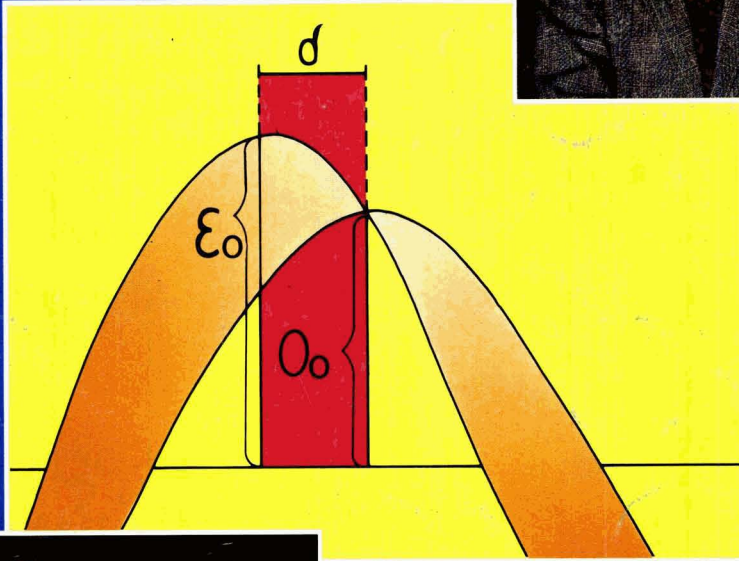
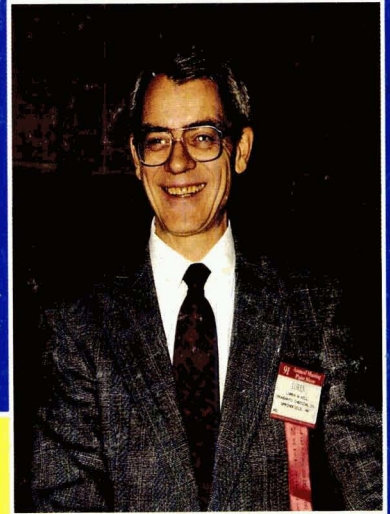


# jct JOURNAL OF COATINGS TECHNOLOGY

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MEMORIAL LECTURE



Structure/  
Property  
Relationships  
of Thermoset  
Coatings





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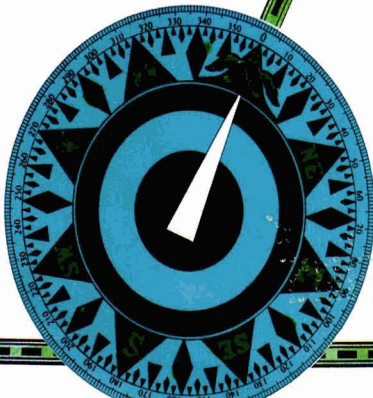
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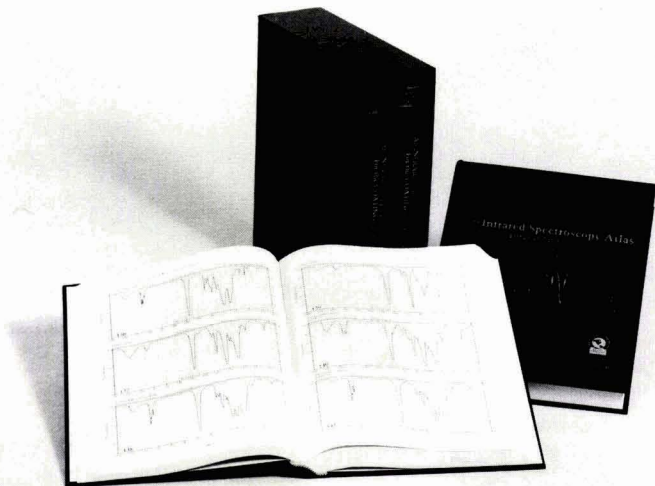
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The JOURNAL OF COATINGS TECHNOLOGY is published monthly by the Federation of Societies for Coatings Technology for its membership of approximately 7,300 in 26 Constituent Societies in the United States, Canada, Great Britain, and Mexico. The JOURNAL is devoted to the advancement of knowledge in the science and technology of surface coatings, the materials comprising such coatings, and their use and performance.

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 All manuscripts will be assumed to be previously unpublished writing of the authors, not under consideration for publication elsewhere.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

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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).

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*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.

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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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## Changing of the Guard

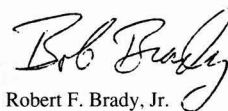
I'm sure many of you can identify with the mixture of pleasure and anxiety that comes with a promotion or an assignment to an important new task. This is how I felt when I was asked to become Technical Editor of the JOURNAL OF COATINGS TECHNOLOGY. It is an honor to be asked to take on this responsibility and a pleasure to become more closely associated with Pat Viola and her staff, who create such an attractive and readable publication.

However, it will be a challenge to maintain the quality and reputation of the technical section of the Journal. Much of this is owed to Tom Miranda, who has served as Technical Editor for the past 19 years. During this time the JCT has preserved and enlarged its reputation as the most important publication in coatings science and technology in the world. I would like to express the gratitude of all of us who know Tom and have worked with him for his unstinting service to the Journal and to the Federation as Chairman of the Publications Committee. Tom leaves me a rock-solid foundation on which to build.

Perhaps I should write a few words about myself. I've been actively involved in the coatings industry for 17 years. The first seven were spent at the General Services Administration where I supervised the people who wrote the federal specifications and test methods for coatings, brushes, and related products. If you've ever used a federal specification written between 1975 and 1982, or performed any of the tests in Federal Test Method Standard 141, I'm the one to blame!

For the past 10 years I've directed a small team of people who formulate and test coatings for the U.S. Navy. We've specialized in nonskid coatings for aircraft carrier decks and linings for fuel storage tanks, to name a few, and our products are in use on Navy ships and shore facilities all over the world.

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Robert F. Brady, Jr.  
Technical Editor



# Abstracts of Papers in This Issue

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

## Structure/Property Relationships of Thermoset Coatings—L.W. Hill

Journal of Coatings Technology, 64, No. 808, 28 (May 1992)

More than 50 years ago, Joseph J. Mattiello was a leader in the use of scientific principles to understand and improve decorative and protective coatings (see *Appendix 1*). In Mattiello's spirit of scientific innovation, progress is reviewed in establishment of relationships between structures of starting materials and physical properties of cured thermoset films. The effectiveness of coatings scientists to adopt and adapt new concepts for use in coatings research is discussed. Under-utilized and remarkably simple methods for calculating crosslink density (XLD) of stoichiometric networks are presented. Use of dynamic mechanical analysis (DMA) and solvent induced swelling for determination of XLD is described. DMA plots are shown for many types of coatings.

## Water-Reducible, Unsaturated Polyesters as Binders and Clear Coatings for UV-Curable Furniture Coatings—M.J. Dvorchak, and B.M. Riberi

Journal of Coatings Technology, 64, No. 808, 43 (May 1992)

The paper describes the development and properties of water-reducible, unsaturated polyesters which can be used to formulate UV-curable furniture coatings.

The development of water-reducible, unsaturated polyesters had to overcome two problems. First, the monomer copolymerizable with the polyester, usually styrene, cannot be used. Therefore, a system needed to be developed that produced coatings with adequate properties even in the absence of copolymerizable monomers and that produced stable coatings systems with good chemical and water-resistant properties even though it was water reducible.

The incorporation of polyols which had allyl ether groups allowed the formulation of unsaturated polyesters that do not require styrene to cure into a film with good properties. The further incorporation of special nonionic emulsifiers into the polymer backbone made the unsaturated polyester water reducible.

Coatings formulations based on this water-reducible, unsaturated polyester have attained VOCs as low as 0.2 lbs/gal without using monomer diluents or amine solubilizing agents. These formulations can be cured by a cobalt peroxide mechanism. UV-curing allows for faster curing. The curing condi-

## Relations Structure/Propriete des Revêtements Thermodurcissables—L.W. Hill

Journal of Coatings Technology, 64, No. 808, 28 (May 1992)

Il y a plus de cinquante ans, Joseph J. Mattiello était un pionnier dans l'utilisation des principes scientifiques visant à la compréhension et à l'amélioration des revêtements décoratifs et protecteurs (voir l'annexe 1). Dans l'esprit créateur scientifique de Mattiello, le progrès scientifique actuel est revu afin d'établir la relation entre les structures des matières premières et les propriétés physiques des feuillets thermodurcissables cuits. La facilité avec laquelle les scientifiques du domaine des revêtements s'adaptent aux nouveaux concepts de la recherche est discutée. Des méthodes simples pour le calcul du degré de réticulation des réseaux stoechiométriques sont présentées. L'utilisation de techniques telles que l'analyse mécanique dynamique ainsi que le gonflement induit par un solvant dans la détermination du degré de réticulation est décrite. Des diagrammes obtenus par la technique de l'analyse mécanique dynamique sont démontrés pour différents genres de revêtements.

## Polyesters Insaturés Hydrosolubles Comme Liants et Revêtements Transparents pour les Levêtements Durcissant aux Rayons Ultra-Violetes—M. Dvorchak and B.H. Riberi

Journal of Coatings Technology, 64, No. 808, 43 (May 1992)

Cette publication décrit le développement et les propriétés des polyesters insaturés hydrosolubles pouvant être utilisés dans la formulation des revêtements durcissant aux rayons ultraviolets.

Le développement des polyesters insaturés hydrosolubles ont dû faire face à deux problèmes. Premièrement, le monomère copolymérisable avec le polyester, habituellement le styrène, ne peut être utilisé. Donc, il a fallu produire des revêtements copolymérisables et qui produisent des revêtements stables avec des résistances aux produits chimiques et à l'eau, même si ces derniers sont diluables à l'eau.

L'addition de polyols contenant des groupes éthers a permis la formulation de polyesters insaturés ne requérant pas de styrène pour le durcissement d'un feuil possédant de bonnes propriétés. L'incorporation subséquente d'émulsifiants non-ioniques a rendu le polyester insaturé hydrosoluble.

## Relaciones de Estructura/Propiedad de Recubrimientos Termofijos—L.W. Hill

Journal of Coatings Technology, 64, No. 808, 28 (May 1992)

Hace más de 50 años, Joseph J. Mattiello fué el pionero en el uso de los principios científicos para entender y mejorar los recubrimientos decorativos y de protección (ver apéndice I). En el espíritu de Mattiello por la innovación científica, el progreso se revisa para establecer relaciones entre las estructuras de las materias primas y las propiedades físicas de las películas curadas termofijas. Se mejora la efectividad de la ciencia de los recubrimientos para adoptar y adaptar nuevos conceptos para ser usados y discutidos dentro de la investigación. Bajo métodos simples ya utilizados se presentan calculos en la densidad de reticulación (XLD) y nuevos trabajos estequiométricos. Se utilizan el análisis mecánico dinámico (DMA) y la inducción por empaque de solvente para determinar la XLD. Se muestran los resultados DMA para varios tipos de recubrimientos.

## Poliesteres Insaturados Reducibles Con Agua, Como Ligantes y Recubrimientos de Limpieza Para Recubrimientos de Muebles Curados Por UV—M.J. Dvorchak and B.H. Riberi

Journal of Coatings Technology, 64, No. 808, 43 (May 1992)

El artículo describe el desarrollo y las propiedades de poliesteres insaturados reducibles con agua, los cuales se pueden usar para formular recubrimientos para muebles curados por UV.

El desarrollo de los poliesteres insaturados reducibles con agua tiene dos problemas. Primero, el monómero copolimerizable con el poliéster, usualmente estireno, no se puede usar. Sin embargo, un sistema necesita ser desarrollado para producir recubrimientos con las propiedades adecuadas aun se tenga la ausencia de monómeros copolimerizables, además de tener que ser sistemas de recubrimientos estables con buena resistencia química y al agua a pesar de que este sea reducible con agua.

La incorporación de polioles los cuales tienen grupos alilietar a lo largo de la formulación de los poliésteres insaturados no requiere el uso de estireno que cure dentro de la película proporcionandole buenas propiedades. Una seguida incorporación de emulsificantes especiales no-iónicos en el esqueleto de sistema proporciona la reducibilidad con agua a los poliésteres.

# NOBODY PUTS MORE SILICONE EXPERIENCE INTO EVERY BOTTLE.

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**DOW CORNING**





tions and properties of the resulting coatings are discussed. The proper selection of UV-photoinitiator makes it possible to UV-cure even pigmented systems.

#### **Fundamental Studies of Epoxy Resins for Can and Coil Coatings III. Effect of Bisphenol Structure on Resin and Coating Flexibility—R.A. Dubois and P.S. Sheih**

Journal of Coatings Technology, 64, No. 808, 51 (May 1992)

Many workers have reported improvements in coating flexibility by blending and grafting flexibilizers with Bisphenol A based epoxy coatings or by using aliphatic epoxies. These techniques are often limited by the rigidity of the Bisphenol A unit or by loss of other desirable properties. Systematic variation in the structure of the groups bridging the phenol units of diphenols is correlated with flexibility by the corresponding epoxy resins and ultimately with coating performance of the cured resins. This technique, combined with optimum curing conditions, can give coatings as flexible as aliphatic epoxies while retaining the adhesion, chemical resistance, and reactivity characteristic of Bisphenol A based epoxies.

