections covering regression and linear correlation, and use of Student's *t* tests are available.

### SUBCOMMITTEE G03.02 NATURAL ENVIRONMENTAL TESTING

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

The G03 ballot of G 90, "Standard Practice for Performing Accelerated Outdoor

Weathering of Nonmetallic Materials Using Concentrated Natural Sunlight," passed with no negatives and is proceeding to society ballot.

The G03.02 ballot of G 7, "Standard Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials," did not receive enough votes to obtain a valid ballot. Negative votes and comments will be incorporated into a new draft which will be rebalotted.

The G03.02 ballot of G 24, "Standard Practice for Conducting Exposures to Daylight Filtered Through Glass," did not receive enough votes to obtain a valid ballot and will be rebalotted prior to the January 1995 meeting. This document contains a provision to require pre-aging of glass used in under-glass exposure testing.

### SUBCOMMITTEE G03.03 SIMULATED AND CONTROLLED ENVIRONMENTAL TESTING

#### R.M. Fischer, Chairman

The G03 ballots for the revision of the precision and bias statement of G 23, "Standard Practice for Operating Light Exposure Apparatus (Carbon Arc Type) With and Without Water for Exposure for Non-Metallic Materials," G 26, "Standard Practice for Operating Light Exposure Apparatus (Xenon Arc type) With and Without Water for Exposure of Non-metallic Materials," and G 53, "Standard Practice for Operating

Light- and Water-Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Non-metallic Materials," returned with one negative vote. The negative was deemed non-persuasive. The negative asserted that precision and bias is material dependent and judgment about precision cannot be assumed to be generally valid based on one material type which was used in the G03.03 round-robin studies. This same argument had been found non-persuasive during G03.03 ballot. In addition, the negative maintained that the use of weathering reference materials would be precluded by the current wording of the precision statement. The subcommittee decided that an editorial change in wording would clarify the precision statement. The precision and bias statement for these standards (with the editorial change) now pass on to society ballot.

The G03 ballot of G 26 to include Suga Test Instruments Co. as a device supplier in footnote 8 and to eliminate footnote 10, which listed suppliers of xenon-arc optical filters, received no negatives and will pass on to society ballot.

The G03.03 ballot of G 53 to make figures two and three (lamp placement schematics for apparatus types A and B) more generic received no negative votes. This revision now proceeds to G03 ballot.

The subcommittee ballot of a draft Standard Practice for Conducting Light-Exposure Tests for Non-metallic Using Horizontal Specimen Tray Xenon-Arc Exposure Apparatus received six negative votes.

The consensus of the subcommittee was to table work on this standard for one year.

The draft *Guide for Addressing Variability in Natural and Accelerated Testing of Non-metallic Materials* received negatives during G03.03 ballot. The negatives were deemed persuasive. This item is withdrawn, will be redrafted using the suggested changes and will again be submitted for subcommittee ballot.

### **SUBCOMMITTEE G03.05 REFERENCE MATERIALS FOR EXPOSURE TESTS**

**W.D. Ketola, Chairman**

W. Ketola distributed a copy of his final report on the round-robin exposures of polyethylene films in filtered open flame carbon-arcs. The results for this round-robin showed much more variability between labs than in the other round-robins involving exposures of polysulfone in fluorescent UV devices or polystyrene in xenon arc devices. The major source of the variability was the exposure, although the film contributed much more to overall variability than in the other round-robins. This was primarily due to the effect of exposure position, as films exposed at the top of the specimen holder showed a systematically smaller carbonyl index. When these films were removed from the analysis repeatability standard deviation was consid-

erably improved. However, there was no improvement in reproducibility.

An initial draft of a proposed Standard Guide for Characterization of Reference Materials Used for Exposure Tests was distributed for comments and discussion. Several revisions and additions were agreed to and will be incorporated into a draft for subcommittee ballot.

### **SUBCOMMITTEE G03.08 SERVICE LIFE PREDICTION OF NON-METALLIC MATERIALS**

**J.W. Martin, Chairman**

The scope of G03.08 was balloted. There were several negatives. The most common objections were that the scope was too ambitious and that some of the task proposals overlapped the work of other subcommittees. It was agreed that the scope would be rewritten, and that the specific changes suggested in the negatives would be incorporated. Also, the name G03.08 will be changed to "Service Life Prediction of Non-Metallic Materials" in order to avoid any confusion that might arise regarding potential overlap with other ASTM Committees.

Two documents were distributed. The first "Terminology," was a collection of terms commonly used in "reliability analysis." The list of terms was compiled from several cited sources. The second was

JEDEC Standard JESD37 (EIA, October 1992), "Standard for Lognormal Analysis of Uncensored Data, and of Singly Right-Censored Data Utilizing the Persson and Rootzen Method." These two documents provide examples of existing information that can be used as a foundation for the development of future G03.08 standards (i.e., terminology, test methods, practices, specifications, and guides).

Bill Murray gave a brief presentation describing how he uses "reliability theory" to predict the service life of 3M's magneto-optical products. His goal is to be able to predict, with 95% confidence, the rate of early product failures. Since 3M provides a warranty, business profitability depends on being able to determine that more than 97.5% of the units produced will exceed the service life guaranteed by the warranty.

In Murray's example, 16 samples were placed on test. During the time of the test, 10 samples failed and 6 samples survived. The test results were analyzed using four different statistical lifetime distributions. Normal, exponential, Weibull, and lognormal. In this case, the lognormal distribution provided the best fit to the test data. Therefore, service life predictions were made using the lognormal distribution. Mr. Murray used the results of accelerated artificial testing to predict Florida testing performance. An excellent correlation was obtained between predicted performance and actual field performance.

# Society Meetings

## CDIC ..... SEPT.

### Tour of Whirlpool

The meeting began with a tour of the Whirlpool Dryer Plant, in Marion, OH, sponsored by the CDIC Society's Manufacturing Committee. The members viewed the plant finishing lines that included the following application methods: electrocoat, powder coat, and reciprocating turbo belts. The coatings applied included acrylic and polyester topcoats, and epoxy primers.

Society Representative to the Ohio Paint Council, Bill M. Hollifield, of Perry & Derrick Co., Inc., reported that the September issue of the *Federal Register* contains the Proposed Rule for Lead Abatement. He noted that this rule should be examined by all paint companies and concerned coatings people. Mr. Hollifield reminded the members that the Ohio Paint Council's Legislative Day is scheduled for March 26, 1995. He encouraged all members to attend.

Don Roettler presented Society President Al Macheck, of Dow Corning Corp., with the Hüls Gavel.

WILLIAM E. JELF, III, *Secretary*

## CHICAGO ..... SEPT.

### "Solvent-Free Coatings"

The meeting's guest speaker was Andrea Metz, of Rohm and Haas Co., and her topic of discussion was "SOLVENT-FREE ARCHITECTURAL COATINGS UPDATE."

Ms. Metz's presentation covered new developments in polymer and resin technology for solvent-free architectural coatings. She stated that house paints using this new technology do not require the use of coalescing agents, plasticizers, or cosolvents to achieve good film formation. Ms. Metz reported that in addition to the low odor of the paints, test results have shown that formulated solvent-free paints maintain appearance, durability, low-temperature film formation, and open time normally expected from quality conventional latex paints.

According to the speaker, formulation of solvent-free architectural coatings focused on the selection of raw materials that would not contribute any solvents or VOCs to the system. These ingredients include pigment dispersions and surfactants, rheology modifiers, preservatives, and surface wetting agents. Ms. Metz mentioned that guidelines and recommendations for formulating with

solvent-free raw materials were developed through testing the various types and levels of the ingredients. She proceeded to give the results from the testing along with recommended formulations. Physical and performance properties of solvent-free paint formulations were comparable with those formulations containing coalescents and cosolvents, she stated.

During the business portion of the meeting, Mark Biel, Director of the Chemical Industry Council of Illinois, briefed attendees on the importance of their active participation in legislative matters prior to be enacted into law.

CLIFFORD O. SCHWAHN,  
*Publicity*

*Editors Note: For the past six years, Mr. Schwahn has been providing the JCT with the monthly minutes of the Chicago Society. The September minutes mark Mr. Schwahn's last submission, as he is retiring to the sunny state of Arizona. We wish him the best of luck!*

## CHICAGO ..... OCT.

### "Good Tests—Bad Testing"

New York Society Member, Saul Spindel, of D/L Laboratories, delivered the evening's talk entitled, "Good Tests—Bad Testing."

Mr. Spindel's presentation depicted the results of round-robin testing conducted by various industry laboratories of common paint parameters such as grind, gloss, density, viscosity, leveling, and color. The tests,

said the speaker, were done on replicate samples using ASTM test methods. The wide-variety of results reported by Mr. Spindel could only lead to the conclusion that the participating laboratories, in reality, all had secular methods of performing the ASTM tests.

It was announced during the business portion of the meeting that the Coating Technology course sponsored jointly by the Chicago Society and the Chicago Paint and Coatings Association will begin in January 1995.

VICTOR M. WILLIS,  
*Publicity*

## DALLAS ..... SEPT.

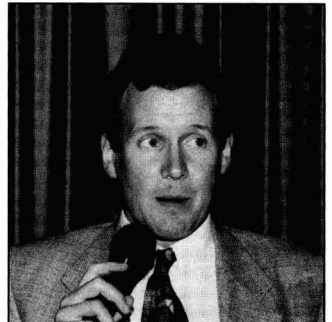
### "Moisture Scavengers"

Society President Benny Puckett, of Kelly-Moore Paint Co., Inc., received the Hüls Gavel from Mike Templin.

President Puckett discussed the idea of establishing a Manufacturing Committee within the Society. Duties of this committee would be to visit paint plants (by invitation), and report on ways to implement the principle tenets of OSHA.

Thomas Johnson, of ANGUS Chemical Co., was the speaker for the evening. He spoke on moisture scavengers and their efficacy in high-solids polyurethane systems.