#### **Strongly Adherent, Smooth Coatings of Polypyrrole Oxalate on Iron—F. Beck and R. Michaelis**

Journal of Coatings Technology, 64, No. 808, 59 (May 1992)

Black polypyrrole coatings on iron can be obtained through anodic electrodeposition from aqueous electrolytes, containing the monomer and oxalic acid. Adherence strength of the layers is larger than  $11.5 \text{ mm}^2$  for  $1 \mu\text{m}$  coatings. Surface roughness is found to be relatively low in comparison to other polypyrrole salts, even at thicker layers. The polymer films are essentially nonporous. A pronounced leveling action for the electrodeposition was found. Overcoating by cathodic or anodic electrodeposition of paint was demonstrated. The application of these polypyrrole coatings as corrosion inhibiting polymer interlayers, eventually substituting phosphatation, is proposed.

Les formulations de revêtements à base de polyesters insaturés hydrosolubles ont atteint des contenus en matières organiques volatiles aussi bas que  $0.2 \text{ lb/gal}$  sans utilisation de diluants monomériques ou d'agents solubilisant à base d'amines. Ces revêtements peuvent être réticulés par un peroxyde de cobalt. Le durcissement aux rayons ultraviolets permet un durcissement beaucoup plus rapide. Les conditions de durcissement et les propriétés des revêtements obtenus sont discutés. La sélection des photoinitiateurs a permis le durcissement aux rayons ultraviolets de revêtements contenant des pigments.

#### **Etudes Fondamentales des Resines Epoxydique pour les Revêtements en Continu III. Effet de la Structure du Biphenol sur la Resine et la Flexibilité du Revêtement—R.A. Dubois and P.S. Sheih**

Journal of Coatings Technology, 64, No. 808, 51 (May 1992)

Plusieurs chercheurs ont noté améliorations dans la flexibilité d'un revêtement en mélangeant et en réticulant des agents flexibles avec soit des revêtements époxy à base de biphénol A ou par l'utilisation d'époxy aliphatique. Ces techniques sont cependant limitées par la rigidité de l'unité biphénol A ou par la perte de diverses propriétés souhaitables. La variation de la structure des groupes liant les unités phénoliques des biphénols est corrélée avec la flexibilité des résines époxydiques correspondantes, ainsi qu'avec la performance des revêtements des résines durcies. Cette technique, combinée avec des conditions de durcissement optimales peut donner des revêtements aussi flexibles que les époxy aliphatiques tout en conservant l'adhésion, la résistance chimique ainsi que les caractéristiques de réactivité des époxy à base de biphénol A.

#### **Revêtements Lisses et Adhérents d'Oxalate de Poly-pyrrole sur un Subjectile Ferreux—F. Beck and R. Michaelis**

Journal of Coatings Technology, 64, No. 808, 59 (May 1992)

Les revêtements noirs à base de polypyrrole sur un subjectile ferreux peuvent être obtenus par électrodeposition anodique avec l'aide d'électrolytes aqueux contenant le monomère et de l'acide oxalique. La force d'adhésion est supérieure à  $11.5 \text{ N mm}^2$  pour un revêtement de  $1 \mu\text{m}$  d'épaisseur. La rugosité de la surface est relativement faible par rapport à d'autres sels de polypyrrole, même en couches plus épaisses. Les pellicules de polymères possèdent une porosité presque nulle. Un effet prononcé de nivellement par le procédé d'électrodeposition a été noté. Le recouvrement d'une peinture à partir du procédé d'électrodeposition cathodique ou anodique a été démontré. L'application de ces revêtements à base de polypyrrole par multicouches inhibitrices de corrosion, pouvant remplacer la phosphatation, est suggérée.

Las formulaciones de recubrimientos basadas en estos poliésteres, han proporcionado un valor de VOC tan bajo como  $0.2 \text{ lbs/gal}$  sin usar diluyentes de monómero o agentes solubilizantes aminicos. Estos sistemas se pueden curar mediante un mecanismo de peróxido de cobalto. Para un curado rápido se aplica UV. Se discuten las condiciones de curado y las propiedades resultantes del recubrimiento. La adecuada selección del fotoinicador UV hace posible el manejar sistemas pigmentados.

#### **Estudios Fundamentales de Resinas Epoxicas para Latas y Coil Coatings III. Efecto de la Estructura del Bisfenol en la Resina y la Flexibilidad del Recubrimiento—R.A. Dubois and P.S. Sheih**

Journal of Coatings Technology, 64, No. 808, 51 (May 1992)

Muchos trabajadores han reportado mejoras en la flexibilidad de un recubrimiento mediante la mezcla y acople de flexibilizadores con el Bisfenol A en la base de los recubrimientos epóxicos o usando epóxicos alifáticos. Estas técnicas son limitadas debido a la rigidez en la unidad del Bisfenol A o por la baja indeseable de otras propiedades. La variación sistemática en la estructura de los grupos de enlace en las unidades de fenol o difenol se correlacionan con la flexibilidad de la correspondiente resina epóxica y termina con la mejora de la calidad del recubrimiento de las resinas curadas. Esta técnica, combinada con las condiciones óptimas de curado, pueden proporcionar al recubrimiento la flexibilidad de un epóxico alifático mientras retiene las propiedades de adhesión, resistencia química y características de reactividad de los epóxicos con base de Bisfenol A.

#### **Poder Adherente, Recubrimientos Ligeros de Oxalato de Polipirrolidona Sobre Metal—F. Beck and R. Michaelis**

Journal of Coatings Technology, 64, No. 808, 59 (May 1992)

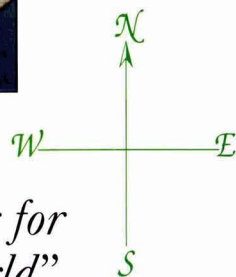
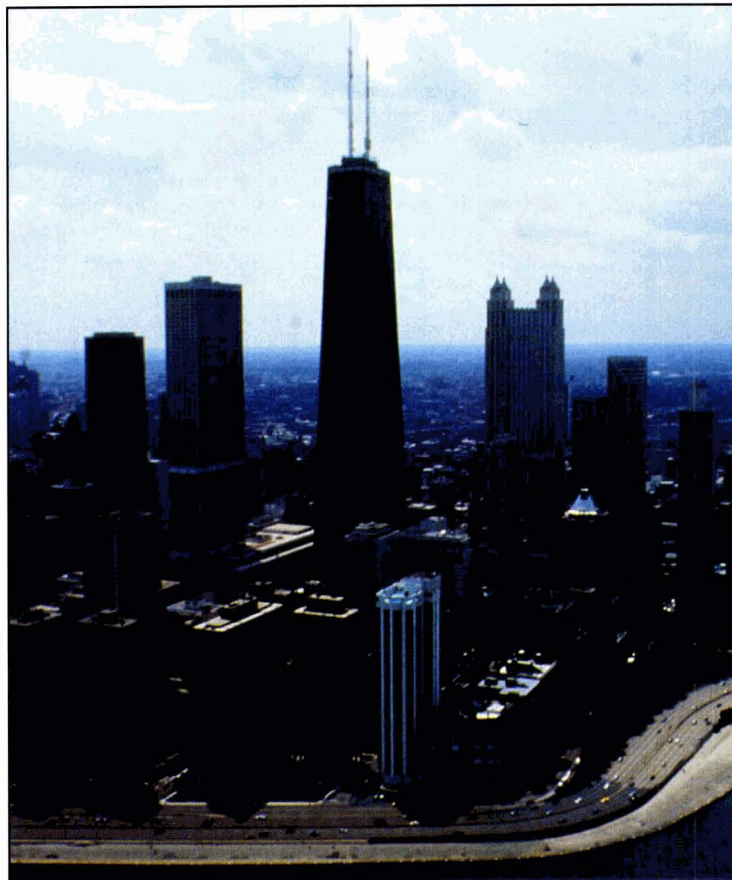
Se pueden obtener recubrimientos negros de polipirrolidona sobre metal mediante un electrodepósito anódico en electrolitos acuosos, conteniendo el monómero y el ácido oxálico. La fuerza de adherencia de las capas es más grande que  $11.5 \text{ N mm}^2$  para recubrimientos de  $1 \mu\text{m}$ . Se encontró que la dureza de la superficie es relativamente más baja en comparación a otras sales de polipirrolidona, aún en capas más delgadas. Las películas del polímero son esencialmente no porosas. Se encontró un alto nivel de acción en el electrodepósito. Se demostró un sobrecubrimiento tanto en los sistemas de electrodepósito anódico como catódico. Se propone que la aplicación de estos recubrimientos de polipirrolidona como inhibidores de corrosión en intercapas poliméricas, eventualmente pueden eliminar o sustituir la fosfatación.

Federation of Societies for Coatings Technology

# 1992 Annual Meeting & Paint Industries' Show

Hotel Information/Reservation Application

• Advance Registration Form •



*“New Directions for  
a Changing World”*

McCormick Place North

Wednesday, Thursday, Friday • October 21-22-23, 1992

Chicago, Illinois



*The combined Annual Meeting and Paint Industries' Show is a major educational activity of the Federation. This international coatings manufacturing industry event consists of three days of technical program sessions and exhibits, running concurrently. Registration is required for admission.*

### ***From the President:***

It's been four long years since the Federation has held its Annual Meeting and Paint Show in Chicago. The industry has changed greatly since 1988, especially in the areas of compliance with regulations and the technology needed to maintain and prosper in these difficult times.



As always, the mission of the FSCT is the technical advancement and education of both its membership and the industry. The highlight of our efforts, the Annual Meeting and Paint Show,

offers the best opportunity around to get the knowledge and insight to help your company—and to help yourself.

Please take a few moments to read over the following pages. I'm sure you will see many topics being presented in the Technical Program or suppliers participating in the Paint Show that will make the difference for you in 1992 and beyond.

Meanwhile, I look forward to seeing you in Chicago!

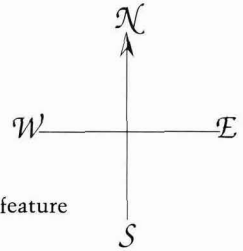
A handwritten signature in black ink that reads "W.F. Holmes". The signature is fluid and cursive.

William F. Holmes  
President, FSCT



Photo courtesy of Gene Hickmott/Chicago Convention & Tourism Bureau

**McCormick Place Complex—site of the Federation's 70th Annual Meeting and 57th Paint Industries' Show**



## ***“New Directions for a Changing World”***

Looking towards the future, the theme of this year’s Annual Meeting Program, “New Directions for a Changing World,” focuses on the need to change existing business strategies in formulating products, responding to regulatory and safety demands, and prudent management of resources. The “new directions” being addressed in presentations include adopting merging and emerging technologies to drive innovation, developing environmentally friendly products and processes, and embracing total quality systems to effectively compete in a global market.

Under development are the following program session themes:

- Advanced Topics
- Formulation Factors for the Design of Corrosion-Resistant Direct-to-Metal Coatings (DTM)
- Material Quality
- Employee Involvement—Overcoming the Obstacles to Empowerment
- Waste Minimization
- Merging/Emerging Technologies
- Roon Award Competition Papers

Additional topics will feature presentations on:

- Powder Coatings
- Pigments for High Performance Coatings
- Wood Finishing
- Recycling
- EB Curing Systems
- Waterborne Paint Systems
- Corrosion Inhibitors
- Low VOC Coatings
- Paint Filtration
- Epoxy Resins
- Adhesion Promoters

Highlighting the technical program will be the Mattiello Memorial Lecture by Dr. John L. Gardon, of AKZO Coatings, Inc., Detroit, MI. Dr. Gardon’s presentation will be made during the Friday morning session. The Keynote Address, scheduled for Wednesday morning, will be given by noted columnist, Jack Anderson.

A Poster Session, featuring non-commercial work in new ideas and techniques in coatings research will be held on Thursday in the exhibit hall.

## ***Record-Setting Paint Show to Feature Products of Over 260 Companies***

The largest Paint Show in Federation history—indeed, the largest coatings-related exhibition in the world—will be held in conjunction with the Annual Meeting in McCormick Place North. Over 260 supplier companies to the coatings manufacturing industry will be present to discuss their newest products and services. In over 90,000 sq. ft. of exhibits will be displayed a wide variety of raw materials, production equipment, containers and filling equipment, laboratory apparatus, and testing devices for the paint and coatings producer.

Key personnel from the top technical and sales staff will be on-hand to provide attendees with an opportunity to learn of the latest developments in their products and services. Listed elsewhere are the exhibitors who have reserved space in the Paint Industries’ Show.

Exhibit hours will be:

10:00 am-5:30 pm ..... Wednesday, October 21  
 9:00 am-5:30 pm ..... Thursday, October 22  
 9:00 am-12:00 Noon ..... Friday, October 23

## ***Jane Byrne, Former Mayor of Chicago, to Speak at Federation Luncheon***

The annual Federation Luncheon will be held on Friday, October 23, at McCormick Center Hotel. The featured speaker will be Jane Byrne, former Mayor of Chicago. Tickets for the luncheon may be

purchased for \$25 either in advance by filling in the appropriate information on the registration form or on-site at the Registration Area of McCormick Place North.

## Hotel Information

Whether you wish the value of a moderately priced hotel, or the luxury of an upscale property, the choice is yours. The FSCT has arranged for convention rates at seven official Paint Show hotels. All reservations must be placed through the Chicago Convention & Tourism Bureau's FSCT Housing Bureau to obtain the preferred rates. A confirmation will be mailed from the Bureau.

### Deposits

A \$100 deposit per guest room and \$300 deposit per suite is required in order to process requests. This is a refundable deposit. The following methods of payment are acceptable: checks made payable to the FSCT Housing Bureau or credit cards. Credit card choices include American Express, Mastercard, or Visa. Please note that the deposit will be applied immediately to the credit card used.

### Deadlines

Reservations must be placed by September 18 to obtain the convention rates. After September 18, call the hotel directly.

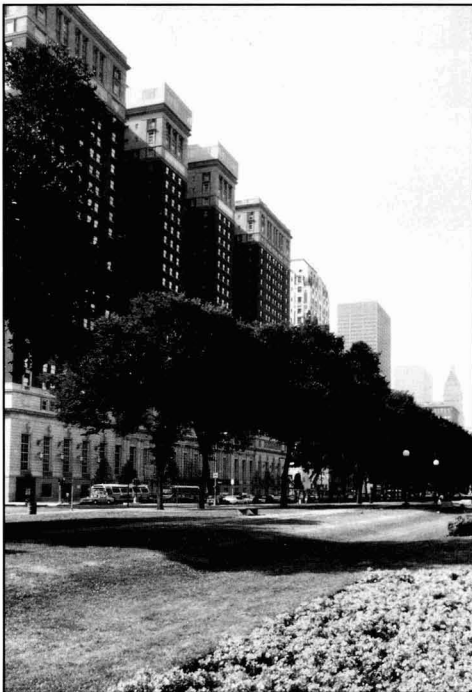


Photo courtesy of Ron Schramm/Chicago Convention & Visitors Bureau

The Chicago Hilton—headquarters hotel for the FSCT Annual Meeting & Paint Industries' Show

### Changes/Cancellations:

For changes or cancellations prior to September 18, call the FSCT Housing Bureau at 312-567-8507 or fax your change to 312-567-8577. After September 18, call the hotel directly.

### Do it Your Way and Save!

You have the convenience of placing a phone call, mailing, or faxing your request for hotel accommodations to the FSCT Housing Bureau.

### Call! 800-723-2000 or 312-567-8507

Have the information requested on the Hotel Reservation Form available before you place your call. Reservationists are available 8:30 a.m. - 6:30 p.m., Monday through Friday, Central Standard Time. Reservations will be immediately confirmed over the phone and also by mail.

### Fax! 312-567-8577

Anytime, any day. Fax the Hotel Reservation Form at your convenience. Be sure to include a phone number and retain your copy of the form for your records. Confirmations will be sent via fax within one working day of receipt of request.

### Mail!

Send your form to the FSCT Housing Bureau for processing. Be sure to include a phone number and keep a copy of the form for your records. Your confirmation will be mailed.

### Airline Information

The Federation's Travel Desk has negotiated super deals on United and USAir to Chicago featuring up to 40% discounts. To make your transportation arrangements to the Paint Show, call the FSCT Travel Desk at 1-800-448-FSCT or 215-628-2549 and mention "Paint Show 92." An agent will make your reservations, write your tickets using your credit card number, and mail the tickets directly to you.

or

Call the official carriers directly. Be sure to reference the file numbers provided:

USAir 1-800-334-8644  
REFERENCE Gold File#62330000

United\* 1-800-521-4041  
REFERENCE Meeting Code: 533GU

\* Seven day advance purchase required.

For each airline, certain restrictions may apply.



## Participating Hotels

### **Chicago Hilton and Towers** (headquarters)

Luxury property located on Grant Park and five minutes from McCormick Place. Shuttle service, twenty-four hour coffee shop and room service. Fine dining at Buckingham's, Kitty O'Sheas Irish Pub, Fast Lane Deli plus entertainment in Lakeside Green Lounge. Special Towers accommodations for business travelers. Services include: health club with indoor pool, business center, drug store, unisex hair salon, boutiques and indoor parking garage.

312-922-4400

(Requests for rooms limited to 10 per company)

### **Hyatt Regency Chicago**

Thirty six-story, twin-tower hotel with 10 restaurants and lounges. Located on the Chicago River and at the beginning of Chicago's Magnificent Mile, offering immediate access to shopping, entertainment, parks, museums, and other cultural attractions.

312-565-1234

### **McCormick Center Hotel**

Features newly remodeled guest rooms. Connected to McCormick Place North by covered pedestrian walkway. Hotel operates three restaurants, complimentary indoor pool and full service health club.

312-791-1900

(Requests for rooms limited to 10 per company)

### **Palmer House Hilton**

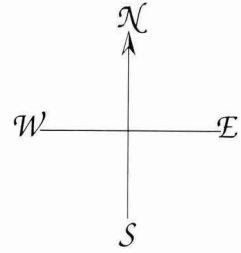
Historic hotel with newly restored guest rooms in downtown location. Five blocks to the Chicago Hilton and over 50 restaurants nearby. Deluxe category hotel, featuring five restaurants, fitness center, business center and ticket/tour desk. (NPCA headquarters.)

312-726-7500

### **Executive Plaza Hotel**

The Executive Plaza Hotel is conveniently located just west of Michigan Avenue, at the Chicago River. Walk to the finest restaurants, night clubs, and Chicago's most popular attractions. Large guest rooms feature a separate seating area, work area, remote control TV, mini bar, coffee maker, and clock radio...additional services include multi-lingual concierge staff, exercise facility and business center.

312-346-7100



### **Essex Inn on Grant Park**

Across from Grant Park and close to the FSCT headquarters hotel. Free courtesy shuttle downtown. New York style deli and lounge. Outdoor heated pool. Free parking for guests. Airport limousine service.

312-939-2800

### **Stouffer Riviere Hotel**

New hotel located at the corner of State St. and Wacker Dr. overlooking the Chicago River. Within walking distance to Chicago's business, culture and shopping districts. Guests enjoy complimentary coffee, newspaper, with wake up call. Complimentary shoeshine service, complimentary health club.

312-372-7200

## Useful Phone Numbers

Federation's Travel Desk ..... 1-800-448-FSCT  
or 215-628-2549

Reference: "Paint Show 92"

USAir: 1-800-334-8644, Ref. Gold File #62330000.

United: 1-800-521-4041, Ref. Meeting Code 533GU.

FSCT Housing Department ..... 800-723-2000  
312-567-8507

## Dates to Remember

Hotel Reservations (deadline) ..... September 18

Advance Registration (deadline) ..... September 18

Annual Meeting @ Paint Show ..... October 21-23

## Hotel Room and Suite Rates\*

Code	Property	Singles	Doubles/Twins	1 BR Suite *	2 BR Suite *
#111	Chicago Hilton Hotel	\$120,145,175	\$145,155,185	\$395-560	\$525-735
#119	Chicago Hilton Towers	\$210	\$225	\$450-675	\$665-895
#113	Hyatt Regency Chicago	\$142	\$162	\$356-2500	\$507-2690
#110	McCormick Center Hotel	\$129,145	\$149,165	\$375-504	\$673-1200
#112	Palmer House Hilton	\$115,130,145	\$130,145,160	\$295-710	\$615-865
#107	Executive Plaza Hotel	\$110	\$125	\$175	\$250
#106	Essex Inn on Grant Park	\$96	\$108		
#116	Stouffer Riviere Hotel	\$145	\$165	\$450 & up	\$550 & up

\* Rates do not include 12.4% hotel tax. (Subject to change.)

Requests for rooms at the Chicago Hilton and Towers and McCormick Center Hotel will be limited to 10 rooms per company.

Room Type Key: Single (1 person, 1 bed); Double (2 people, 1 bed); Twin (2 people, 2 beds); 1BR Suite (parlor + 1 bedroom); 2 BR Suite (parlor + 2 bedrooms).

### Shuttle Service

Shuttle Bus service will be provided between the cooperating hotels\* and the McCormick Place North. The routes and schedules are listed below.

#### Route 1

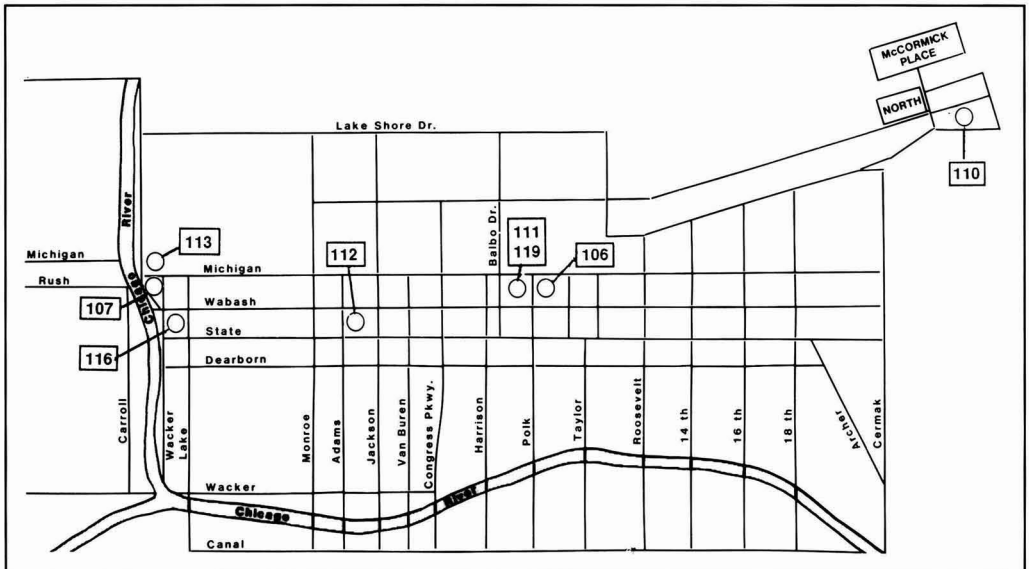
Chicago Hilton—  
8th Street Entrance  
  
Essex Inn on Grant—  
Walk to Chicago Hilton  
  
Palmer House—  
Wabash Avenue Entrance

#### Route 2

Executive Plaza Hotel—  
Wacker Drive  
  
Stouffer Riviere Hotel—  
Walk to Executive Plaza Hotel  
  
Hyatt Regency Chicago—  
Wacker Drive Eastbound

#### Shuttle Schedule Hours of Operation

Tuesday, October 20—  
8:00 am - 8:30 pm  
  
Wednesday, October 21—  
7:30 am - 6:30 pm  
6:30 - 12 midnight\*  
\*(Shuttle service between the  
cooperating hotels and the  
Chicago Hilton)  
  
Thursday, October 22—  
7:30 am - 6:30 pm  
Friday, October 23—  
7:30 am - 3:00 pm







## HOW TO MAKE YOUR ARRANGEMENTS

1. To place AIRLINE reservations, call the Federation's Travel Desk at 1-800-448-FSCT or 215-628-2549 and mention "Paint Show 92". To contact carriers directly, call the following numbers and reference the file numbers provided: US Air: 1-800-334-8644, Ref. Gold File #62330000. United: 1-800-521-4041, Ref. Meeting Code 533GU.
2. To make HOTEL reservations, mail or fax the housing application to the FSCT Housing Department. Housing cut-off date is September 18.
3. REGISTER IN ADVANCE for the Annual Meeting and Paint Industries' Show by filling out the form and mailing it as instructed with your registration payment.
4. To register your SPOUSE or GUEST, fill out the spouse portion of the advance registration form.
5. Mark OCTOBER 21-23 on your calendar. Don't forget — you get a discount if you register by September 18.

## Registration Instructions

Advance register to attend the 1992 Annual Meeting and Paint Industries' Show by filling out and mailing the registration form and fees to the FSCT Headquarters Office.

The registration options are listed below. Advance registration forms must be received by September 18.

Register in Advance and SAVE!

	<b>Full Time</b>	<b>Advance</b>	<b>On-Site</b>
Member .....	\$65		\$75
Non-member .....	\$80		\$95
Spouse .....	\$50		\$60

### Advance Registration

If you register in advance you may pick up your badge at the McCormick Place North Registration Area during the following hours:

Tues., Oct. 20 .....	8:00 am - 5:00 pm
Wed.-Thurs., Oct. 21-22 .....	7:30 am - 5:30 pm
Fri., Oct. 23 .....	7:30 am - 12:00 noon

### On-Site Registration

Register at McCormick Place North.

Tues., Oct. 20 .....	8:00 am - 5:00 pm
Wed.-Thurs., Oct. 21-22 .....	7:30 am - 5:30 pm
Fri., Oct. 23 .....	7:30 am - 12:00 noon

## Cancellation and Refund Policy

All cancellations must be submitted in writing to the FSCT Headquarters Office. Cancellations received by October 16 will be subject to a \$10 handling charge. A \$25 charge will be made after that date.

## Hotel Reservation Instructions

(1) Reservations must be placed by September 18. Reservations may be phoned, faxed or mailed to the FSCT Housing Bureau.

(2) Confirmations will be mailed from the Housing Bureau. Please allow 30 days for receipt of confirmation.

(3) A one-night's deposit **must** accompany each reservation request. Requests will not be processed without deposit or credit card. Acceptable payments include: personal check, bank draft, and certified check. Checks should be made payable to FSCT Housing Bureau. Credit cards may be used.

(4) Keep a photocopy of your housing request.

(5) Prior to September 18, all changes must be made through the FSCT Housing Bureau. After September 18, all changes should be made directly with the Bureau, subject to availability.

## Airport & City Transportation

### From Midway Airport

Airport Shuttle (service to downtown hotels, provided by Continental Air Transport)	
.....	\$9.50 one way, \$16.75 round trip
Limousine .....	\$45
Taxi .....	\$20-22

### From O'Hare International Airport

Airport Shuttle (service to downtown hotels, provided by Continental Air Transport)	
.....	\$12.50 one way, \$22.00 round trip
Limousine .....	\$45
Taxi .....	\$20-22

(There is also CTA subway service from O'Hare to the Loop on a 24-hour basis, seven days a week. Board trains on the lower level of Terminal 4. The fare is \$1.00 and the ride takes approximately 34 minutes.)