H. EDWARD SPRADLIN,  
*Publicity*



SEPTEMBER 1994 MEETING OF CHICAGO SOCIETY—Andrea Metz (left picture) updates the attendees on "Solvent-Free Architectural Coating," and Mark Biel (right) discusses legislative matters



## Constituent Society Meetings and Secretaries

**BALTIMORE** (Third Thursday—Martin's West, Baltimore, MD). CONNIE SAUER, Duron, Inc. 10460 Tucker St., Beltsville, MD.

**BIRMINGHAM** (First Thursday—Strathallan Hotel, Birmingham, England). DAVID C. MORRIS, PPG Industries (UK) Ltd., P.O. Box 359, Rotton Park St., Birmingham, B16 0ADS, England.

**CDIC** (Second Monday—Location alternates between Cincinnati, Columbus, Dayton, and Indianapolis). WILLIAM JELF, III, Akzo Nobel Coatings, Inc., P.O. Box 147, Columbus, OH 43216-0147.

**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). RICHARD A. MIKOL, Tremco Inc., 10701 Shaker Blvd., Cleveland, OH 44104.

**DALLAS** (Second Thursday following first Wednesday—Radisson Hotel, Dallas, TX). CHIP NEWCOMB, 1448 N. Joe Wilson Rd., Cedar Hill, TX 75104.

**DETROIT** (Second Tuesday—meeting sites vary). JAN SPALDING, BASF Corp., 26701 Telegraph Rd., Southfield, MI 48086-5809.

**GOLDEN GATE** (Monday before third Wednesday—alternates between Francisco's in Oakland, CA, and Holiday Inn in S. San Francisco). DON MAZZONE, Dowd & Guild, Inc., 14 Crow Canyon Ct., #200, San Ramon, CA 94583.

**HOUSTON** (Second Wednesday—Medallion Hotel, Houston, TX). GUY SULLAWAY, Courtaulds Coatings, P.O. Box 4806, Houston, TX 77210.

**KANSAS CITY** (Second Thursday—Cascone's Restaurant, Kansas City, MO). RANDALL L. EHMER, Walsh & Associates, Inc., 500 Railroad Ave., N. Kansas City, MO 64116.

**LOS ANGELES** (Second Wednesday—Steven's Steakhouse, Commerce, CA). JOSEPH B. EVANS, Trail Chemical Corp., 9904 Gidley St., El Monte, CA 91731.

**LOUISVILLE** (Third Wednesday—Executive West Motor Hotel, Louisville, KY). DAN FORTNEY, American Dispersions Inc., P.O. Box 11505, Louisville, KY 40211.

**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 Bifithèque Steakhouse, St. Laurent, Quebec). LUC MILLETTE, Frank E. Dempsey & Sons Ltd., 2379-46nd Ave., Lachine, Que., H8T 3C9, Canada.

**NEW ENGLAND** (Third Thursday—Sheraton Lexington Hotel, Lexington, MA). GENE C. ANDERSON, Chemecontrol Corp., 38 Spindlewick Dr., Nashua, NH 03062.

**NEW YORK** (Second Tuesday—Landmark II, East Rutherford, NJ). JOHN W. DU, Hills America, Inc., P.O. Box 365, Piscataway, NJ 08854.

**NORTHWESTERN** (Tuesday following first Monday—Jax Cafe, Minneapolis, MN). JOSEPH J. MILLS, Milsolv® Corp./Minnesota, 2340 Rose Pl., Roseville, MN 55113.

**PACIFIC NORTHWEST** (PORTLAND SECTION—Tuesday before third Wednesday—Tony Roma's, Mall 205, Portland, OR; SEATTLE SECTION—Third Wednesday—Wyndham Garden Hotel, Sea-Tac, WA; VANCOUVER SECTION—Thursday after third Wednesday—Abercorn Inn, Richmond, B.C.). EDWARD LINTON, Cloverdale Paint Co., 6950 King George Hwy., Surrey, B.C., V3W 4Z1, Canada.

**PHILADELPHIA** (Second Thursday—Williamson's Restaurant, GSB Bldg., Bala Cynwyd, PA). THOMAS G. BROWN, Consultants Consortium, 209 Fox Ln., Wallingford, PA 19086.

**PIEDMONT** (Third Wednesday—Ramada Inn Airport, Greensboro, NC). ROY MODJEWSKI, Akzo Nobel Coatings, Inc., 1431 Progress St., High Point, NC 27261.

**PITTSBURGH** (Second Monday—Monamurro's Restaurant, Sharpsburg, PA). JAMES REDISKE, Miles Inc., Mobay Rd., Pittsburgh, PA 15205.

**ROCKY MOUNTAIN** (Monday following first Wednesday—Zangs Brewery, Denver, CO). CHARLES SHROEDER, Fel-Pro Inc., 6120 E. 58th Ave., Commerce City, CO 80022.

**ST. LOUIS** (Third Tuesday—The Salad Bowl Restaurant, St. Louis, MO). MICHAEL P. HEFFERON, Walsh & Associates, Inc., 1801 S. Hanley Rd., St. Louis, MO 63144.

**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). WALTER R. NAUGHTON, JR., Scott Paint Corp., 7839 Fruitland Rd., Sarasota, FL 34240.

**TORONTO** (Second Monday—Meeting sites vary). NATALIE JANOWSKY, Degussa Canada Ltd., 4261 Mainway Dr., Burlington, Ont., L7R 3Y8, Canada.

**WESTERN NEW YORK**—MARKO MARKOFF, 182 Farmingdale Rd., Cheektowaga, NY 14225.

## GOLDEN GATE ..... OCT.

### Past-Presidents Night

Golden Gate Society Past-Presidents in attendance at the meeting included: Garnet Grieve (1939); A. Gordon Rook (1965); Barry Adler (1966); A. Lynn Axelson (1967); Neil Estrada (1968); Larry Graubart (1969 and 1970); Lowell Cummings (1973); Ken Trautwein (1985); Patricia Shaw (1987); Tim Donlin (1988); Gordon Pioch (1991); Marnie Hartmann (1993); and Dennis Owens (1994).

Bob Backlin performed his annual presentation of the Hills Gavel to incoming President Donald Nolte, of John K. Bice Co.

Society and Federation Past-President Neil Estrada presented Garnet "Rosie" Grieve with an FSCT 50-year membership pin.

At this meeting, the Golden Gate Society awarded three \$1,000 scholarships to the following individuals: Benjamin Taylor, son of Rinette Taylor, of The Glidden Co.; Stephen Conn, son of Jonny Conn, of Triangle Coatings; and Alfred Nicolas, Jr., son of Alfred Nicolas, of Connell Brothers.

DON MAZZONE, *Secretary*

## NORTHWESTERN ..... SEPT.

### "Novel Waterborne Technology"

It was reported that Society member Richard Gurney passed away on September 12.

Incoming President Michael Grivna, of Hirshfield's Paint Mfg., Inc., received the President's Gavel from Joe Wirth, of Consolidated Container Corp.

President Grivna made mention that the Society, in conjunction with the Minnesota Office of Waste Management, will be co-sponsoring the Industrial Paint and Powder Exposition.

The meeting's first speaker was Frederick A. Giles, Jr., of Union Carbide Corp. The topic of his discussion was, "NOVEL WATERBORNE TECHNOLOGY FOR HIGH-GLOSS ALKYD REPLACEMENT."

Mr. Giles began by stating that waterborne resin technology has made great strides in recent decades to fulfill the requirements of architectural coatings. Performance features associated with solvent-borne alkyd coatings include excellent application rheology leading to high gloss, good adhesion to most surfaces, early block resistance, and high pigment binding capacity. Less favorable features include UV-induced chalking,



SEPTEMBER DALLAS SOCIETY MEETING—Mike Templin (left) presents Dallas President Benny Puckett with the Hüls Gavel

yellowing, loss of gloss, and film embrittlement.

According to the speaker, recent advances in emulsion polymerization technology have now made possible latex vehicles which meet the requirements of low VOC/low odor systems at competitive prices. One advantage of emulsion polymerization, compared to solution polymerization, is the development of high molecular weight polymers while maintaining low dispersion viscosity. A second key feature of emulsion polymerization is the ability to produce "structured particles" that can aid in film formation without being too soft for architectural applications.

Mr. Giles highlighted the fact that the driving force for the film forming process is the reduction of surface area that exists because of the particulate nature of emulsion polymers. It is a three-step process:

- (1) Water evaporation from the film;
- (2) Plastic deformation and coalescence of the particles; and
- (3) Cohesive strength development through inter-diffusion of molecular chains across the interface.

Film formation, stated the speaker, is a function of a variety of chemical and physical variables. The most common modification of latex polymers to impact film formation is to include in the monomer mix relatively low levels of polar monomers (e.g., methacrylic acid).

The evening's second speaker was Michael S. Dosch, of Primerica Financial Services, and he spoke on the "FIVE CORNERSTONES FOR FINANCIAL SECURITY."

Mr. Dosch went into detail on the five cornerstones for financial security. They are:

- (1) Fully protect your family.
- (2) Build up your rainy day fund/emergency fund.
- (3) Maximize long-term investments.
- (4) Have a written financial plan and budget, be debt free.
- (5) Keep a current written estate plan.

The key to success, according to Mr. Dosch, is to make consistent progress in each of these key areas.

JOSEPH J. MILLS, *Secretary*

## NORTHWESTERN.....OCT.

### "Environmentally Friendly Coatings"

Educational Committee Chairman H. Mustapha Bacchus, of The Valspar Corp., updated members on three projects currently underway by the committee: (1) the development of an "educational kit" to be used at the high school level; (2) trying to help boost dwindling membership; and (3) creating a public service video on "Coatings Science Education."

The members were informed that Lowell Wood has retired as a Technical Advisor on the North Dakota State University's Coating Panel after 23 years of service.

The technical presentation entitled, "ADDITIVE USAGE IN ENVIRONMENTALLY FRIENDLY COATINGS," was delivered by Edward W. Orr, of BYK-Chemie USA.

Mr. Orr stated that, for the past few decades, dramatic technology shifts have occurred in the coatings industry. Rapid changes in pigments, resins, and solvents have helped make environmentally friendly coatings possible. However, such changes have also created many technical hurdles.