To get around Chicago, taxis are readily available throughout the downtown and near north areas. You'll find most of Chicago's best-known restaurants and entertainment are near downtown hotels. The public transportation system runs 24 hours a day, and routes are clearly posted on CTA vehicles, bus stops, and at subway and El stations. If you have a question about modes of transportation, ask the concierge or front desk personnel at your hotel.

## ***Columnist Jack Anderson to Give Keynote Address at Annual Meeting***

Noted columnist Jack Anderson will present the Keynote Address at Wednesday's Opening Session at the FSCT 70th Annual Meeting. With his *Merry Go Round* column appearing in over 1000 newspapers daily, Anderson is the most widely syndicated columnist in the world. Additionally, he is a regular contributor to *The Real Story*, CNBC's nightly live information-based show; a daily radio commentator for UPI Radio Network's 1100 stations; the best-selling author of numerous books (the most recent being *Stormin' Norman*); and the most dynamic speaker on the nation's lecture circuit.

It was from his column that we first heard of the Savings and Loan scandal, the Iran/Contra Arms-for-Hostages deal, and the danger of Saddam Hussein. Anderson fascinates and educates audiences with his insights and inside stories—always standing for the public's right to know.

## ***Spouses to Tour Chicago Sites***

Spouses Activities begin on Wednesday, October 21, with a wine and cheese social in the Chicago Hilton Hotel.

On Thursday, following a continental breakfast in the Chicago Hilton, registered spouses will depart on deluxe motorcoaches for the Art Institute of Chicago and the John G. Shedd Aquarium.

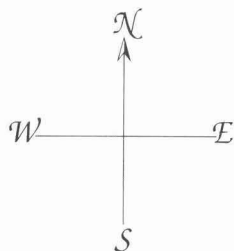
To enhance the viewing of the Art Institute's magnificent collection, spouses will receive a private lecture on "Highlights of the Art Institute of Chicago." The monumental stained-glass America Window by Chagall, and the great gilded Trading Room of Chicago's famous Stock Exchange Building will be among the highlights. After the presentation, spouses will have ample time to view the galleries.

Chicago's newest attraction, the Oceanarium at the Shedd Aquarium, will also be included in the tour. The Oceanarium is the world's largest indoor marine pavilion. Home to the Harbor Seals, Sea Otters, and Rockhopper Penguins, the Oceanarium features an elaborate re-creation of the Pacific Northwest temperate rain forest habitat. Spouses will stroll through scenic nature trails to an amphitheater where the Oceanarium staff will demonstrate the behaviors of beluga whales and white side dolphins.

A delicious luncheon will be served at the Mid-America Club located atop one of the tallest buildings in Chicago, offering a spectacular view of the Chicago skyline.

Comfortable walking shoes are recommended.

**The tour limit is 650. Advance registration for the spouses activities is recommended.**



## ***FSCT Board of Directors to Meet On Tuesday at Chicago Hilton***

The Board of Directors of the Federation will meet on Tuesday, October 20, at 9:00 am in the Chicago Hilton Hotel.

## ***NPCA Annual Meeting Same Week***

The National Paint & Coatings Association will hold its annual meeting on October 19-21, 1992, at the Palmer House, in Chicago.

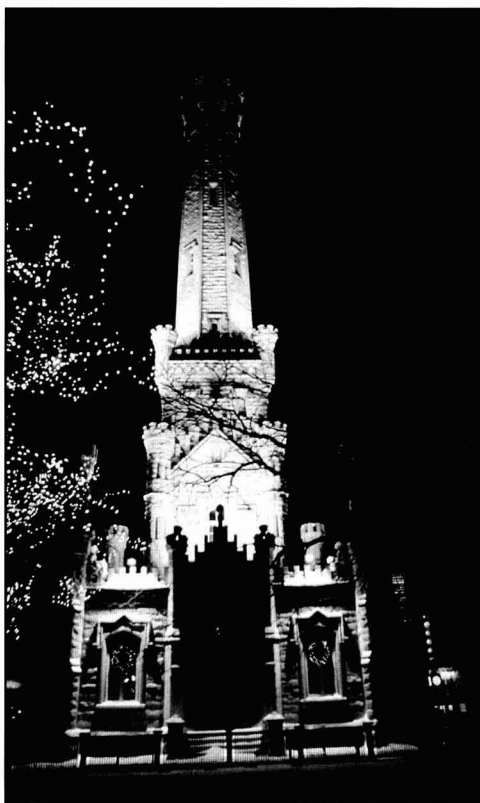


Photo courtesy of Ron Schramm/Chicago Convention & Tourism Bureau

Chicago's famous landmark— Water Tower Place

# 1992 PAINT INDUSTRIES' SHOW

## Current List of Exhibitors

- Aceto Corp.  
Advanced Coatings Technologies  
Advanced Software Designs  
Aggio Recovery, Inc.  
Air Products & Chemicals, Inc.  
Ajinomoto U.S.A., Inc.  
Akzo Chemicals & Resins  
Alcan-Toyo America, Inc.  
Alcoa Industrial Chemicals  
Allied-Signal Corp.  
Ambrose Co.  
ACS/C&E News  
ACS/Industry Relations  
American Cyanamid Co.  
American Iron & Steel Institute  
Amoco Chemical Co.  
ANGUS Chemical Co.  
Anker Labelers USA, Inc.  
Aqualon Co.  
Arco Chemical Co.  
Ashland Chemical, Inc. IC&S Div.  
Atlas Electric Devices Co.  
Aztec Catalyt Co.
- B&P Environmental Resources  
B.A.G. Corp.  
BASF Corp.  
T.J. Bell, Inc./Erichsen Instruments  
Blackmer Pump, Dover Resources  
Bohlin Reologi, Inc.  
Brookfield Engineering Labs., Inc.  
Brookhaven Instruments Corp.  
Buckman Laboratories, Inc.  
Buhler, Inc.  
Bulk Lift International, Inc.  
Burgess Pigment Co.  
BYK-Chemie USA  
BYK-Gardner, Inc.
- C B Mills, Inc.  
CR Minerals Corp.  
Cabot Corp., Cab-O-Sil & Special Blacks Div.  
Calgon Corp., Div. of Merck & Co.  
Cappelle, Inc.  
The Carborundum Co.  
Cardolite Corp.  
Cargill, Inc.  
Carri-Med Americas, Inc.  
Carroll Scientific, Inc.  
Celite Corp.  
Chemical Marketing Reporter  
Chemical Week  
CIBA-GEIGY Corp.  
Coatings Magazine  
Color Corp. of America  
Colorgen, Inc.  
Colortec Associates  
Columbian Chemicals Co.  
Consolidated Research, Inc.  
Cookson Pigments, Inc.  
Coulter Corp.  
Cray Valley Products, Inc.  
Crosfield Chemicals, Inc.  
Cuno Process Filtration Products  
Cyprus Industrial Minerals Co.
- D/L Laboratories  
DSA Consulting, Inc.  
Daniel Products Co.  
Dantco Mixers Corp.  
Datacolor International  
Day-Glo Color Corp.  
Defelsko Corp.  
Degussa Corp.  
University of Detroit Mercy
- Distil-Kleen, Inc.  
Dominion Colour Corp.  
Dow Chemical USA  
Dow Corning Corp.  
Draiswerke, Inc.  
Drew Industrial Div.  
Dry Branch Kaolin Co.  
Du Pont Co.
- ECC International  
EM Industries, Inc.  
Eagle Picher Minerals, Inc.  
Eagle Zinc Co.  
Eastern Michigan University  
Eastman Chemical Co.  
Ebonex Corp.  
Eiger Machinery, Inc.  
Elektro-Physik USA, Inc.  
Elf Atochem  
Elmar Worldwide  
Engelhard Corp.  
Epworth Manufacturing Co., Inc.  
Etna Products Inc.  
Exxon Chemical Co.
- FMC Corp., Food & Pharmaceutical Prod. Div.  
Fawcett Co., Inc.  
**Federation of Societies for Coatings Technology**  
Filter Specialists, Inc.  
Fischer Technology, Inc.  
Fluid Management Ltd. Partners  
FMJ International Publications Ltd.  
Freeman Polymers  
H.B. Fuller Co.
- Paul N. Gardner Co., Inc.  
B.F. Goodrich Co., Spec. Polym. & Chem. Div.  
Goodyear Tire & Rubber Co., W.R. Grace & Co., Davison Chemical Div.  
Guer-Tin Bros. Polymers
- Halox Pigments, Div. of Hammond Lead Products  
Harcros Pigments, Inc.  
Henkel Corp.  
Heraeus DSET Laboratories, Inc.  
Heucotech Ltd.  
Hilton-Davis Co.  
Hitox Corp. of America  
Hockmeyer Equipment Corp.  
Hoechst Celanese Corp., Pigments Div.  
Hoechst Celanese Corp., Waxes & Lubricants Group  
Horiba Instruments, Inc.  
J.M. Huber Corp.  
Hüls America, Inc.  
Hunterlab
- ICI Americas, Inc.  
ICI Resins U.S.  
ISP Filters, Inc.  
ITT Marlo Pumps  
Ideal Manufacturing & Sales  
Industrial Finishing Magazine  
Interfibe Corp.  
International Compliance Center  
International Resources, Inc.  
International Specialty Products
- S.C. Johnson Polymer
- KTA-Tator, Inc.  
K-T Feldspar Corp.  
Kemira, Inc.  
Kenrich Petrochemicals, Inc.  
Kent State University  
King Industries, Inc.  
KRONOS, Inc.
- Lawter International  
Leeds & Northrup, Microtrac Div.  
LeSac Corp.  
Lightnin  
Liquid Controls Corp.  
The Lubrizol Corp.
- 3M, Industrial Chemical Prod. Div.  
Macbeth, Div. of Kollmorgen Corp.  
Malvern Instruments  
Malvern Minerals Co.  
Matec Applied Sciences  
McWhorter, Inc.  
The Mearl Corp.  
Michelman, Inc.  
Micro Powders, Inc.  
Microfluidics Corp.  
Micromeritics Instrument Corp.  
Miles Inc.  
Millipore Corp.  
Mineral Pigments  
MiniFibers, Inc.  
Minolta Corp.  
Mississippi Lime Co.  
University of Missouri-Rolla  
Modern Paint & Coatings  
Morehouse Cowles, Inc.  
Morton International/Universal Color Dispersions  
Mountain Minerals Co., Ltd.  
Myers Engineering
- NYCO Minerals, Inc.  
Nacan Products Ltd.  
National Chemical Co.  
Netzsch Incorporated  
Neupak, Inc.  
Neville Chemical Co.  
New Way Packaging Machinery  
Nicolet Instrument Corp.  
Nippon Shokubai Co., Ltd.  
North Dakota State University
- Obron Atlantic Corp.  
Ohio Polychemical Co.  
Olin Chemicals  
ORTECH International
- PPG Industries, Inc., Silica Products  
PPG Industries, Inc., Specialty Chemicals  
PQ Corp.  
Pacific Micro Software Engineering  
Paint & Coatings Industry Magazine  
Peninsula Polymers  
Pen Kem, Inc.  
Pfeizer Specialty Minerals  
Philips Container Co.  
Physica USA  
Pico Chemical Corp.  
Plastican, Inc.  
Polar Minerals  
Pollution Control Industries  
Poly-Resyn, Inc.  
Premier Mill Corp.  
Progressive Recovery, Inc.  
Pyosa, S.A. de C.V.
- The Q-Panel Co.  
The Quackenbush Co.  
Quantachrome Corp.
- Raabe Corp.  
Ranbar Technology Inc.  
Reichhold Chemicals, Inc.  
RHEOX, Inc.  
Rhône-Poulenc Inc.  
Rohm and Haas Co.  
Rosedale Products, Inc.  
Charles Ross and Son Co.  
Russell Finex Inc.
- Sandoz Chemicals Corp.  
Sannor Industries, Inc.  
Sartomer Co. Inc.  
Schold Machine Corp.  
SCM Chemicals  
Scott-Bader  
Semi-Bulk Systems, Inc.  
Serac, Inc.  
Shamrock Technologies, Inc.  
Shell Chemical Co.  
Sherwin-Williams Chemicals Co.  
Shimadzu Scientific Instruments  
Silberline Manufacturing Co.  
Sino-American Minerals, Inc.  
Sloss Industries Corp.  
South Florida Test Service, Inc.  
Southern Clay Products, Inc.  
University of Southern Mississippi  
Spartan Color Corp.  
Startex Chemical Co.  
Steel Structures Painting Council  
Sub-Tropical Testing Service  
Sun Chemical Corp.  
Systech Environmental Corp.
- Tego Chemie Service USA  
Teledyne Taber  
Texaco Chemical Co.  
Thiele Engineering Co.  
Tioxide, Inc.  
Troy Corp.
- U.S. Borax  
R.T. Silica Co.  
Unimin Specialty Minerals Inc.  
Union Carbide Corp.  
Union Process, Inc.  
United Catalysts, Inc., Rho. & Perf. Minerals Group  
United Mineral & Chemical Corp.  
United States Testing Co., Inc.
- Van Waters & Rogers  
R.T. Vanderbilt Co., Inc.  
Velcol Chemical Corp.  
Versa-Matic Tool, Inc.  
Viking Pump, Inc., a Unit of IDEX  
Vortri-Siv., Div. of M&M Industries
- Wacker Silicones Corp.  
Wallon Machinery, Inc.  
Warren-Rupp, Inc., a Unit of IDEX  
Wilden Pump & Engineering Co.  
Witco Corp.
- X-Rite, Inc.
- Zeelan Industries, Inc.