With modern day coatings having special needs in regard to rheology, film performance, and surface characteristics, Mr. Orr mentioned that new additives have evolved to meet the challenge. For instance, a particular high-solids coating may require special wetting and dispersing additives in order to match the performance of comparable low-solids systems. In another high-solids coating, remarked the speaker, interfacially active silicones and defoamers may be necessary to maximize properties.

Answers to the crucial question as to how can such parameters be optimally controlled can be outlined by describing the proper methodologies to control interfacial tension. According to Mr. Orr, interfacial tension itself is an important phenomenon which can serve as the basis to describe what happened at the various interfaces encountered in the manufacture, application,

and use of modern coating systems.

The speaker continued by stating that physicochemical, steric, and dynamic particle movement variables must be controlled and coordinated within the coating system so that the final coating product is a completely stabilized system. Pigment and resin design area, of course, important methods of initial control.

Mr. Orr emphasized that interfacial tension is the most important determining variable in the study of wetting, dispersing, and surface control, whether the coating system is water- or solvent-based, UV-cured, or even powder based. Many defects in the surface of a paint film can be explained by differences in interfacial tension. They are:

- poor substrate wetting;
- spray dust sensitivity;
- cratering;
- formation of Benard cells;
- ghosting, wipe marks;
- poor recoatability; and
- air-draft sensitivity.

Eliminating these differences, said the speaker, will prevent surface and interfacial defects.

Mr. Orr noted that silicone additives are ideally suited to adjust and control interfacial tension; furthermore, they can minimize interfacial tension differences.

In conclusion, the speaker stated that the control of interfacial tension service as the prime driving force behind the design of new additives. New generation additives for waterborne and high-solids coatings face an increasing array of demands in regard to wetting, dispersing, and surface control.

The second speaker of the meeting was Albert C. Holler, who was representing the Boy Scouts of America—Viking Council. He discussed "BOY SCOUTING ANECDOTES."

JOSEPH J. MILLS, *Secretary*

## Anniversaries

### New York Society 25-Year Members

RICHARD O. INNES—O.G. Innes Corp.,  
New York, NY

JAMES A. O'NEILL—Tenax Finishing  
Products Co., Newark NJ

NORMAN M. RUSSO—Daicolor-Pope,  
Inc., Paterson, NJ

RONALD G. SPITZER—Troy Chemical  
Corp., E. Hanover, NJ

JOSEPH A. VONA—Westfield, NJ

EDWARD WANDERMAN—Marlboro, NJ

WILLIAM P. WEBER—New York, NY

# Elections

## CLEVELAND

### Active

*Bauer, Paul D.*—Kool Seal Inc., Twinsburg, OH.  
*Brooks, Lamar E.*—Coatings Research Group Inc., Cleveland, OH.  
*Buhovecky, Allen M.*—Plasti-Kote Co., Inc., Medina, OH.  
*Crites, Donald*—The Sherwin-Williams Co., Bedford Hts., OH.  
*Fortuna, Robert B.*—Pioneer Manufacturing Co., Cleveland.  
*Ilko, Mary M.*—Sprayon Products, Bedford Hts.  
*Pearce, Tyrone D.*—The Sherwin-Williams Co., Bedford Hts.  
*Rudowski, Margaret A.*—The Sherwin-Williams Co., Bedford Hts.  
*Sajner, George S.*—Pioneer Manufacturing Co., Cleveland.  
*Slapnickner, Randy L.*—Spray-Cure Co., Madison, OH.

### Associate

*Armstrong, Virginia A.*—Davies Can Co., Solon, OH.  
*Lueffen, Michael S.*—Lajack & Associates, Rocky River, OH.  
*Roberts, Tina E.*—The Goodyear Tire & Rubber Co., Akron, OH.  
*Yosh, James P. Jr.*—Henkel Corp., Aurora, OH.

## DETROIT

### Active

*Baghdachi, Jamil*—BASF Corp., Southfield, MI.  
*Brunori, David J.*—Finish Technologies, Walled Lake, MI.  
*Clifford, John M.*—Red Spot Paint, Plymouth, MI.  
*Freeman, Manju Kogpeh*—United Paint & Chemical Corp., Southfield.  
*Fry, Michael C.*—Standard Paint Co., Detroit, MI.  
*Gentle, Cindy L.*—Red Spot Westland Inc., Westland, MI.

*Grilli, David A.*—The Sherwin-Williams Co., Troy, MI.  
*Gunn, Valerie E.*—Solvent Chemical Co., Inc., West Warwick, RI.  
*Henry, Karl F.*—BASF Corp., Southfield.  
*Horn, Ellen M.*—BASF Corp., Southfield.  
*Kow, Cheong*—The Sherwin-Williams Co., Troy.  
*Liles, Donald T.*—Dow Corning Corp., Midland, MI.  
*Locascio, Thomas S.*—Red Spot Westland Inc., Westland.  
*McNeal, Karen M.*—Shell Chemical Co., Houston, TX.  
*Meissner, Tracy L.*—BASF Corp., Southfield.  
*Mitkus, Marianne J.*—The Sherwin-Williams Co., Troy.  
*Modarressia, Antony H.*—Standard Paint Co., Detroit.  
*Myren, James T.*—Seibert Oxidermo, Romulus, MI.  
*Parsons, Dennis R.*—BASF Corp., Detroit.  
*Patel, Raman B.*—Standard Paint Co., Detroit.  
*Plank, Adam E.*—Akzo Nobel Coatings, Troy.  
*Pohl, Mark P.*—BASF Corp., Southfield.  
*Porter, Kathleen A.*—Morton International, Rochester Hills, MI.  
*Remer, Thomas A.*—The Sherwin-Williams Co., Troy.  
*Salisbury, Gary W.*—BASF Corp., Southfield.  
*Smallwood, Jeffrey A.*—Glidden Paint Co., Niles, MI.  
*Stayart, Scott H.*—Chidley & Peto, Grand Haven, MI.  
*Swart, Daniel J.*—Dow Chemical Co., Midland.  
*Verville, Martin W.*—Cooper Industries, Inc., Sturgis, MI.  
*Ziemer, Patrick D.*—Akzo Nobel Coatings, Troy.

### Associate

*Erickson, Robert L.*—McWhorter Technologies, Carpentersville, IL.

## LOS ANGELES

### Active

*Adams, Lionel*—San Gabriel, CA.

*Adan, Ernesto R.*—United Coatings, Inc., Los Angeles, CA.  
*Burkes, Louie E.*—Peralta, NM.  
*Cline, Mary A.*—Synergistic Performance, Fullerton, CA.  
*Conde, Blessy L.*—Ellis Paint Co., Los Angeles.  
*DeLeon, Robert O.*—PFI Paints For Industry, Santa Fe Springs, CA.  
*Dickinson, Sandra*—FineLine Paint, Santa Fe Springs.  
*Ebrahim, Yousry, M.*—Courtaulds Aerospace, Burbank, CA.  
*Flores, Richard*—Smiland Paint Co., Los Angeles.  
*Holzrichter, Geoffrey R.*—Dura Coat Products, Fontana, CA.  
*Ghazarian, Ara B.*—United Coatings, Los Angeles.  
*Grubbs, Charles L.*—Lilly Industries, Inc., Montebello, CA.  
*Hong, Myung K.*—Dura Coat Products, Fontana.  
*Jerge, David C.*—Frazee Industries, San Diego, CA.  
*Kapadia, Shailan J.*—Engineered Polymer Solutions, Inc., Los Angeles.  
*Kipnis, Igor*—Dunn-Edwards Corp., Los Angeles.  
*Ko, Renita U.*—Guardsman Products, South Gate, CA.  
*Lauritis, Daniel J.*—DuPont White Pigments & Minerals Products, San Ramon, CA.  
*Lazarus, Richard M.*—SDC Coatings Inc., Anaheim, CA.  
*Lim, William T.*—Consolidated Color Corp., Hawaiian Gardens, CA.  
*Martinez, Jaime A.*—Dunn-Edwards Corp., Los Angeles.  
*Mislang, Feliciano B.*—Consolidated Color Corp., Hawaiian Gardens.  
*Morales, Ezequiel S.*—Frazee Industries, San Diego.  
*Muldown, Patrick E.*—Consolidated Color Corp., Hawaiian Gardens.  
*Quintana, Zita*—Dura Coat Products, Fontana.  
*Peters, Frank AC*—Delta Technical Coatings, Inc., Whittier, CA.  
*Radnia, P. Matthew*—Brogdex Co., Pomona, CA.  
*Shchors, Inna*—Dunn-Edwards Corp., Los Angeles.  
*Skabin, Alan J.*—Galpin, Sepulveda, CA.  
*Subbaraman, Ramesh B.*—Independent Ink Inc., Gardena, CA.  
*Teets, John B.*—Smiland Paint Co., Los Angeles.  
*Tilley, Mark C.*—SDC Coatings, Inc., Anaheim.

### Associate

*Berger, Mark*—EPS, Los Angeles, CA.  
*Dearth, Geoffrey J.*—Air Products and Chemicals, City of Industry, CA.  
*Favreau, Mike*—Dowd and Guild Inc., Pico Rivera, CA.  
*Morgan, Doug*—Whittaker, Clark & Daniels, Laguna Hills, CA.  
*O'Donnell, Margaret A.*—H.M. Royal of California Inc., Buena Park, CA.  
*Pabich, Dan*—Harcros Pigments, Inc., Irvine, CA.  
*Pica, Dana*—Ashland Chemical, Inc., Orange, CA.

### Q. How can subscribing to the JOURNAL OF COATINGS TECHNOLOGY help you in your daily life in the Coatings Industry?

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Powell, Steven K.—Dicalite, Aliso Viejo, CA.  
Savard, Michael D.—Frazee Industries, San Diego, CA.

Yurosko, Richard N.—R.N. Yurosko & Associates, Orange.

#### Educator/Student

Draper, Glenn H.—Union Carbide Corp., Torrance, CA.

Mejia, Hector A.—Cal-Poly Pomona, Cudahy, CA.

Squires, Joe—Cal-Poly University, Los Osos, CA.

## PIEDMONT

### Active

Cox, Randolph G.—Akzo Nobel Coatings, High Point, NC.

Frye, Gene—Guardsman Products, High Point.  
Harris, David C.—High Point Chemical, High Point.

Holman, Ronnie G.—Chemical Coatings, Hudson, NC.

Patterson, Teena M.—Guardsman Products, High Point.

Reynolds, Steven T.—Union Carbide Corp., Tucker, GA.

Robinson, Greg F.—Akzo Nobel Coatings, High Point.

Warren, Ernest R.—Guardsman Products, High Point.

### Associate

Bateman, Henry W.—Magnesium Elektron, Matthews, NC.

Hoffman, Clarence D.—Kuhl Marketing, Greensboro, NC.

Kraniak, Lynn G.—Luzenac America, Roswell, GA.

Linhart, Theodore V.—NPI, Catawba, SC.

Sheek, Jeffrey K.—Perstorp, Roswell.

Wajalovsky, Steve A.—Penn Color, Doylestown, PA.

## PITTSBURGH

### Active

Docherty, Michael J.—Concurrent Tech. Corp., Johnstown, PA.

Wright, Michael W.—J.M. Huber Corp., Coraopolis, PA.

## TORONTO

### Active

Forber, Christine L.—ICI Autocolor, Toronto, Ont.

Graham, Kelly S.—Lorama Chemicals Inc., Milton, Ont.

LeAndres, Antonio P.—Akzo Coatings, Woodbridge, Ont.

Mitra, Sarbani—DuPont Canada, Ajax, Ont.

Nazarali, Shabnaz—Lorama Chemicals Inc., Milton.

### Associate

Stumpft, Mark L.—Imperial Oil, Toronto, Ont.

# Future Society Meetings

## Baltimore

(Nov. 17)—39th Annual Awards Night.

## CDIC

(Dec. 12)—“PROPERTIES OF CROSSLINKED POLYURETHANE DISPERSIONS”—Valentino Tramontano, King Industries.

(Jan. 9)—“ACRYLIC-POLYURETHANE HYBRID DISPERSIONS AND THEIR USE IN WATERBORNE COATINGS”—Charles R. Hegedus, Air Products and Chemicals.

(Mar. 13)—Manufacturing Program.

(Apr. 10)—CDIC 75th Anniversary.

(May 8)—“EVALUATION OF NEW GENERATION COALESCING AGENTS FOR INDUSTRIAL ACRYLIC LATEXES”—Thomas M. Larson, Exxon Chemical Co.

## Chicago

(Jan. 9)—“REACTIVE OXAZOLIDINE MOISTURE CONTROL ADDITIVES FOR POLYURETHANE COATINGS”—Thomas L. Johnson, ANGUS Chemical Co.

(Feb. 6)—“DESIGNING FOR QUALITY”—Laura J. Hansen, Orr & Boss.

(Mar. 6)—“SOLVING PRACTICAL ADHESION PROBLEMS IN COATINGS WITH FUNDAMENTAL STUDIES IN SURFACE AND BULK EFFECTS OF ADHESION”—Krishan C. Sehgal, Union Carbide Chemical & Plastics Co., Inc.

(Apr. 4)—“NEW TECHNOLOGY IN SMALL MEDIA MILLING”—Harry Way, Netzsch, Inc.

(May 12)—Annual Awards Banquet.

## Cleveland

(Jan. 17)—Joint Meeting with CPCA, State of the Industry—John Danziesen, Glidden/ICI America.

(Feb. 21)—“FOAM & FOAM CONTROL AGENTS”—Andrew Romano, Drew Division/Ashland Chemical.

(Mar. 21)—“USING KAOLIN PIGMENTS TO REPLACE CRYSTALLINE SILICA”—Thad T. Broome, J.M. Huber Corp.

(Apr. 18)—“UNIQUE WETTING & DISPERSING ADDITIVES”—Edward Orr, BYK-Chemie.

(Apr. 28)—Educational Symposium.

(May 16)—Awards/Past President's/Spouses' Night.

## Golden Gate

(Jan. 16)—“UTILIZATION OF A NEW REACTIVE DILUENT TO FORMULATE LOW VOC COATINGS”—Dharma Kodali, Cargill Technical Oils.

(Mar. 13)—“USE OF SMECTITE CLAY MINERALS IN WATERBORNE SYSTEMS”—Dwayne Siptak and Bob Briell, Southern Clay Products.

(Apr. 17)—“EASILY DISPERSIBLE, LOW OIL ABSORPTION, OPACIFYING ORGANIC PIGMENTS ENCAPSULATING TITANATED LITHOPONE”—Michael Issel, Sino American Pigments.

(May 15)—“HIGH-SPEED DISPERSION TECHNIQUES”—Rocky Courtain, Morehouse Industries, Inc.

(June 19)—Manufacturing Committee Seminar.

## Montreal

(Jan. 11)—“ZERO VOC COLORANTS”—Eliot Burrows, Engelhard Corp.

(Feb. 2)—Mini Symposium.

(Mar. 1)—“ACCELERATED TESTING”—Dwight Weldon, KTA-Tator.

(Apr. 5)—“RHEOLOGY MODIFIERS”—Bob Briell, Southern Clay Products.

(May 3)—“EPOXY TECHNOLOGY”—Elizabeth Wrobel, Air Products/Pacific Anchor.

## Pittsburgh

(Jan. 9)—“COMPLIANT COATINGS/PROBLEMATIC PAINTS”—Clifford Schoff, PPG Industries, Inc.

(Feb. 13)—“NATURAL VS. ARTIFICIAL WEATHERING AND THE EQUIPMENT AVAILABLE”—Atlas Electric Corp.

(Apr. 10)—Joint Meeting with SSPC, PDCA, NACE, and PSCT.

(May 8)—“COLOR TRENDS FOR THE COMING YEAR”—PPG Industries, Inc. Past-Presidents' and Spouses' Night.

## Toronto

(Nov. 19)—75th Anniversary Celebration.

(Jan. 9)—“WATERBORNE INDUSTRIAL DISCUSSION”

(Feb. 13)—“POWDER COATINGS—YESTERDAY, TODAY, AND TOMORROW”—David Barlow, The Valspar Corp.

(Mar. 6)—“RADIATION CURING TECHNOLOGY FOR THE 90s AND BEYOND”—Robert A. Lie Berman, Henkel Corp.

(Apr. 10)—Technical Symposium.

(May 8)—“Non-Toxic Anticorrosive Pigments in Aqueous Media”—Robert E. Snyder, A.R. Monteith (77) Ltd. (Toronto Society Technical Committee Presentation)

## People

Kelco Division of Merck & Co., Inc., San Diego, CA, has announced the reorganization of its Marketing Department. Taking on new titles are: **Robert Speights**—Vice President of Marketing; **David Izzard**—Director of Marketing; and **John Johnstone**—Director, Product Planning and Market Research.

Liquid Carbonic, Oak Brook, IL, has announced the appointment of three Vice Presidents in the Industrial Gases Operations. The Vice President, Operations is **David Keierleber**. Mr. Keierleber will spearhead the production and distribution of the company's domestic carbon dioxide and atmospheric gases, as well as for plant engineering and construction.

**Jim Muth** accepted the position of Vice President, Engineering. Mr. Muth will be responsible for customer service, engineering, freezing equipment manufacturing, and technical support.

**Jim Nesmith** was named Vice President, Information Technology. Mr. Nesmith will redesign and manage the computer and information systems.



M.H. McGrath

PPG Industries, Pittsburgh, PA, has named **Margaret H. McGrath** President of its PPG Canada Inc. subsidiary and Vice President, Coatings and Resins, Canada. Ms. McGrath succeeds **James W. Craig**, who was recently appointed President, PPG Europe, PPG Industries International, and Vice President, Coatings and Resins, Europe.

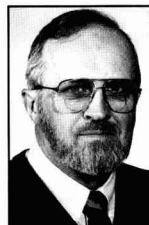
**Michael A. McPherson**, was named Market Development Manager, Powder Coatings. In his new role, Mr. McPherson will work with can manufacturers and equipment suppliers to provide solutions to the packaging industry using powder coatings. He will manage the application laboratory and all customer projects in the Americas region.

CYTEC Industries Inc., West Paterson, NJ, has elected **Charles C. Corcoran** as Vice President Asia/Pacific. Mr. Corcoran will be responsible for operations in the Asia/Pacific region. In addition, he is Chairman of Mitsui-CYTEC Ltd., a joint venture with Mitsui Toatsu Chemicals.

**William C. Feist**, Research Chemist at the USDA, Forest Service, Forest Products Laboratory (FPL), Madison, WI, is retiring after 30 years of research on polymers and wood finishing science.

Mr. Feist joined the FPL staff in 1964 and focused on wood-water relationships, wood/polymer interactions, and wood surface chemistry. For the last 20 years, his research has been directed at the study of surface treatments and finishes that improve and protect outdoor wood surfaces, the chemistry of wood surface/coating interactions, and the weathering of wood and its control.

A member of several national and international research, honor, and professional societies, Mr. Feist has written more than 180 technical, scientific, and practical publications that emphasize finishing and protecting exterior wood surfaces.



W.C. Feist

**Martin Lassen** has joined the Environmental Products Group at Johnson Matthey, Wayne, PA, as a Market Development Specialist. Mr. Lassen's responsibilities will be to identify, quantify, and develop marketing strategies targeting both new and existing markets.

**Gary A. Thompson** has accepted the promotion to Technical Service Representative for the Specialty Coating Color Lab area of Red Spot Paint and Varnish Co., Inc., Evansville, IN.

Also, **Barbara L. Catt** was promoted to Programming Supervisor of the Information System Department.

In addition, **Timothy A. DeVault** was promoted to Research Center Facility Manager for the Red Spot Research and Development Center. Mr. DeVault has over six years experience in the maintenance area and will be responsible for the operation of the mechanical and janitorial maintenance of the center.

**Robert J. Poemer** has accepted the promotion to Director of Business Development within the Colorants Group at Hüls America Inc., Piscataway, NJ. Mr. Poemer will develop, coordinate, and implement business strategies for the company.