# 1992 Advance Registration

**FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY**  
**492 Norristown Rd., Blue Bell, PA 19422-2350**

<b>C</b>	Office Use Only
<b>U</b>	Date Received _____
<b>V</b>	Amount \$ _____
	Check No. _____

Please fill out this form and mail with a check in the correct amount (made payable to the FSCT) **to the Federation address shown above.** All checks must be payable in U.S. Funds. Any that are not will be returned. **DEADLINE DATE FOR ADVANCE REGISTRATION IS SEPTEMBER 18.** NONE WILL BE ACCEPTED AFTER THAT DATE.

*NO CREDIT CARDS WILL BE ACCEPTED. NO FAXES WILL BE ACCEPTED.*

**INDUSTRY REGISTRATION FEES:**

**INFORMATION FOR REGISTRATION BADGE:**

**A**  **MEMBER**      **\$65.00**

Please name the Federation Society in which you are a paid-up member:

Federation Constituent Society \_\_\_\_\_

**B**  **NON-MEMBER**      **\$80.00**

**G**  **SPECIAL FEE FOR RETIRED MEMBERS**      **\$25.00**

Federation Constituent Society \_\_\_\_\_

NICKNAME

\_\_\_\_\_

FIRST NAME      LAST NAME

\_\_\_\_\_

COMPANY

\_\_\_\_\_

STREET

\_\_\_\_\_

CITY

STATE (U.S. only)

POSTAL CODE

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

COUNTRY (OTHER THAN U.S.)

\_\_\_\_\_

TELEPHONE NO.

\_\_\_\_\_

**BUSINESS CLASSIFICATION DATA FOR THE ABOVE REGISTRANT:**

**YOUR COMPANY (CHECK ONE BLOCK)**

AA  Manufacturers of Paints, Varnishes, Lacquers, Printing Inks, Sealants

BB  Manufacturers of Raw Materials

CC  Manufacturers of Equipment and Containers

DD  Sales Agent for Raw Materials + Equipment

EE  Government Agency

FF  Research/Testing/Consulting

GG  Educational Institution Library

HH  Paint Consumer

JJ  Other

**YOUR POSITION (CHECK ONE BLOCK)**

KK  Management/Administration

LL  Manufacturing and Engineering

MM  Quality Control

NN  Research and Development

PP  Technical Sales Service

QQ  Sales and Marketing

RR  Consultant

SS  Educator/Student/Librarian

TT  Other

**SPOUSES REGISTRATION AND INFORMATION FOR REGISTRATION BADGE:**

**D**  **SPOUSE**      **\$50.00**

NICKNAME

\_\_\_\_\_

FIRST NAME      LAST NAME

\_\_\_\_\_

**SPECIAL FEE FOR THE SPOUSES OF RETIRED MEMBERS ONLY:**

**H**       **\$25.00**

CITY

STATE (U.S. only)

POSTAL CODE

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**TICKETS FOR FEDERATION LUNCHEON, FRIDAY, OCTOBER 23 (@ \$25.00)**

**NUMBER REQUIRED:** \_\_\_\_\_  
**\$25.00 EACH.**

A \$10.00 CHARGE WILL BE MADE FOR CANCELLATIONS RECEIVED PRIOR TO OCTOBER 16. A \$25.00 CHARGE WILL BE MADE FOR CANCELLATIONS RECEIVED AFTER THAT DATE.

# PICTORIAL STANDARDS OF COATINGS DEFECTS

Revised and updated edition of this manual (previously titled "Exposure Standards Manual") has been compiled in conjunction with the American Society for Testing and Materials, and includes definition, description, and photographic standards for each of the following defects: Adhesion; Blistering; Chalking; Checking; Cracking; Erosion; Filiform Corrosion; Flaking; Mildew; Print; Rust; Traffic Paint Abrasion and Chipping.

Also included is reference information on supplementary standards, along with sample record sheets for compiling exposure data.

Bound in handsome 10" × 11½" × 1½" three-ring, vinyl-covered binder which readily accommodates additional material as it is developed.

**Complete manual . . . \$100**  
**Individual Standards . . . \$3 each, plus \$3 for each photograph**  
**Record Sheets (pad of 100 sheets) . . . \$3.50**

*Please make all checks payable in U.S. funds.*

*\*Pennsylvania residents add 6% sales tax.*

Send orders to: Federation of Societies for Coatings Technology  
492 Norristown Rd., Blue Bell, PA 19422-2350

Federation of Societies for Coatings Technology

## OSHR Invalidates Target Labeling Requirements; NPCA Completes Paint Exposure Study

The National Paint and Coatings Association (NPCA), Washington, D.C., has reported that a recent ruling by the Occupational Safety and Health Review Commission (OSHR) has invalidated the Occupational Safety and Health Administrator's (OSHA) requirements for target organ labeling under the Hazard Communication Standard (HCS).

According to the NPCA, the ruling has the potential to affect long-standing compliance programs for the labeling of products containing hazardous chemicals.

In its ruling, the OSHRC noted that the standard does not define "appropriate hazard warnings" required for labels, and thus allows industry to adopt a performance-oriented approach. Also, OSHRC reported that the purpose of the HCS was to provide a "system of information for workers," and that the label was intended to provide only the immediate information needed for safe use of the chemical, while the Material Safety Data Sheet could conceivably contain more information, such as target organ health effects.

The possibility exists that OSHA may yet appeal the OSHRC decision to invalidate requirements for target organ labeling under the HCS.

In other news, NPCA reported the results of a recently completed research project to evaluate worker and consumer exposure to the volatile components of a typical vinyl acrylic housepaint. According to the study, it was determined that exposures *never* exceeded any applicable workplace limits, even in "closed room" conditions using spray application.

The research indicated that, in most cases, the concentration of volatile components was reduced below detectable limits within six hours of application.

In the study, the test paint was applied to a series of similar rooms in a dormitory setting. Variables included the method of application (roller vs airless spray) and rate of fresh air entry (0.5 vs 5.0 changes per hour).

According to officials at NPCA, the volatile components project was the first of its kind for NPCA, and illustrates the benefits of close cooperation between paint manufacturers and raw materials suppliers in research aimed at expanding and improving product stewardship initiatives. Also, additional benefits of the study are expected to

be derived in the likely upcoming discussions between NPCA and the Environmental Protection Agency's Office of Indoor Air Quality.

The study was conducted by International Technology Corporation in conjunction with the University of Cincinnati. Funding was provided by NPCA, Union Carbide Chemicals & Plastics Company, Unocal Chemicals & Minerals Division,

Rohm and Haas Company, Hoechst Celanese Chemical Group, and Reichhold Chemicals Inc.

A Safety and Health Bulletin is being prepared on the study results. For more information, contact Steve Sides, NPCA, Division Director, Health, Safety & Environmental Affairs, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005-5597; Telephone: (202) 462-6272.

### Collaborative Testing Services Develops Pencil Hardness Test for Paint Labs

Collaborative Testing Services (CTS), Inc., Herndon, VA, and the Federation of Societies for Coatings Technology, Blue Bell, PA, have announced the addition of a pencil hardness test in the Paint & Coatings Collaborative Reference Program for 1992.

The test will expand the ability of paint laboratories to ensure that quality control standards are being maintained, as well as provide a means for comparing performance with other laboratories on a national basis.

The Federation serves as a program affiliate for the Paint & Coatings program, and provides technical assistance in the development of all CTS paint tests. In addition, the entire program meets the criteria established for an external proficiency testing program by the American Association for Laboratory Accreditation, as well as the National Volunteer Laboratory Accreditation Program at the U.S. Department of Commerce.

The new test is known as Film Hardness by Pencil Test. Two coated panels will be supplied by CTS to the participants, who will conduct the test in accordance with ASTM Method D 3363. Laboratories will indicate lead or pencil used, as well as report gouge hardness and scratch hardness. The deadline for enrolling in the program is June 8, 1992.

For more information, or to become a participant in the program, contact: Charles G. Leete, President, CTS, 340 Herndon Pkwy., P.O. Box 1049, Herndon, VA 22070; Telephone: (703) 742-9107.

### Henkel Corporation Increasing Production Capacity

The Coatings & Inks Division of Henkel Corporation has announced increased production capacity, a new applications laboratory, and an expanded technical service staff.

Production facilities in Charlotte, NC, are being expanded to increase capacity for the production of Photomer® radiation curable acrylate monomers for use in ultraviolet/electron beam curable coatings and inks. The new expansion will incorporate a pro-

prietary esterification process. This production expansion is scheduled to be completed in 1993.

An investment in new applications laboratory equipment to better test and understand the performance characteristics of Photomer products is underway at Henkel's Ambler, PA, facility. Also, the Inks business sector has expanded its technical service staff.



## Dow Corning Corp. and Van Leer Containers Launch Steel Drum Recycling Program

Dow Corning Corporation, Midland, MI, has instituted a program designed to address environmental, economic, and quality issues related to using 55-gallon drums containing silicone-based and other specialty chemical products.

The program, cosponsored by Dow Corning and Van Leer Containers, Inc., Chicago, IL, was launched on January 1, to provide customers with a way to reuse/recycle empty 55-gallon drums. In addition, the program promotes proper container-emptying methods.

The Steel Drum Recycling Program has approximately 10 drum reconditioners across

the U.S. as part of the program. The drum reconditioners are audited by both Dow Corning and Van Leer to ensure that the reconditioners' facilities meet the necessary criteria governing financial, administrative, and process procedures and capabilities.

Also, pick-ups of drums usually will be made within 15 days of the request, regardless of the location or number of drums.

Officials at Dow Corning noted that the program meets or exceeds guidelines established by the U.S. Department of Transportation and the National Barrel and Drum Association/National Association of Container Reconditioners.

For additional details, write Dow Corning Corporation, Midland, MI 48686-0994.

### Cray Valley Coating Resins Relocates to South Wales

The Coating Resin Department of Cray Valley Ltd., Kent, England, is relocating its headquarters from Farnborough, in Kent, to Machen, in South Wales.

Machen has served as Cray Valley's major United Kingdom production site. Now, Machen also will serve as the main location for Cray Valley Coating Resin research and development, as well as being the administrative center.

The relocation has involved construction of a new headquarters and research buildings which are due to be completed in June. The entire relocation process is expected to be completed by the end of July.

### Lord Corporation Opens Technical Center in Mexico

Lord Corporation, Erie, PA, dedicated the Lord de Mexico's Technical Center, a general office, and an industrial facility, at Toluca Industrial Park, near Mexico City, Mexico, on February 28.

The 5,000-sq ft facility will be used initially as a technical center, and will have manufacturing capability for the principal Chemlok® products. The plant will directly market the Fusor®, Versilok®, and Tyrite® industrial adhesives lines along with the Tycel® laminating adhesives product line.

The new technical center will offer customer support by testing the application of products, and later will expand its service to more specialized activities.

### Du Pont Begins Operation Of Brazilian TiO<sub>2</sub> Pigment Plant

Du Pont, Wilmington, DE, has begun operation of its Ti-Pure titanium dioxide pigment sizing and packaging facility in Uberaba, Brazil.

The plant receives semi-finished pigment from Du Pont's North American plants and performs the final processing steps in Brazil. The facility is on schedule to process 15,000-20,000 tons of pigment a year.

The pigment plant is located on 235 acres in Minas Gerais, about 200 miles northwest of Sao Paulo. The finishing facility was built using a modular construction concept and is designed to be expanded to a fully-integrated titanium dioxide manufacturing plant.

## Mergers & Acquisitions...

### Ashland Acquires Unocal Chemical Distribution Business

Ashland Chemical, Inc., Columbus, OH, has acquired the chemical distribution business of Unocal Corporation. According to officials at Ashland, the purchase price was approximately \$90 million.

The business involves the distribution of a wide range of organic chemicals, hydrocarbon solvents, and specialty and inorganic products through a network of distribution centers and bulk terminals.

The business will become part of Ashland's Industrial Chemicals & Solvents Division.

### Hüls Sells Metal Stearates Business to Synthetic Products

Hüls America Inc., Piscataway, NJ, has sold its metallic stearates business to Synthetic Products Company, a subsidiary of Cookson America Inc. The transaction was completed on March 13.

The sale includes Hüls' diffusion technology patent for producing aluminum, calcium, magnesium, and zinc stearates. Hüls will retain its Nuodex trademark and its Piscataway plant where it has manufactured stearates.

### Du Pont Purchases Land from Union Camp Corporation

Du Pont, Wilmington, DE, has completed negotiations with the Union Camp Corporation to purchase 15,400 acres in Charlton County, GA, for the potential mining of mineral sands.

The minerals include ilmemeite ore, used in the production of titanium dioxide, and additional minerals for use in ceramic, refractory, and foundry industries.

The acreage is an extension of a 7,200-acre site in northeastern Florida.

In addition, Du Pont and Union Camp have negotiated a long-term lease that will allow Union Camp to continue management of the property for current uses, including timber production and harvesting.

### Pioneer Paint Acquires Jordan Paint Assets

Pioneer Paint Products, Inc., Melrose Park, IL, has acquired the assets of the Jordan Paint Manufacturing Company, Forest Park, IL.

The Jordan product lines will be manufactured at the Pioneer Paint site.

# Regulatory UPDATE

MAY 1992

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. and edited by members of the FSCT Environmental Affairs Committee.

## **Environmental Protection Agency**

**April 2, 1992—57 FR 11394**

### **National Pollutant Discharge Elimination System (NPDES) Application Deadlines, General Permit Requirements, and Reporting Requirements for Storm Water Discharges Associated with Industrial Activity**

**Action: Final rule**

On April 6, the U.S. Environmental Protection Agency (EPA) issued a final proposal setting forth a national strategy for issuing NPDES permits for industrial storm water discharges. In August 1991, EPA requested public comment on several regulatory and policy issues regarding storm water associated with industrial activity. The final rule, effective May 4, 1992, is based on those comments.