Also, **George Parnell** has been appointed Corporate Counsel. He will assist in all legal activities related to Hüls America Inc.

**Ying-Kao Lee**, DuPont Research Fellow, DuPont Automotive, is the recipient of an Honorary Professorship bestowed by The Institute of Chemistry, Beijing, China. Dr. Lee became a DuPont Fellow in 1989. He works at the company's Marshall Laboratory, in Philadelphia, PA, and his 29-year career at DuPont includes contributions to the development of paints for the automotive industry.

**Dennis Tabisz** was elected President and Chief Executive Officer of E.W. Kaufmann Co., Southampton, PA. Mr. Tabisz most recently served as Executive Vice President in charge of sales.

Ultra Additives, Inc., Paterson, NJ, has named **Roe Canning** as Information Services Analyst. Ms. Canning will be responsible for all programming and hardware specifications.

The Dow Chemical Co., Midland, MI, has announced three appointments in the Plastics business group. **R.N. (Bo) Miller** was appointed Market Group Manager for North American Epoxy Resin Products.

**Christopher Gann** was named Business Operations Manager for the North American Epoxy and Intermediates Group.

The group also appointed **Dave Basque** to the position of Product Manager for Converted Epoxy Resins and Market Manager for Brominated Epoxy Resins for North American Epoxy Resin Products.

SC Johnson Polymer, Racine, WI, has reorganized the company's top management. The following have taken on new titles and responsibilities: **Dennis K. Bollenbach**—Regional Director, Americas; **Larry W. Berger**—Global Director, Coatings; **Denise Schendel**—Regional Director, Asia/Pacific; **Adrie L. Kok**—Regional Director, Europe; and **Richard A. Barry**—Global Director, Graphic Arts and Adhesives.

## Obituary

**Richard (Dick) Gurney**, a member of the Northwestern Society, died recently. Mr. Gurney's employment history included The Glidden Co., H.B. Fuller Co., and Albeco Consulting.



### 22nd Western Coatings Societies' Symposium and Show To Feature 39 Papers in Concurrent Sessions

The 22nd Western Coatings Societies' Symposium and Show (WCSSES) will be held on February 20-22, 1995 at the Hilton Hotel and Towers, San Francisco, CA. The event is sponsored by the Golden Gate, Los Angeles, Pacific Northwest, and Rocky Mountain Societies for Coatings Technology.

The symposium on "New Opportunities, Challenges & Solutions" will feature three days of concurrent sessions of technical presentations. A total of 39 papers are scheduled for presentation in the following topic areas: additives, epoxy, urethane, regulatory affairs, solvents, computers and color measurement, pigments and extenders, polymers, latex paints, and miscellaneous. Topics to be discussed during the symposium include:

"Polyethylene Homopolymers and Copolymers in VOC Compliant Coatings Systems"—Anne Tiefenthaler, of AlliedSignal Corp.;

"Additive Use in Environmentally Friendly Coatings"—Edward Orr, of BYK-Chemie;

"Unique Wetting and Dispersing Additives in High-Solids and Waterborne Systems"—Robert McMullin, of BYK-Chemie;

"Aqueous Architectural Coatings from an Additives Perspective"—Marc Hirsh, of Rhone-Poulenc;

"The Determination of VOCs in UV-Curable Coatings: Practical Considerations for the Use of ASTM Method D 5403"—John P. Guarino, of UCB Radcure, Inc.;

"Fluorescence, Velvet, and Candy Apples Problems with Instrumental Color Measurement"—R.D. Athey Jr., of Athey Technologies;

"Managing Operations Efficiently with PC-Based Software: Manufacturing, Formulating and Compliance Control"—Christy

Hudson, of Pacific Micro Software Engineering;

"Innovative Curing Agents for High-Solids Epoxy Coatings"—David Dubowik, of Air Products;

"The Influence of the Solvent Polarity on the Curing Properties of Phenalkamines"—Anton Constantinescu, of Cardolite Corp.;

"Cost Effective, High Performance Epoxy Coatings for Today's Marine and Maintenance Applications"—Zhisheng Dai, of Cardolite Corp.;

"Innovations in Epoxy Technology"—Marcel Gashke, of Ciba-Geigy Corp.;

"Acrylic High-Solids Clearcoats—How to Reduce VOC and Improve Performance

Using Glycidyl Esters"—Richard Ryan, of Exxon Chemical;

"A Review of Ambient Cure, Waterborne Epoxy Resin Coatings Technology"—D.J. Weinman, of Shell Development;

"New Development in Acrylic Technology for Exterior Architectural Coatings"—William Hill, of Rohm and Haas;

"Low VOC Latex Paint; TiO<sub>2</sub> Product Selection and Formulation Guidelines"—Cal Tatman, of SCM Corp.;

"Development of Hydrophobic Waterborne Facade Coatings"—Jay Adams, of Tego Coatings;

"Color Acceptance of Latex Paints Formulated with Opaque Polymer"—Dane R.

*(Continued on next page.)*

### Cleveland Society To Host Environmental Symposium

Plans have been announced for the Cleveland Society's Environmental Seminar scheduled for November 15 at the Hilton Inn, Cleveland, OH.

Scheduled topics to be discussed include: "Clean Air Act: Title V Rules"—Jeanne Mallett and Tom Rigo, of Ohio EPA;

"90s Initiative for Industry"—Ed Bish, of Jamestown Paint;

"Clean Water Program: Ohio's Current and Long-Range Plans"—Speaker from Ohio EPA Northeast District;

"Ohio Paint Council: Working with Ohio's Paint Industry"—John Milgrim, Excel Corp.; and

"Waste Management"—Speaker from Ross Environmental.

The agenda also includes a visit by Federation Officers and the first "Bosses Night." Following the monthly meeting, Jim Sainsbury, of The Glidden Co., will deliver

the evening presentation. The day's events will conclude with an attendees' lottery.

For more information on the CSCT's Environmental Seminar, contact Michael Wolfe, Seegott Inc., 5400 Naiman Parkway, Solon, OH 44139.

### "Clean Air & Technology" Theme for the Piedmont Society's Mini Show and Symposium

The Piedmont Society for Coatings Technology will host a Mini Technical and Exhibition Show on March 15, 1995, at the Showcase On The Park, High Point, NC.

The theme for the technical sessions and show is "Clean Air and Technology" and is designed to provide a forum for technical, purchasing, production, and plant personnel from local and regional coatings, ink, adhe-

sives, plastics, and rubber manufacturers. Suppliers to these industries will have an opportunity to display their products and services to technicians, formulators, purchasing, and production personnel.

For more information, contact Richard Chodnicki, Van Horn, Metz & Co., 320 Townsontown Blvd., Ste. 205, Baltimore, MD 21204.

### Call for Papers New York Society Symposium

May 3-4, 1995  
Holiday Inn North  
Newark Airport, NJ

The New York Society for Coatings Technology is inviting authors to submit papers for their symposium "Recent Advances in Modifiers for Modern Coatings" to be held May 3-4, 1995, in Newark, NJ.

All prospective authors interested in presenting a paper on this theme should submit an abstract of 150 words or less. The abstract should detail the main theme and principal points and should be comprehensive enough to allow assessment of the scope and content of the paper.

To submit an abstract or receive additional information, contact Larry Waelde, Troy Chemical Corp., One Avenue L, Newark, NJ 07105.

## WCSSS Technical Session Features 39 Presentations

(Continued from previous page)

Jones, of California Polytechnic State University;

"Re-Engineering Your Dispersion Line"—J.A. Schak, of Kady International;

"The Cal Poly SLO Polymers and Coatings Program from Student to Coatings Chemist"—James Westover, of California Polytechnic State University;

"Aluminum Trihydroxide as a Filler for Transparent UV-Curable Polymers: Effects of Loading, Particle Size, and Resin Composition"—Terrance Clever, of Alcoa;

"Atapulgite Thickeners—Proven Rheology Modifiers"—Ashok Khokhani, of Engelhard Corp.;

"A Challenge: Aluminum Pigments in Aqueous Coatings"—David Chapman, of Silberline Manufacturing Co.;

"A New Class of Opacifying, Dispersible, Organic/Inorganic Hybrid Pigments"—Mike Issel, of Sino American Pigment Systems, Inc.;

"Organic Pigments for Waterborne Industrial Coatings"—Romesh Kumar, of Hoechst Celanese Corp.;

"The Safe Handling and Usage of Silica Containing Minerals"—Gary Hendrickson, of Unimin Specialty Minerals, Inc.;

"A New Reactive Diluent with Excellent Functional Properties to Reduce VOCs in Solvent-Borne Paints and Coatings"—Dharma Kodali, of Cargill Central Research;

"Glydext Glycidyl Esters as Reactive Diluents"—Richard Ryan, of Exxon Chemical;

"High Performance Waterborne Dispersions for Very Low VOC Coatings"—Rich Johnson, of McWhorter Technologies;

"Volatile Silicone Fluids as Potential Exempt Solvents in Protective Coatings"—William Finzel, of Dow Corning Corp.;

"The Role of Water Immiscible Solvents in the Water Resistance of Acrylic/Urethane Paints"—Tom Larson, Exxon Chemical;

"Evaluation of Diacetone Alcohol as a Coating Solvent"—R.W. Lee, of Shell Development;

"Solvent Selection in Today's Environment—Challenges and Opportunities"—Don Sullivan, of Shell Development;

"The Effect of Coalescent Choice on Film Properties of Waterborne Industrial Latexes"—Peter A. Doty, of Dow Chemical Co.;

"Formulating Water-Based Systems with P-Series Glycol Ethers"—Bernadette Corujo, of ARCO Chemical Co.;

"Smog Wars: The Regulatory Strikes Out"—Robert Wendoll, of EL RAP;

"Reactive Moisture Control Additives for Polyurethane Systems"—Thom Johnson, of ANGUS Chemical;

"Waterborne Oil Modified Urethane"—Glen Petschke, of Reichhold Chemicals, Inc.;

"The Current Status of Regulatory Problems and Regulatory Affairs"—Ned Kisner, of Triangle Coatings; and

"Braving the Business Climate in California"—Matt Dustin, of California Paint Council.