Also promulgated are revisions to the minimum monitoring requirements, and minimum requirements for filing notices of intent to be authorized to discharge under NPDES general permits. A deadline of October 1, 1992 has also been set for filing Part 2 of storm water group applications.

For further information, contact the NPDES Storm Water Hotline at (703) 821-4823 or write Kevin Weiss, Office of Wastewater Enforcement and Compliance (EN-336), U.S. EPA, 401 M Street, S.W., Washington, D.C. 20460, (202) 260-9518.

## **Department of Transportation**

### **Research and Special Programs Administration (RSPA)**

**April 8, 1992—57 FR 11984**

### **City of New York; Application for a Waiver of Pre-emption Determination**

**Action: Notice**

The city of New York has applied for a pre-emption waiver under the Hazardous Materials Transportation Act (HMTA). The waiver request concerns the transportation of flammable and combustible liquids and flammable and compressed gases for pickup or delivery within the city.

During a March 25 hearing before the Appropriations Committee's Subcommittee on Transportation, Sen. Alphonse D'Amato (D-NY), warned the Research and Special Programs Administration (RSPA) that it would be a "terrible mistake" if RSPA were to deny the city of New York a waiver of Federal pre-emption for the city's tank truck rules.

Under the rules of the HMTA, there are specific guidelines as to whether a state or local law should be exempted from a pre-emptive Federal rule. The safety factors of the state or local law must be at least as stringent as the Federal rules, and must not unduly disrupt commerce.

Reportedly, Sen. D'Amato has indicated that he would add a provision to the appropriations bill denying 1993 funding to RSPA for a specified period of time until it decides to issue New York City a waiver (RSPA already denied the city a temporary waiver on March 12). In response to Sen. D'Amato's remarks, RSPA has re-opened the comment period on New York City's application for a waiver until the end of April. RSPA's Associate Administrator for Hazardous Materials Safety will issue an administrative ruling after reviewing the comments.

For further information, contact Frazer C. Hilder, Office of the Chief Counsel, Research and Special Programs Administration, U.S. Department of Transportation, Washington, D.C. 20590, (202) 366-4400.

## **Environmental Protection Agency**

**March 20, 1992—57 FR 9678**

### **Approval and Promulgation of Implementation Plans; California State Implementation Plan Revision; Bay Area Air Quality Management District Action: Notice of proposed rulemaking**

The EPA has proposed to approve two volatile organic compound (VOC) rules submitted by the California Air Resources Board (ARB) as revisions to the California State Implementation Plan (SIP). The rules were adopted by the Bay Area Air Quality Management District (BAAQMD) in June 1990, but were ordered set aside by a California Superior Court last year because they were found to be in violation of the California Environmental Quality Act (CEQA). The decision is being appealed.

The rules, regulation 8, rule 3—architectural coatings, and regulation 8, rule 48—industrial maintenance coatings, regulate the VOC content of various architectural and industrial coatings within the BAAQMD. EPA claims it has evaluated the rules and is proposing them despite the court ruling.

For further information, contact John Ungvarsky, Southern California and Arizona Rulemaking Section (A-5-3), Air and Toxics Division, U.S. EPA, Region IX, 75 Hawthorne Street, San Francisco, CA 94105.

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

**Lead**—On April 2, the House Energy and Commerce Subcommittee on Transportation and Hazardous Materials marked up and approved a substitute version of H.R. 3554, The Lead Exposure Reduction Act. The revised version reflects various issues raised at a previous hearing and meetings with environmental, health and industry groups.

The bill includes a ban on zinc-enriched industrial paints that have an incidental presence of more than 0.19% lead. This type of paint is typically used on bridges and infrastructures; requires the EPA to study possible substitutes for lead chromate pigments (e.g., pigments in paints used for yellow highway lines and school buses and for emergency vehicles); expands worker training requirements for lead abatement; and provides that lead abatement contractors must be certified by the Federal government. Also in the bill is a provision for the establishment of a hotline to provide lead abatement information to the general public.

Members of the subcommittee are still trying to resolve provisions that would mandate labeling requirements for all products containing lead. The full committee is expected to take up the measure sometime in May. The Senate counterpart, S. 391, has been approved for floor action but has not yet been scheduled.

**Resource Conservation and Recovery Act (RCRA)**—The House Energy and Commerce Subcommittee on Transportation and Hazardous Materials rejected several amendments to the Resource Conservation and Recovery Act at a markup on March 24.

Rep. Gerry Sikorski (D-MN) offered a series of amendments that would ban incineration of recyclables and non-combustibles, establish minimum content standards for packaging, and require "take-back" of large durable goods—all of which were overwhelmingly rejected. Rep. Don Ritter (R-PA), a ranking member on the committee, was also handed a defeat when he offered an amendment that would allow the Administrator of EPA to consider "practicable capability" when developing rules governed by the solid waste provisions of the bill. Rep. Ritter's amendment pre-empting states from developing their own minimum content standards for recycling was also rejected.

The subcommittee is currently circulating a proposed amendment to RCRA which would add a second section to the current RCRA Subtitle C hazardous waste management scheme, creating a separate regulatory scheme for "hazardous reclaimable material." The new category would cover materials "used or reused as an ingredient in an industrial process to make a product"; used as "an effective substitute for a commercial product"; returned to the original process from which it is generated; extracted as a by-product from a hazardous material and reused; or reclaimed as a "commercial chemical product" that was originally a hazardous waste. Any material being discarded or placed directly on land or incinerated for energy recovery or used in a manner the EPA Administrator determines is "sham" recycling, would not qualify for the alternative regulatory system. The full Energy

and Commerce Committee is expected to markup the legislation at the end of May.

The Senate RCRA bill, S. 976 is slated for markup by the Environment and Public Works Committee also at the end of April. Both the Senate and House would like to have their respective bills on the floor before the July 4th recess, and a final version on the President's desk by October. It is possible that some form of RCRA legislation will be passed this year, but it will probably focus only on solid waste issues. As in the past, members have been unable to come to a working agreement on controversial hazardous waste issues. The contentious issues du jour are toxics use reduction (TUR) and Community-Right-to-Know-More.

**Pollution Prevention**—Legislation that would expand the Toxics Release Inventory (TRI) was introduced on March 17 by Sen. David Durenberger (R-MN). The bill is an amendment to Section 313 of the Emergency Planning and Community-Right-to-Know Act (EPCRA). Under the bill, entitled the Toxics Release and Pollution Prevention Act, S. 2360, each reporting facility would be required to prepare a pollution prevention plan every three years.

Provisions include a requirement that EPA establish a list of 250 toxic chemicals not already listed that are the most harmful to human health and the environment; expansion of the types of facilities that must report toxic release information to include, among others, the Standard Industrial Classification (SIC) codes for petroleum handling, paint supply, and solvent recovery. The definition of "pollution prevention" would also be modified to make toxics use reduction the top priority.

Sen. Durenberger's bill is waiting to be heard by the Senate Environment and Public Works Committee and will undoubtedly be considered during markup of the RCRA reauthorization bill.

**Hazardous Waste**—During the month of March, three hazardous waste bills were introduced in the House. Rep. Pat Williams (D-MT) introduced legislation that would require a two-year moratorium on the burning of hazardous waste in cement kilns. The legislation, H.R. 4413, would also require the EPA to conduct a study of the effects of handling, storing, and burning hazardous waste with regard to human health and the environment. The legislation is now pending in the Energy and Commerce Committee. Sen. Tim Wirth (D-CO), introduced similar legislation (S. 1108) last May but no further action has been taken.

Rep. John Spratt (D-SC), introduced two bills amending the Solid Waste Disposal Act. H.R. 4465 would mandate that EPA impose standards on acceptable locations for treatment, storage, and disposal facilities. Under his other bill, H.R. 4466, owners and operators of treatment, storage, and disposal facilities would be required to pay permit fees and be subjected to new standards including post-closure and financial responsibility requirements.

## States Proposed Legislation and Regulations

### California

**Packaging**—CA A. 2393 (Cortese) requires the California Integrated Waste Management Board to conduct a study of heavy metals in product packaging, and to report the results of the study to the Governor and the Legislature by January 1, 1995.

**Graffiti**—CA A. 3457 (Elder) includes graffiti abatement as a specified purpose of a community services district and

would enact procedures for the establishment of graffiti abatement districts with specified power for the purpose of abating graffiti. Authorizes districts to impose a special tax on real property for the purposes of abating graffiti and would authorize districts to levy a tax, pursuant to the existing authority granted to cities and counties.

CA S. 1642 (Watson) includes within the crime of vandalism the defacing of real or personal property with a felt tip marker or other marking substance. The aerosol tax provi-

sion was eliminated from the bill but provisions allowing municipalities to lock up or otherwise ban aerosols remain in the bill.

*Lead*—CA A. 3487 (Friedman) adds lead-related work, as defined, to the list of employments or places of employment that require the issuance of a permit. Requires the Division of Occupational Safety and Health to propose a regulation containing specified requirements relating to lead-related work to the Occupational Safety and Health Standards Board for its review and adoption.

*Chemical Diversion*—CA S. 1820 (Killea) provides that any manufacturer, wholesaler, retailer, or other person who sells, transfers, or otherwise furnishes any chemical reagent or solvent where the value exceeds \$100.00 or any specified chemical substance, with knowledge or reasonable cause to believe that the recipient will use the goods or chemical substance to unlawfully manufacture a controlled substance, is guilty of a misdemeanor.

*Labeling*—CA S. 428 (Ayala) requires any person, on and after July 1, 1993, who sells, in California, liquid hazardous material in a container that has a capacity of five gallons or more, to provide specified information to the purchaser.

*Hazardous Materials Transportation (Regulation)*—The Department of the California Highway Patrol has adopted rules extending the valid term of the temporary Hazardous Materials Transportation License (HMTL) from 30 to 60 days. For further information, contact Greg Biklian, Hazardous Materials Section, California Highway Patrol, P.O. Box 942898, Sacramento, CA 94298.

*Household Hazardous Waste (Regulation)*—The Integrated Waste Management Board has proposed to amend provisions implementing the Household Hazardous Waste Grant Program. For further information, contact Fernando Berton, Household Hazardous Waste Management Program, California Integrated Waste Management Board, 8800 Cal Center Drive, Sacramento, CA 95826, (916) 255-2348.

## Connecticut

*Lead*—CT H. 5287 (Dyson et al.) allows homeowners who have lead paint in their dwellings to receive a low interest loan for lead paint abatement.

*Health and Safety*—CT H. 5069 (Committee on Labor and Public Employment) promotes health and safety in places of employment in this State by requiring each employer of 11 or more employees to establish and administer a safety and health committee.

## Florida

FL H. 721 (Bronson) prohibits food carriers from transporting food items in a vehicle or rail car that has been used or is being used to transport solid waste or other hazardous waste.

## Georgia

*Packaging*—GA H. 124 (Dobbs et al.) relates to waste management, so as to provide for the regulation of toxic heavy metals in packaging waste.

## Idaho

*Aerosols*—S. 1274 (Committee on Judiciary) relates to possession of inhalants by minors; prohibits the possession of inhalants or aerosol spray cans by minors for the purposes of utilizing the contents to become intoxicated or be placed in an altered consciousness state; provides that minors illegally

possessing inhalants shall not come under the purview of the Youth Rehabilitation Act.

## Illinois

*Labeling*—IL H. 2925 (Matijevich) creates the American Content Labeling Act, which requires manufacturers to label manufactured products to be sold at retail with the percentage of manufacturing costs spent in the United States.

*Aerosols*—IL H. 3423 (Burke) provides that anyone selling aerosol paint in the city of Chicago would be guilty of a misdemeanor.

*Lead*—IL H. 1852 (Roman) creates the Lead Poisoning and Lead Abatement Act; requires blood testing for lead poisoning for certain individuals; provides for inspection of dwelling units for lead and remedial action to remove lead from dwelling units.

## Iowa

*Lead*—IA H. 2439 (Committee on Energy and Environmental Protection) relates to lead abatement by establishing a lead inspection and abatement program and provides a penalty.

*Underground Storage Tanks (Regulation)*—The Petroleum Underground Storage Tank Fund Board has adopted rules setting forth Board policy on payment assistance to owner/operators for system upgrades or replacements. The policy is designed to assist the owner/operators in meeting secondary containment requirements. For further information, contact Robb Hubbard, Iowa Comprehensive Petroleum Underground Storage Tank Fund Board, 200 East Grand, Suite 390, Des Moines, IA 50309.

## Kentucky

*Occupational Safety and Health*—KY H. 462 (Cyrus) strengthens the protection available to a worker who is wrongfully discharged for reporting an occupational safety or health violation, permits the Secretary of the State Labor Cabinet to order the immediate reinstatement of an employee pending a final decision by the Occupational Safety and Health Review Commission in instances when the Commissioner of Workplace Standards determines that the employer violated the employee's rights.

## Maine

*Lead*—ME H. 1584 (Mitchell et al.) amends laws governing lead poisoning control so as to make achievable the goal of eliminating lead poisoning of children in the state; establishes the Public Health Lead Poisoning Advisory Committee and outlines the duties and responsibilities of the Advisory Committee in counseling the Department of Human Services in rulemaking, administrative, and enforcement functions; defines what constitutes a hazard from exposed lead.