For more information, contact Gordon Pioch, c/o Eureka Chemical Co., P.O. Box 2205, S. San Francisco, CA 94083. For booth reservations, contact Tom Dowd, Dowd & Guild, Inc., 14 Crow Canyon Court, Ste. #200, San Ramon, CA 94583.

*Congratulations!*  
*to the*  
*Toronto Society for Coatings Technology*  
*on their 75th Anniversary*  
*November 19, 1994*

### Training Course in Coatings Technology Underway

The Joint Educational Committee of The New York Society for Coatings Technology and the Metropolitan New York Paint and Coatings Association is sponsoring a training course on "Understanding the Basics of Coatings I." This course, which is currently underway, is designed for persons without previous courses in Chemistry, as well as those technologists with some prior training.

The topics which are being studied include basic raw materials employed in the coatings industry, paint formulations and calculations, dispersion techniques, application equipment, and test methods.

For more information contact Mildred Leonard, NYSCT Office, Rm. 208, 520 Westfield Ave., Elizabeth, NJ 07208.

### ISPAC-8 Slated for May 1995

The Eighth International Symposium on Polymer Analysis and Characterization (ISPAC-8) will be held May 22-24, 1995 in Sanibel Island, FL.

ISPAC-8 will focus on polymer separations, spectroscopy, scattering techniques, microscopy, and copolymer characterization. In addition, a short course on polymer analysis and characterization methodologies will be held on May 21.

For more information, contact ISPAC Registration, 815 Don Gaspar, Sante Fe, NM 87501.

### Montreal Society To Host Mini-Symposium in February

The Montreal Society for Coatings Technology will host a mini-symposium entitled "Chemistry for the Changing Times" on February 2, 1995.

Tentative topics to be discussed include: "Urethane Technology Waterborne/UV"—speaker to be determined;

"Waterborne and High-Solids Polyols"—Steve Knight, of King Industries;

"Low Formaldehyde Release U.F. Resins"—Ralph Stange, of BASF Corp.; and

"Powder Coatings"—Joe Knauer, of Protec.

Additional information may be obtained from Alain Charbonneau, Miles Canada Inc., 7600 Trans-Canada Hwy., Pointe-Claire, Que., H9R 1C8.

### Chicago Society for Coatings Technology

in conjunction with

### Chicago Paint and Coatings Association

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Beginning January 10, 1995

DePaul University

For more information, contact Alison Azar, Seegott Inc.,  
1675D Holmes Rd., Elgin, IL; (708) 468-0400

# Literature

## Color Measurement

A hand-held spectral analyzer for color quality control is detailed in a press release. This instrument, which is designed for gathering, saving, and downloading data, is currently being offered at a special introductory price. ColorTec™, P.O. Box 386, 74 Main St., Lebanon, NJ 08833, can be contacted for further details on the ColorTec-PCM™ personal colorimeter.

Circle No. 30 on Reader Service Card

## Color Formulation

A color formulation software system for the plastics, inks, paints and coatings, and textile industries is available. This system allows the user to switch from formulating colors with white to formulating without white without switching to another database. For further information on the ProPalette, contact Macbeth, 405 Little Britain Rd., New Windsor, NY 12553-6148.

Circle No. 31 on Reader Service Card

## Stencil Ink

A product release describes a non-flammable water-based industrial stenciling ink. The dye-type black ink is formulated to be environmentally friendly and contains no heavy metals in the ink or its packaging. Diagraph Corp., 3401 Rider Trail South, St. Louis/Earth City, MO 63045-1110, can provide more details on RW-1 black stencil ink.

Circle No. 32 on Reader Service Card

## Interior Enamel

An interior architectural alkyd enamel which meets current VOC emissions standards is highlighted in literature. Reported characteristics include durability, flow and leveling, and hiding qualities. Further details on Craft Kote Alkyd Semi-Gloss are obtainable from Duron Paints & Wallcoverings, 10406 Tucker St., Beltsville, MD 20705.

Circle No. 33 on Reader Service Card

## Atomic Spectroscopy

A peer-reviewed journal featuring applications and research papers concerning atomic spectroscopy, atomic fluorescence, atomic emission, and ICP-MS is available. Subscription and submission information for *Atomic Spectroscopy* is available from The Perkin-Elmer Corp., 761 Main Ave., Norwalk, CT 06859-0105.

Circle No. 34 on Reader Service Card

## Bonding Primer

An adhesion-promoting, low VOC, waterborne, acrylic bonding primer for use over pre-finished metal siding and other slick surfaces is introduced in literature. The primer is designed to be rust and moisture resistant. Request DTM Bonding Primer (SWS-3906) from The Sherwin-Williams Co., c/o HKM Direct, 5501 Cass Ave., Cleveland, OH 44102, for more details.

Circle No. 35 on Reader Service Card

## Ceramic Pumps

A brochure provides information on magnetic drive ceramic pumps. Data is provided on performance curves, head/capacity and pressure/temperature limits, corrosion and erosion resistance statistics, and construction and materials. Copies of the brochure on EZ Series pumps can be obtained from QVF Process Systems Inc., 35 W. William St., Corning, NY 14830.

Circle No. 36 on Reader Service Card

## Pail Inserts

Literature introduces new contoured-lip seamless inserts for five gallon plastic pails. These inserts, which are vacuum-formed in either low or high density polyethylene, are nested for easy installation on high speed production lines. Free samples and more information on the inserts for plastic pails are available from CDF Corp., 100 Enterprise Dr., Marshfield, MA 02050.

Circle No. 37 on Reader Service Card

## Fibre Drum Disposal

A press release discusses a new global information network to provide fibre drum users with post-use disposition, environmental, and regulatory information. For more information on this service, contact General Manager, Recycling Operations, Sonoco Products Co., 1850 Parkway Pl., Marietta, GA 30067.

Circle No. 38 on Reader Service Card

## Zinc Sulfide Pigments

A book highlighting zinc sulfide pigments has been printed. The volume covers fundamentals of pigment optics, applications, environmental issues, contemporary problems concerning toxicology, and test methods. For more information on *Zinc Sulfide Pigments*, contact Vincentz Verlag, Postfach 62 47, D-30062 Hannover, Germany.

Circle No. 39 on Reader Service Card

## Paint Finishing System

A multi-color, two-step paint finishing system is the topic of literature. This system consists of an odorless, water-based acrylic latex base and finishing coat followed by a flecked finish coat providing tiny speckles of color. Information on the Aquafleck® system is available from Mercury Paint, 14300 Schaefer Hwy., Detroit, MI 48227.

Circle No. 40 on Reader Service Card

## Tetrol

A press release describes an environmentally and hygienically friendly tetrol which forms ester bonds with a high hydrolytic stability. The polyol is designed to improve gloss and flexibility in polyesters, and applications include radiation cured coatings, inks, and adhesives. Technical reports are available from Perstorp Polyols, S-284 80 Perstorp, Sweden.

Circle No. 41 on Reader Service Card

## Color Control

Literature introduces a high-end benchtop spectrophotometer for color quality control of plastics. The instrument features flat holographic grating and SPD array, diffuse transmittance, and pulsed xenon illumination. More information on the CM-3700d benchtop spectrophotometer is available from Minolta Corp., 101 Williams Dr., Ramsey, NJ 07446-1293.

Circle No. 42 on Reader Service Card

## Liquid Particle Counters

A new line of liquid particle counters, reportedly capable of providing particle measurement in ultra-pure and/or aggressive fluids and high viscosity liquids, is the topic of literature. Off-line batch measurement is possible, and six channels of data can be displayed in real time. For additional details, contact Horiba Instruments Inc., 17671 Armstrong Ave., Irvine, CA 92714.

Circle No. 43 on Reader Service Card

## Polyurethane Coating

Literature highlights a polyurethane coating used for interior automotive applications, including air bag facia covers. This low-gloss, low-reflection coating reportedly demonstrates color retention and durability after severe ultraviolet exposure. For additional details on Durasoft®, contact Akzo Nobel Coatings Inc., P.O. Box 7062, Troy, MI 48007-7062.

Circle No. 44 on Reader Service Card

## Synthetic Resin

Literature highlights a cyclohexanone-aldehyde resin designed to improve gloss, hardness, and adhesion in coatings. Applications include clear and pigmented wood lacquers, quick-dry sanding sealers, nitro-cellulose adhesives, and printing inks. Additional information on synthetic resin CA is available from Hüls America Inc., Colorants & Additives, P.O. Box 456, 80 Centennial Ave., Piscataway, NJ 08855-0456.

Circle No. 45 on Reader Service Card

## Defoamer

A silicone-free defoamer for water-based formulations is highlighted in a data sheet. This organic defoamer is designed to provide rapid initial and sustained defoaming in water-based formulations such as inks, coatings, and adhesives. Air Products and Chemicals, Inc., 7201 Hamilton Blvd., Allentown, PA 18195-1501, can be contacted for a copy of Publication No. 120-9426, "Surfynol<sup>®</sup> DF-210 Defoamer."

Circle No. 46 on Reader Service Card

## Performance Additives

Technical literature has been issued describing performance additives for powder coatings. This brochure outlines mechanical, optical, and surface properties attainable through the use of these additives. A copy of "Technical Data for Coatings: Properties of Performance Additives for the Powder Coating Industry" can be obtained from AlliedSignal Inc., Performance Additives, 101 Columbia Rd., Morristown, NJ 07962-1039.

Circle No. 47 on Reader Service Card

## Cleaning System

The introduction of a high-pressure cleaning system has been made through technical literature. This system pneumatically evacuates all solvent lines after each washing cycle to decrease VOCs and to ensure that there are no solvent droplets left to drip. For complete technical details on the SP50/SP80 Tote Tank, write Disti-Kleen, Inc., 22 Hudson Pl., Hoboken, NJ 07030-5512.

Circle No. 48 on Reader Service Card

## Durability Testing

A 280-page hardcover book discusses accelerated testing and proper use of results. Included are 15 peer-reviewed papers covering research in characterization of exposure tests and new developments in exposure equipment and exposure test design. ASTM, 1916 Race St., Philadelphia, PA 19103-1187, can provide more details on *STP 1202: Accelerated and Outdoor Durability Testing of Organic Materials*.

Circle No. 49 on Reader Service Card

## Continuous Processing


A continuous processing system that allows for a variable mixture of three or more components is detailed in literature. This system consists of two precision rotor/stator mills and three positive displacement pumps with variable frequency drive. Information on the multiple component continuous processing system is available from Kady International, 127 Pleasant Hill Rd., P.O. Box 847, Scarborough, ME 04070-0847.

Circle No. 50 on Reader Service Card

## Viscometer

A new process viscometer for the paint, ink, and coatings industries, with optional integral temperature measurement and intrinsic safety package, has been introduced in a press release. The sensor has no moving parts, facilitating cleanup and maintenance, and the instrument comes with a flow through mounting cell. For further details on the Model 300 PIC, write Nametec Co., 101 Forrest St., Metuchen, NJ 08840.

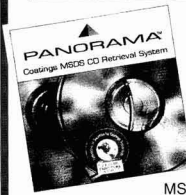
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## Flattening Agent

Information on a flattening agent which can be post-added or "stirred in" faster has been released. Reported features include reduced batch times, minimized foam formation, low dusting, film clarity, enhanced burnishing resistance, and clean production. Contact Grace Davison, W.R. Grace & Co. Conn., P.O. Box 2117, Baltimore, MD 21203-2117, for more information on Syloid® W 500.

Circle No. 52 on Reader Service Card

## Adhesion Promoter

A data sheet highlights an aminofunctional metal organic adhesion promoter with a pH of 8.5, enabling the product to be added directly to latex and polymer dispersions having a pH of seven. This adhesion promoter is designed to enhance adhesion of aqueous industrial coatings to metal as well as plastics, rubber, concrete, wood, and ceramics. Request information on Chartwell B-515.4 from Chartwell International, Inc., 32 Condor Rd., Sharon, MA 02067.

Circle No. 53 on Reader Service Card

## Weathering Tester

A product release presents a redesigned accelerated weathering tester which can perform both standard testing and high irradiance exposures. Features include microprocessor controls, automatic fault finding, and precision irradiance control. For further details on the QUV accelerated weathering tester, contact The Q-Panel Co., 26200 First St., Cleveland, OH 44145.

Circle No. 54 on Reader Service Card

## Glycol Diether

A product release introduces a specialty propylene oxide-based glycol diether. This aprotic solvent is designed to be used in proton-sensitive systems such as polyurethane coatings, is not considered a hazardous air pollutant, and has a high active solvency. Additional details on Proglyde™ DMM are obtainable from The Dow Chemical Co., Dow Corporate Communications, Midland, MI 48674.

Circle No. 55 on Reader Service Card

## LC/GPC Detection

A product release introduces a liquid/gel permeation chromatography detector for use with compounds that do not have high absorptivity in the ultraviolet range. Features include internal temperature control of the flow cell, offset adjustment, autozero, and autopurge of the reference cell. More information on the Series 200 refractive index detector is available from The Perkin-Elmer Corp., 761 Main Ave., Norwalk, CT 06859-0012.

Circle No. 56 on Reader Service Card

## Product Catalog

A full-color catalog contains product selection guides and information on product groups. Defoamers, deaerators, flow control additives, mar/slip agents, pigment wetting agents, and substrate wetting agents are described. Requests for a copy of the product catalog can be directed to Tego Chemie Service USA, 914 E. Randolph Rd., Hopewell, VA 23860.

Circle No. 57 on Reader Service Card

## Aluminum Pigments

A company highlights its line of aluminum pigments in a press release. One pigment mentioned offers small particle sizes with a whitish, brilliant, metallic shade; all pigments in this line are engineered to combine a light, bright, silky shade with limited color change when viewed from different angles. Information on and samples of Metallux 9000 are available from Obron Atlantic Corp., P.O. Box 747, Painesville, OH 44077.

Circle No. 58 on Reader Service Card

## Base Sheet

A base sheet to eliminate seamlines on surface printed wallcoverings with dark grounds is detailed in literature. This premium gray strippable base sheet is designed to help achieve maximum color consistency when installing wallcoverings. Further details on the dark base sheet are obtainable from Eisenhart Wallcoverings Co., P.O. Box 464, 1649 Broadway, Hanover, PA 17331-0464.

Circle No. 59 on Reader Service Card

## Deep Colored Bases

Deep colored bases in an interior/exterior premium quality paint are described in a product release. These are available in either a latex semi-gloss or an alkyd gloss, and are engineered to provide color depth and quality, hiding, and durability. Inquiries about deep colored bases are being addressed by Duron Paints & Wallcoverings, 10406 Tucker St., Beltsville, MD 20705.

Circle No. 60 on Reader Service Card

## Lead Regulation

A new manual explains and offers practical approaches for complying with OSHA's Interim Final Lead in Construction Standard. Topics covered include exposure monitoring, engineering and work practice controls, and selection and use of protective clothing and equipment. Contact the Painting and Decorating Contractors of America, 3913 Old Lee Hwy., Ste. 33-B, Fairfax, VA 22030, for information on the "PDCA Compliance Manual for OSHA Lead in Construction Regulation."

Circle No. 61 on Reader Service Card

# Why Renew?

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- A 1-year subscription to the **JOURNAL OF COATINGS TECHNOLOGY**, the technical publication of the coatings industry, is included with your membership.
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# Coming Events

## FEDERATION MEETINGS

For information on FSCT meetings, contact Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422 (610) 940-0777, FAX: (610) 940-0292.

### 1995

(May 17-21)—FSCT Spring Week. Spring Seminar on the 17th and 18th; Board of Directors Meeting on the 20th; Incoming Society Officers Meeting on the 21st. Fiesta Americana, Cancun, Mexico.

(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.

### 1997

(Nov. 5-7)—75th Annual Meeting and 62nd Paint Industries' Show. Georgia World Congress Center, Atlanta, GA.

## SPECIAL SOCIETY MEETINGS

### 1995

(Feb. 2)—"Chemistry for the Changing Times." Mini-symposium sponsored by the Montreal Society for Coatings Technology. (Alain Charbonneau, Miles Canada Inc., 7600 Trans-Canada Hwy., Pointe-Claire, Que., Canada H9R 1C8).

(Feb. 20-22)—Western Coatings Societies' 22nd Biennial Symposium and Show. Sponsored by the Golden Gate, Los Angeles, Pacific Northwest, and Rocky Mountain Societies. Hilton Hotel and Towers, San Francisco, CA. (Gordon Ploch, WCSSS Chairman, Eureka Chemical Co., P.O. Box 2205, S. San Francisco, CA 94083; (415) 761-3536).

(Feb. 22-24)—22nd Annual Waterborne, Higher-Solids, and Powder Coatings Symposium. Sponsored by the Southern Society and The University of 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. 15)—"Clean Air & Technology." Mini-Trade Show and Symposium sponsored by the Piedmont Society for Coatings Technology. Showcase on the Park, High Point, NC. (Richard Chodnicki, Van Horn, Metz & Co., 320 Towsontown Blvd., Ste. 205, Baltimore, MD 21204).

(Apr. 19-21)—Southern Society Annual Meeting. Hyatt Regency, Savannah, GA. (Wayne West, Thompson & Formby, Inc., 10136 Magnolia Dr., Olive Branch, MS 38654).

(May 3-4)—"Recent Advances in Modifiers for Modern Coatings." Symposium sponsored by the New York Society for Coatings Technology. Holiday Inn North, Newark Airport, Newark, NJ. (Larry Waelde, Troy Chemical Corp., One Ave. L, Newark, NJ 07105).

(May 4-6)—48th Annual Spring Symposium. Sponsored by the Pacific Northwest Society. Red Lion Lloyd Center, Portland, OR. (Ken Wenzel, Chemical Distributors, Inc., P.O. Box 10763, Portland, OR 97210; (503) 243-1082).

## OTHER ORGANIZATIONS

### 1994

#### North America

(Dec. 1-2)—1994 Southwest University/Industry Technology Transfer Conference. Sponsored by Technology Transfer Conferences, Inc. Dallas Airport Marriott Hotel, Dallas/Ft. Worth International Airport. (Lucy W. Malone, Technology Transfer Conferences, Inc., 325 Plus Park Blvd, #108, Nashville, TN 37217).

#### Europe

(Nov. 22-23)—"Forum de la Connaissance." Symposium sponsored by the French Association of Technicians of Paints (AFTPVA) in conjunction with The Coatings Research Institute (CoRI). Auditorium Hoechst-Tower Rousset, Hoechst-Paris-La Défense. (AFTPVA, 5, rue Etex, F-75018 Paris, France or CoRI, Avenue P. Holfloffe, B-1342 Limelette, Belgium).

(Dec. 6-7)—"Styrenics '94." Conference sponsored by Maack Business Services. Swissôtel, Zürich, Switzerland. (Maack Business Services, Moosacherstrasse 14, CH-8804 AU/Zürich, Switzerland).

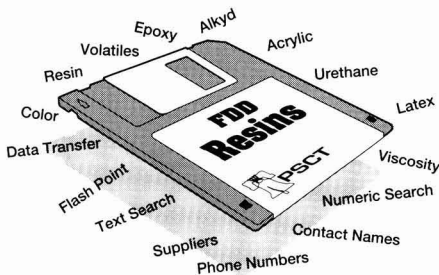


### 1995

#### North America

(Jan. 14-18)—RCMA 1995 Annual Conference & EXPO. Sponsored by the Roof Coatings Manufacturers Association (RCMA). The

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Ritz-Carlton Laguna Niguel, Dana Point, CA. (Sally Choquette, RCMA Meetings Coordinator, RCMA, 6000 Executive Blvd., Ste. 201, Rockville, MD 20852-3803).

(Jan. 22-25)—Meeting of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications. Hyatt Regency Hotel, San Antonio, TX. (ASTM, 1916 Race St., Philadelphia, PA 19103-1187).

(Feb. 1-3)—"Bridging the Environment." The Fourth World Congress on Coating Systems for Bridges and Steel Structures. Sponsored by the University of Missouri—Rolla (UMR). Marriott Airport Hotel, St. Louis, MO. (Michael R. Van De Mark or Norma Fleming, 119 ME Annex, UMR, Rolla, MO 65401).