## Maryland

*Lead*—MD H. 1265 (Rosenberg and Thomas) establishes the Lead Paint Poisoning Prevention and Compensation Commission for purposes of funding medical rehabilitation for lead poisoning claimants. The bill initially contained language stating that the fund would be made up of monies collected from paint retailers who would have been taxed at a rate of between \$.50 to over \$1.00 per gallon. The paint retailing and manufacturing community launched a massive grassroots effort opposing the tax, resulting in the tax being deleted from the final proposal.



MD H. 1408 (Hergenroeder) requires that persons who conduct specified lead paint abatement work receive accreditation; establishes the manner by which persons may receive accreditation; requires that applicable training programs be approved by the Department of the Environment (DENV); requires that the DENV must set reasonable fees to cover specified costs; requires the DENV to adopt implemental rules and regulations; provides for injunctive relief and judicial review; sets civil and criminal penalties.

*Packaging*—MD H. 1346 (Perry et al.) prohibits, on or after July 1, 1993, a manufacturer or distributor from selling or offering for sale or for promotional purposes any package or packaging component or any product in a package or packaging component to which lead, cadmium, mercury, or hexavalent chromium was intentionally added during manufacture or distribution; establishes maximum allowable concentration levels for those toxics that are incidentally present in packaging materials.

MD H. 1367/S. 553 (Pitkin et al.) requires a manufacturer of rigid plastic containers sold, offered for sale, or used in association with the sale or offer for sale of a product in the state to ensure that the container meets established requirements; specifies exemptions; requires a manufacturer of those plastic containers to submit a certification to the Department of the Environment by October 1, 1993, and on or before March 1 each year thereafter; requires the Department to review those certifications and report to the General Assembly by July 1, 1992.

### Minnesota

*Lead*—MN S. 2496 (Johnson et al.) relates to Housing Finance Agency programs; allows certain loans made in part with Federal funds to exceed loan maximums for lead abatement requirements; deletes limit on funding for lease-purchase housing grants and loans; extends existing housing program to new housing if accessible where the borrower or a family member is disabled.

*Lead*—MN H. 1934 (Clark and Ogren) relates to lead abatement and education programs; reduces standard for elevated blood lead levels to 10 micrograms in children up to age six and pregnant women, and requires state program providing a proactive lead education program; includes cleaning-up and swab team services; requires Board of Health to provide health education to parents of children with certain levels of blood lead. The original funding mechanism for the bill was a \$.10 tax on each gallon of paint, but the paint industry lobbied heavily to get the tax deleted from the bill. The new funding will come from an increase in mortgage and deed taxes that are imposed when a residence is sold.

*Solvent Abuse*—MN S. 979 (Pappas) relates to glue and solvent sales to minors. Adds butane compounds to the list of restricted items which may not be sold to minors.

*Mercury*—MN S. 2042 (Dahl et al.) relates to recycling requirements; prohibits, as of August 1, 1992, the placement of mercury in solid waste or any processing or disposal facility; bars sales of mercury without a material safety data sheet and requires purchasers to sign statements that they will not place mercury in the solid waste stream; requires labeling of products containing mercury.

### New Jersey

*Lead*—NJ A. 729 (Kenny) establishes a presumption that lead toxicity in children under the age of seven is caused by lead paint.

### New York

*Packaging*—NY A. 2039 (Harenberg et al.) imposes a ban on the use of multi-material containers. Objective: to reduce solid waste.

NY A. 9245 (Hinchey et al.) enacts the Environmentally Sound Packaging Act; requires packaging to be reusable or recyclable; provides exceptions and makes related provisions.

*Lead*—NY A. 10416 (Clark) increases the penalty for the failure to comply with a notice and demand for the discontinuance of a paint condition conducive to lead poisoning to \$5,000.

*Graffiti*—NY A. 3620 (Weprin) authorizes the New York City Departments of Consumer Affairs, Sanitation, Environmental Protection, and Transportation and the New York City Police Department to issue summons for violations involving the sale of aerosol spray paint cans and broad tipped indelible markers.

NY A. 10496 (Seminario)/S. 7572 (Maltese) prohibits the making of graffiti and possession of graffiti instruments.

### Ohio

*Lead (Regulation)*—The Department of Human Services has proposed rules pertaining to the Healthcheck Program including early and periodic screening, diagnosis, and treatment objectives; definition of program terms; notice of program availability; community out-reach; follow-up of children with elevated blood lead levels. For further information, contact Beth Vogel, Department of Human Services, 30 E. Broad Street, 31st Floor, Columbus, OH 43266.

### Oregon

*Occupational Safety and Health (Regulation)*—The Occupational Safety and Health Division of the Oregon Occupational Safety and Health Agency has proposed rules to require employers to report serious injuries to OSHA within 24 hours; reducing the time period to report fatalities and catastrophes from 24 hours to 4 hours; to require the Agency to adopt revised Federal penalties. For further information, contact Sharon Dey, Standards and Technical Resources, Department of Insurance and Finance, Labor and Industries Building, Salem, OR 97310, (503) 378-3272.

### Pennsylvania

*Hazardous Waste*—PA H. 953 (D. Wright et al.) amends the Hazardous Sites Cleanup Act; provides for waste minimization, for incineration moratorium, for facility siting, and for host community review; further provides for hazardous waste facility siting team and for the host municipality benefit fee.

### Tennessee

*Air Quality*—TN H. 2591 (Kisber et al.) provides 100% tax credit for cost of pollution controls purchased by automobile body paint shops. Amends TCA 67-6-507.

### Texas

*Hazardous Waste (Regulation)*—Rules have been adopted authorizing the Texas Water Commission to establish an industrial solid waste and hazardous waste fee program and to implement fee assessments for the commercial and noncommercial management of hazardous wastes. For further information, contact Stephen Minick, Office of Budget, Planning and Evaluation, P.O. Box 13087, Austin, TX 78711, (512) 463-8069.

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## Loren W. Hill

Loren W. Hill received the B.S. Degree in Chemistry from North Dakota State University, Fargo, ND, in 1961, and the Ph.D. Degree from the Pennsylvania State University, University Park, PA, in 1965.

He became an Assistant Professor in Physical Chemistry at NDSU in 1965. His early research interests included kinetics of reactions catalyzed by surface oxygen groups on carbon blacks. Dr. Hill's teaching assignments included undergraduate physical chemistry courses and graduate courses in kinetics and thermodynamics. He was promoted to Associate Professor in 1968 and to full Professor in 1973. Also in 1973, Dr. Hill transferred from the Chemistry Department to the Department of Polymers and Coatings. His coatings research program included study of water absorption and desorption from coatings films and rheology of waterborne and high solids coating formulations. During this period, Dr. Hill worked closely with Dr. Zeno Wicks, Jr., the 1986 Mattiello Lecturer, and benefited from Dr. Wicks' vast industrial research experience.

While at NDSU, Dr. Hill administered many NSF Science Education grants and research grants from the coatings industry and from government agencies. One of the interesting grants concerned spacecraft sterilization research for NASA. Although Dr. Hill's career at NDSU emphasized his preferred areas of teaching and research, he served as Acting Chairman of the Polymers and Coatings Department from September 1971 to July 1972, and as Dean of the College of Science and Mathematics from February 1974 to June 1975. During the summers of 1977 through 1979, Dr. Hill participated in the Visiting Scientist Program at the Air Force Materials Laboratory, Wright-Patterson AFB, Ohio.

In 1980, Dr. Hill transferred from academia to industry when he accepted a position as Fellow in the Resins Division, Monsanto Chemical Company. In industry, Dr. Hill continued research in rheology of high solids coatings, but his interests broadened to include structure/property relationships of crosslinked coatings. He has used dynamic mechanical analysis (DMA) extensively to determine crosslink density and to support the design of resin structures to obtain optimized combinations of physical properties. Recent publications have shown that DMA is also very useful for following property changes of thermoset films during weathering. In 1987, Dr. Hill was promoted to Senior Fellow at Monsanto.

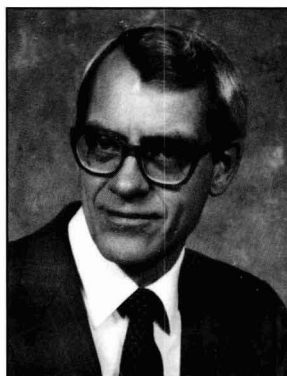
Through his research and publications, Dr. Hill has sought to support the pioneering work of Joseph J. Mattiello in expanding the application of basic sciences to solving problems in the decorative and protective coatings field.

Dr. Hill has been an invited lecturer at many seminars and conferences including: Gordon Research Conferences on coatings and films (1981) and on thermosets (1988), The International Conference in Organic Coatings Science and Technology (1986 and 1989), plenary lecturer at the Water-Borne and Higher Solids Coatings Symposium (1987), and several PMSE ACS Symposia. He has been author or co-author of approximately 35 research papers, including the first prize Roon Award-winning paper at the Federation's Annual Meeting in 1980. Dr. Hill is the author of the monograph "Mechanical Properties of Coatings," in the updated *Federation Series on Coatings Technology* (1987).

Despite his industrial position, Dr. Hill has continued teaching activities by participating in annual short courses for coating industry personnel at North Dakota State University and at Kent State University. He also serves on the Industry Advisory Committee of the Polymers and Coatings Department and holds an appointment as Adjunct Professor at NDSU.

Dr. Hill is a member of the Editorial Board of *Progress in Organic Coatings* and a former chairman of the Gordon Research Conference on Coatings and Films.

A member of the New England Society for Coatings Technology, Dr. Hill currently serves on the Federation's Publications Committee and Editorial Review Board of the *JOURNAL OF COATINGS TECHNOLOGY*. Former Federation activities include serving on the Coatings Industry Education Fund, Mattiello Memorial Lecture Selection Committee, and the Roon Awards Committee.



# Structure/Property Relationships Of Thermoset Coatings

Loren W. Hill  
Monsanto Chemical Company\*

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More than 50 years ago, Joseph J. Mattiello was a leader in the use of scientific principles to understand and improve decorative and protective coatings (see *Appendix 1*). In Mattiello's spirit of scientific innovation, progress is reviewed in establishment of relationships between structures of starting materials and physical properties of cured thermoset films. The effectiveness of coating scientists to adopt and adapt new concepts for use in coatings research is discussed. Under-utilized and remarkably simple methods for calculating crosslink density (XLD) of stoichiometric networks are presented. Use of dynamic mechanical analysis (DMA) and solvent induced swelling for determination of XLD is described. DMA plots are shown for many types of coatings.

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## INTRODUCTION

Basic understanding of how structure variations alter physical and chemical properties helps to transform coatings technology from an art to a science. Coatings chemists have been making this transition for many years. Of the previous 42 Mattiello Lectures, at least 10 have dealt with the relationships between structure and properties of coatings. Mark,<sup>1</sup> Billmeyer,<sup>2</sup> Hoy,<sup>3</sup> and Blank<sup>4</sup> focused on the polymeric portion. Van Loo,<sup>5</sup> Asbeck,<sup>6</sup> and Kumins<sup>7</sup> focused on interactions between polymer and pigment. Bobalek,<sup>8</sup> Wicks,<sup>9</sup> and Provder<sup>10</sup> focused on conversion of a liquid to a viscoelastic solid during cure of thermoset coatings.

The subject matter for this lecture was selected with three purposes in mind: to stimulate activity in certain areas that have been somewhat neglected by coatings

chemists; to demonstrate that crosslink density (XLD) concepts, determinations, and calculations are quite simple at least for stoichiometric systems; and to provide examples of how dynamic mechanical analysis (DMA) can be used with many types of thermoset coatings for understanding cure mechanisms and for confirming or denying that purposeful changes in resin structures have had the desired effects on network structure of cured films. The XLD section is intended to be useful to paint chemists who have not previously delved into network theory. Therefore, this section is similar to "Back to Basics" papers that are published in this journal.

## MECHANICAL PROPERTIES

### Under-Utilized Concepts

Coatings chemists have been slow to adopt and adapt certain mechanical property concepts from other polymer fields. These concepts include: stress concentration; brittle-ductile transition; viscoelasticity; and tertiary structure. Each of these could easily be the subject of the whole lecture, but here only the basic ideas and some related terminology will be given.

Stress concentration in a crack tip is a concept from the field of fracture mechanics.<sup>11</sup> Metallurgists came up with the field of fracture mechanics. Polymer chemists then adopted it and expanded it, and now it is time for coatings chemists to use it. What does the word "use" mean in this context? Usually it means understanding the concepts well enough to make structure changes that will prevent cracking during deformation. The changes should promote dissipation of mechanical energy in harmless ways within the fracture process zone. Then stress will not be highly concentrated at the crack tip. Crack growth will either not be initiated or, if initiated, will not be rapid. Note that we do not necessarily have to carry out fracture

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mechanics experiments in order to make use of the concepts.

Ductile materials can be drawn to considerable lengths without breaking.<sup>12</sup> There is a temperature below which failure will occur in a brittle mode and above which yielding will occur; that is, changes in the tertiary structure will occur, so that a ductile drawing will take place before breaking. This temperature is called the brittle-ductile transition temperature ( $T_b$ ).  $T_b$  lies below the glass transition temperature ( $T_g$ ) with which we coatings chemists are much more familiar. The  $T_b$  may be an important concept for post forming of coated metal as in coil coating technology.<sup>13,14</sup>

Most coatings chemists recognize that paint films are viscoelastic.<sup>15</sup> One aspect of this is the expectation of observing different properties at different rates of testing. We know that Gardner impact tests, performed at high rates, tell us something different from mandrel bend tests, which are performed at more moderate rates of stress application. Most of the time we recognize the need to match the rate of stress application in the test with the rate at which stress is applied to the coatings in a particular use. We tend to forget about the viscoelastic character of our coatings, however, when it comes to specifying the  $T_g$ . We want to say that a film has a particular  $T_g$  value whereas viscoelasticity concepts tell us that  $T_g$  is not a fixed number for a given material but rather depends on rate. This rate is the rate of deformation in a mechanical  $T_g$  determination or rate of heating during the more traditional specific volume versus temperature method. Thus, each quoted  $T_g$  value should have an associated rate. Without the associated rate, the  $T_g$  value doesn't mean much.