(Feb. 19-22)—18th Annual Meeting of The Adhesion Society. Crystal Sands Resort, Hilton Head, SC. (Joseph W. Holubka, Ford Motor Co., MD 3198, P.O. Box 2053, Dearborn, MI 48121).

(Feb. 24-26)—"Spring Decor 1995." Sponsored by the National Decorating Products Association (NDPA). Georgia International Convention Center, Atlanta, GA. (Teri Flotron, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

STATEMENT OF OWNERSHIP, MANAGEMENT AND CIRCULATION  
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B. Paid Circulation		
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2. Mail Subscriptions	9,120	9,087
C. Total Paid Circulation	9,120	9,087
D. Free Distribution by Mail, Carrier, or Other Means, Samples, Complimentary, and Other Free Copies	287	302
E. Total Distribution	9,407	9,389
F. Copies Not Distributed		
1. Office Use, Left Over, Unaccounted, Spoiled After Printing	217	200
2. Returns From News Agents	0	0
G. Total	9,624	9,589

I certify that the statements made by me are correct and complete. (signed)  
Patricia D. Viola, Editor

(Mar. 26-31)—"Corrosion '95." Annual Conference sponsored by National Association of Corrosion Engineers (NACE) International. Orlando, FL. (NACE International, P.O. Box 218340, Houston, TX 77218-8340).

(Apr. 2-7)—38th Annual Technical Conference sponsored by Society of Vacuum Coaters (SVC). Chicago Marriott Downtown, Chicago, IL. (SVC, 440 Live Oak Loop NE, Albuquerque, NM 87122-1407).

(Apr. 5-7)—10th Annual Conference sponsored by Architectural Spray Coaters Association (ASCA). Doral Ocean Beach Resort, Miami, FL. (ASCA, 230 W. Wells St., Ste. 311, Milwaukee, WI 53203).

(May 22-24)—Eighth International Symposium on Polymer Analysis and Characterization (ISPAC-8). Sanibel Island, FL. (ISPAC Registration, 815 Don Gaspar, Sante Fe, NM 87501).

(June 5-9)—"Advances in Emulsion Polymerization and Latex Technology." Short course sponsored by Lehigh University, Bethlehem, PA. (Mohamed S. El-Aasser, Emulsion Polymers Institute, Lehigh University, 111 Research Dr., Bethlehem, PA 18015).

(June 25-29)—"Coating Work in Nuclear Facilities." Symposium sponsored by The National Board of Registration for Nuclear Safety Related Coating Engineers and Specialists. Marriott Hotel, Philadelphia, PA. (Dean M. Berger, Executive Secretary, NBR, P.O. Box 56, Leola, PA 17540).

(Sept. 29-Oct. 1)—"Fall Decor 1995" Sponsored by the National Decorating Products Association (NDPA). Atlanta, GA. (Teri Flotron, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

Asia

(Jan. 16-18)—"Paintindia '95." Nehru Centre, Bombay, India. (R. Rajagopal, Colour Publications Pvt. Ltd., 126-A, Dhuruwadi, A.V. Nagwekar Marg, Prabhadevi, Bombay 400 025 India).

Europe

(Jan. 2-6)—"Organic Coatings." 53rd International Meeting of Physical Chemistry. Sponsored by the Division de Chimie Physique of Soc. Française de Chimie. Ministry of Research, Paris, France. (Dr. Troyanowsky, Div. de Chimie Physique, Laboratoire de Chimie Physique, 11, rue Pierre er Marie Curie, 75005, Paris, France).

(Mar. 14-16)—"European Coatings Show '95." Congress and Exhibition. Exhibition Centre, Nuremberg, Germany. (Vincentz Verlag, Postfach 62 47, D-30062, Hannover, Germany).

(May 15-18)—"Recycle '95." Forum and Exposition. Sponsored by Maack Business Services. Swissôtel, Zürich, Switzerland. (Maack Business Services, Moosacherstrasse 14, CH-8804 AU/Zürich, Switzerland).

(May 23-24)—First Pacific Coating Forum. Sponsored by the Japan Coating Technology Association. Shonan Kokusai Village, Kanagawa, Japan. (Secretariat, Japan Coating Technology Association, Daiichi Naka-Bldg. 4F, 3-4 Nihombashi-Kobunacho, Chuouku, Tokyo 103, Japan).

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## 'Humbug' from Hillman

The Phantom has struck in Vermont. His/her messages arrived via RFD too early to be appropriate for Halloween and too late to match a Halloween publication. Nonetheless, a selection of the Phantom's messages will serve this month's Humbug with a gamut of reactions. Certainly, a fortune cookie manufacturer would be well served.

—Nothing is impossible to the man who doesn't have to do it.

- I don't get headaches, I'm just a carrier!
- Talking non-stop is not communicating.
- The largest room in the world is room for improvement.
- The cicada is a noisy red-eyed pest, too, but at least it only comes once every 17 years.
- From listening comes wisdom and from speaking repentance.

And, also listed were many quotes from "Rabinow's Laws" (who's he?).

Here is enough of Rab's wisdom (?) to get the idea:

- Everyone wants improvements without any changes.
- Everyone knows that I should have built the second model first.
- If the boss is a dope, everyone under him is (or soon will be) a dope.
- In judging a manager, the opinion of his/her subordinates is more important than that of his/her superiors.
- When you have enough money to tell the boss to shove it, you never have to do it.
- The most efficient conferences are held in corridors.
- If you want a 50-50 deal, offer the other party 60 and ask for 40.

Sign in neurologist Dr. Keith Edwards' office:

"If the right side of the brain controls the left side of the body, then only left-handed people are in their right minds."

Note—Dr. Edwards plays cards left handed.

Jeff Sturm, again, has supplied us with a copy of the "Yetter Letter," which always serves Humbug with a giggle or two or more.

—The sailor, home on leave and celebrating the occasion, had gotten himself in a pickle. He had hired a taxi, only to discover when approaching his destination that he was penniless. He used his Navy resourcefulness, shouting to the driver, "Stop!" and jumped out.

"I want to run into this drugstore and get some matches," he explained to the driver. "I've dropped a \$100 dollar bill somewhere in the cab and I can't find it in the dark."

He went into the store and watched as the cab and driver vanished into the night.

In a perfect world . . .

- Your raise would always be far more than you expected.
- Neither chocolate nor booze would contain calories.
- Procrastination would be a virtue.
- Teenagers would rather work on the lawn than talk on the telephone.
- What the cat dragged in would be \$100 dollar bills.
- Children on trips would say, "Isn't riding in the car fun!" and then they'd fall asleep.

And Jeff's favorite: On Being a Stud—

Two county road commissioners died and appeared at the pearly gates. St. Peter acknowledged they were at the right place, but space was temporarily short. Each was given a chance to go back to earth and await a new summons. While waiting, they could be anything they chose, except themselves as they had been in life.

Commissioner No. 1 said he would like to return as an eagle, fly over his district and admire the roads, bridges, and lakes. "Done," said St. Peter, and the temporary eagle flew off.

No. 2 preferred a different lifestyle. "I want to go back as a stud."

No problem. Wish granted.

Three weeks later, the new heavenly subdivision was opened, and St. Peter sent for the two commissioners. The angel asked, "How am I going to find them?"

St. Peter said, "Charlies easy. He's an eagle. You'll find him soaring over the Mississippi Valley. Fred's a little harder. He's nailed between two wallboards at a construction site in Kansas City."

From friend Larry Hill's fun column in *Surface Coatings Australia*:

Here are a few schoolboy howlers:

- J.S. Bach wrote a lot of music and had a lot of children. In between, he practiced on an old spinster he kept in the attic.
- Blood vessels are of three kinds: arteries, veins, and caterpillars.
- The supernatural is about ghosts, goblins, and virgins, and other mystical creatures.
- Hindus wear turbines on their heads.
- The American War of Independence was caused by the English putting tacks in their tea.
- To be a good nurse, you must be absolutely sterile.
- People who live in Moscow are called Mosquitos.
- A man named Peeping Tom looked at Lady Godiva without any clothes on—this was the start of Neighborhood Watch."

Apparently, the kids Down Under aren't much smarter than ours.

Have you heard about the man who's sorry he ever decided to install a car phone?

He finds it such a nuisance to run to the garage every time the phone rings.

"And now, gentlemen," continued the congressman, "I wish to tax your memory."

"Good heavens," muttered a colleague, "why haven't we thought of that before?"

—The Lion

—Herb Hillman  
Humbug's Nest  
P.O. Box 135  
Whitingham, VT 05361



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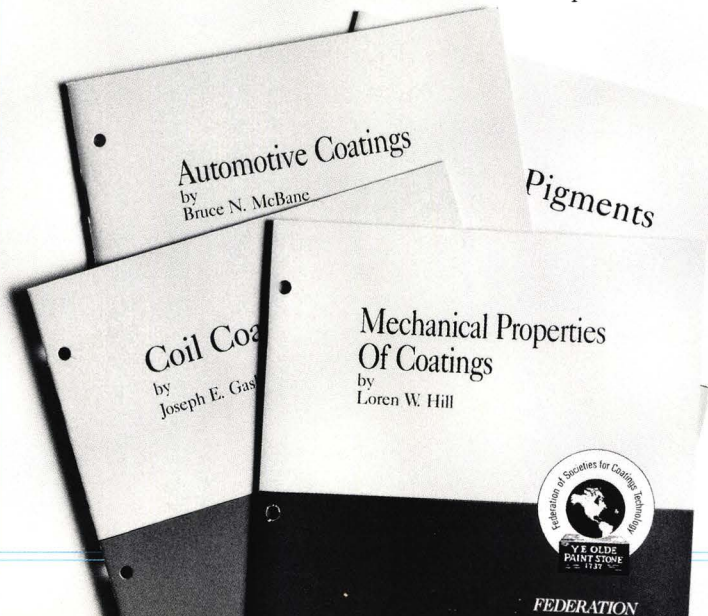
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