The term "tertiary structure" has been borrowed from the biochemists.<sup>16</sup> Primary structure includes atom to atom connections and the empirical formula; that is, the numbers of each atom in a compound. Secondary structure gives the relative positions of atoms within individual molecules, and in this context, we refer to conformations of molecules. We could use the term "tertiary structure" to describe how molecules pack together. A term that some may find synonymous is "polymer morphology." The term "morphology" is often used when material separates into more than one phase whereas tertiary structure applies to molecular packing within a single phase. We cannot explain physical property variations based on primary and secondary structures alone. Here we seem to have a shortage of ideas which may relate to having a shortage of words. For the most part, we use the words amorphous or crystalline and often partially crystalline. Does the latter mean that crystalline regions are totally crystalline and the amorphous regions totally amorphous? Another possible interpretation is that the randomness of amorphous material is not total. Perhaps tendency to randomness comes in a wide range of degrees. Would you call a slightly non-random amorphous material partially crystalline? In any case, almost all polymeric materials including thermoset materials become more dense with time after cooling from above their  $T_g$ . This densification, also called physical aging or volume relaxation or free volume change, causes changes in

physical properties, especially in impact resistance.<sup>17</sup> Examples of physical aging of coatings have been reported for polyester polyols crosslinked with melamine formaldehyde resins<sup>18</sup> and for very highly crosslinked epoxy/amine systems.<sup>19</sup>

## Test Methods

Basic property studies, of course, cannot be expected to replace paint test methods used widely in our industry.<sup>14,20</sup> We need both. Committee D-1 of the American Society for Testing and Materials (ASTM) has broad responsibility for paints and applied coatings. Twenty-nine test methods are given under the category "Physical Strengths and Resistances." With some force fitting, such as placing tensile properties of organic materials into the subcategory of flexibility, these tests break down as follows: six abrasion resistance tests, four adhesion tests, four tests of blocking resistance, eight flexibility tests, three hardness tests, and four tests of impact resistance.<sup>20</sup> Some of these tests differ with respect to rate of stress application. Some are specific to a particular type of coating. It is not always possible to use an ASTM test, but whenever it is possible, I appeal to you to use them. Consistency in test methods facilitates communication among paint chemists, which Mattiello considered to be so important. Uniformity in use of methods is essential as we seek to be more scientific and less dependent on art. I am sure we all owe a hearty "Thank You" to the coatings chemists and engineers who worked on development of these and other test methods. Mattiello was very active in ASTM.<sup>21</sup>

## CROSSLINK DENSITY (XLD)

### Definitions and Terminology

In physical property considerations, the two most important characteristics that can be controlled are  $T_g$  and XLD. Tertiary structure may be of equal importance, but little progress has been made in controlling it. Values of  $T_g$  are determined quite frequently by coatings chemists using methods such as differential scanning calorimetry (DSC) and thermomechanical analysis (TMA).<sup>10</sup> In contrast, XLD is seldom determined by coatings chemists. One consequence of infrequent experimental determination is a rather fuzzy definition of XLD in the minds of many coatings chemists. Ambiguity could be avoided by broad acceptance of the definition:

$$\nu_e = \frac{\text{the number of moles of elastically effective network chains}}{\text{per cubic centimeter of sample}} \quad (1)$$

The symbol,  $\nu_e$ , is firmly associated with this definition in network theory.<sup>22</sup> From the definition, it is evident that units of  $\nu_e$  are moles/cm<sup>3</sup>.

For the purpose of distinguishing between effective and ineffective network chains, it is useful to represent an ideal network as in *Figure 1* and a non-ideal network as in *Figure 2*. In the ideal network, all of the chains have reacted on both ends, and in every case the ends are attached to different junction points. In the non-ideal network, some of the chains have not reacted at all. This material, which is not connected to the network, is called

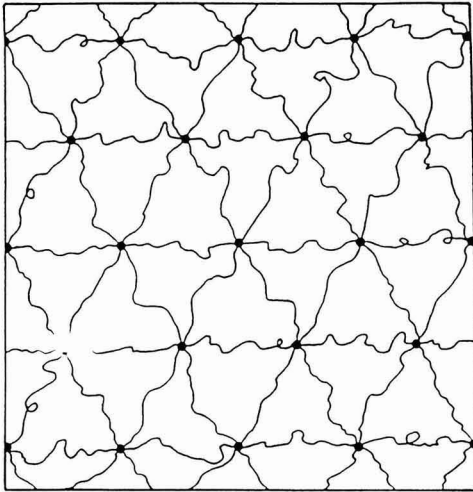
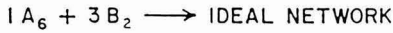


Figure 1—A cartoon representing an ideal network in which all junction points are hexafunctional and all chains are elastically effective

“sol.” Some of the chains in the non-ideal network have reacted on one end but not on the other. These chains are called “dangling ends.” Some chains in the non-ideal network have both ends reacted on the same junction point. These chains are called “loops.” Sol, dangling ends, and loops contribute to sample volume but do not count as effective chains. Therefore, these structural features reduce the value of  $\nu_e$  according to definition (1).

Although the ideal and non-ideal network representations depicted in Figures 1 and 2 are useful, these figures should not be considered to represent actual structures. The tertiary structure of networks is not known. Certainly the actual structure will be more three-dimensional. For addition copolymers, which depend on more or less random placement of functional monomer units along the chain, the chain ends are likely to be nonfunctional. These ends are destined to be dangling even at complete conversion.

For most coatings chemists, it is easier to visualize a network based on the value of  $M_c$ , which is defined as:

$$M_c = \text{weight of sample in grams which contains one mole of elastically effective network chains} \quad (2)$$

Other terms that have been used to define  $M_c$  include “number average molecular weight of effective network chains” and “molecular weight per branch point.” The latter is problematic because there is controversy in the literature about how to count branch points.<sup>23,24</sup> Definition (2) avoids the controversy. From definition (2), it is evident that units of  $M_c$  are g/mole.

Consideration of definitions (1) and (2) indicates that the relationship between  $\nu_e$  and  $M_c$  is:

$$M_c = \frac{\rho}{\nu_e} \quad (3)$$

where  $\rho$  is regular old density in g/cm<sup>3</sup>. A highly cross-

## NON-IDEAL NETWORK

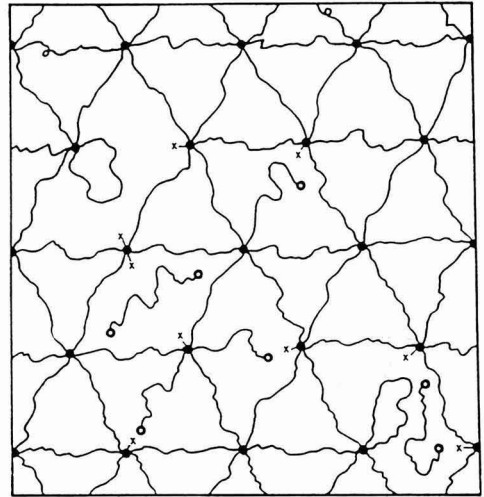


Figure 2—A cartoon representing a non-ideal network which contains unconverted reactant (sol), dangling ends, and loops

linked thermoset coating has a  $\nu_e$  value of about  $1 \times 10^{-3}$  moles/cm<sup>3</sup>. Therefore, since most clear films have a density only moderately greater than 1.0 g/cm<sup>3</sup>, the corresponding value of  $M_c$  is a little less than 1,000 g/mole. Looser networks have higher  $M_c$  values, and tighter networks have lower  $M_c$  values.

## Experimental Determination of XLD

The two most widely used methods for determining XLD are modulus measurements carried out at temperatures well above  $T_g$ , and solvent induced swelling.<sup>25</sup> We have used DMA extensively for modulus measurements.<sup>26-29</sup> With DMA, modulus measurements can be made in a relatively short time over a wide temperature range because the method is nondestructive. Other modulus methods, such as stress-strain determinations, result in breaking the sample at each temperature. It is very arduous to carry out stress-strain tests over a wide temperature range. Determination of XLD by solvent induced swelling works best for network samples that have a very low content of extractable material.<sup>30</sup> Most films of interest in our studies, including those described in the following, had very low levels of extractable material.

Figure 3 shows what is done in a DMA experiment.<sup>31,32</sup> The sample is subjected to an oscillating strain of maximum amplitude  $\epsilon_0$ . The instrument determines the resulting stress of maximum amplitude,  $\sigma_0$ . All dynamic testing involves an oscillatory deformation of this type, but with our instrument the deformation is a direct tensile deformation on a strip of free film. The value of  $\epsilon_0$  is a small fraction of the sample length so the sample does not break or yield. This makes the test nondestructive. The value of  $\delta$ , called the phase lag, is a rather direct indication of viscoelasticity. For an ideal elastic solid

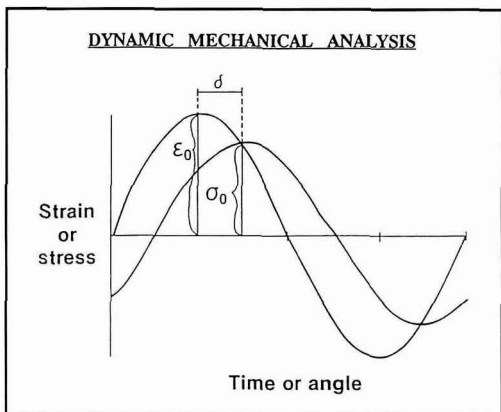


Figure 3—Sine curves for stress and strain in a dynamic mechanical analysis experiment indicating maximum imposed strain ( $\epsilon_0$ ), maximum stress ( $\sigma_0$ ), and the phase lag ( $\delta$ )

$\delta = 0^\circ$ , and for an ideal Newtonian liquid  $\delta = 90^\circ$ . As expected from viscoelasticity concepts,  $\delta$  depends on the rate of oscillatory deformation. The value of  $\delta$  is smaller at faster rates of oscillation. Usually we used 11 Hz, i.e., 11 cycles per sec.

Dynamic properties are defined as:

$$\text{Storage modulus} = E' = \frac{\sigma_0 \cos \delta}{\epsilon_0} \quad (4)$$

$$\text{Loss modulus} = E'' = \frac{\sigma_0 \sin \delta}{\epsilon_0} \quad (5)$$

$$\text{Loss tangent} = \frac{E''}{E'} = \tan \delta \quad (6)$$

where  $\epsilon_0$  is the maximum oscillatory strain,  $\sigma_0$  is the resulting maximum oscillatory stress, and  $\delta$  is the phase lag.<sup>31,32</sup> The storage modulus is an indication of elastic response. The loss modulus is an indication of viscous response. The loss tangent is also related to viscous response, but in this case expressed relative to elastic response. These dynamic properties were determined at 1 min intervals during scans run at 1°C/min or more recently at 2°C/min. DMA was carried out on an Autovibron® instrument (Imass, Inc.). The Autovibron is an automated, computer controlled modification of the Rheovibron® model DDV-II-C (Toyo Baldwin Co.). The advantages and methods of automation have been described.<sup>33</sup>

The relationship between the moduli of definitions (4) and (5) and tensile modulus,  $E$ , as obtained in stress-strain determinations is:<sup>31,32</sup>

$$E^2 = E'^2 + E''^2 \quad (7)$$

However, for most network polymers that are useful as coatings,  $E''$  is much smaller than  $E'$  except in the glass transition region. Therefore, at temperatures that differ significantly from  $T_g$ ,

$$E = E' \quad (\text{at } T \neq T_g) \quad (8)$$

A typical DMA plot is shown in Figure 4 for a clear-coat film prepared from an acrylic polyol and a high

solids methylated melamine formaldehyde crosslinker. Plots are labeled according to definitions (4), (5), and (6) in Figure 4A. The storage modulus level at the left is typical of amorphous, unpigmented films in the glassy state. Modulus values in the glassy state are insensitive to XLD. The middle portion of the plot is the glass transition region where  $E'$  drops sharply and both  $E''$  and  $\tan \delta$  go through a maximum. The nearly flat part of the  $E'$  plot at temperatures well above  $T_g$  is called the rubbery plateau. The same data are repeated in Figure 4B, but certain points, which we refer to as measures of cure,<sup>27</sup> have been indicated. In this work,  $T_g$  is taken as the temperature of the maximum in the  $\tan \delta$  plot. The height of the maximum is also sensitive to cure conditions. The minimum value of  $E'$  in the rubbery plateau is used to calculate XLD.<sup>22,26</sup> In many cure studies, it has been noted that as cure temperature or cure time is increased,  $T_g$  increases,  $\tan \delta$  (max) decreases, and  $E'$  (min) increases.<sup>26-29</sup>

It is proposed that the most simple form of the XLD equation from kinetic theory of rubber elasticity,<sup>22</sup> equation (9), be designated as the "ideal network law"

$$G = \nu_c RT \quad \text{or} \quad \nu_c = \frac{G}{RT} \quad (T \gg T_g) \quad (9)$$

where  $G$  is shear modulus,  $R$  is the gas constant, and  $T$  is temperature in °K. An analogy is intentionally implied to

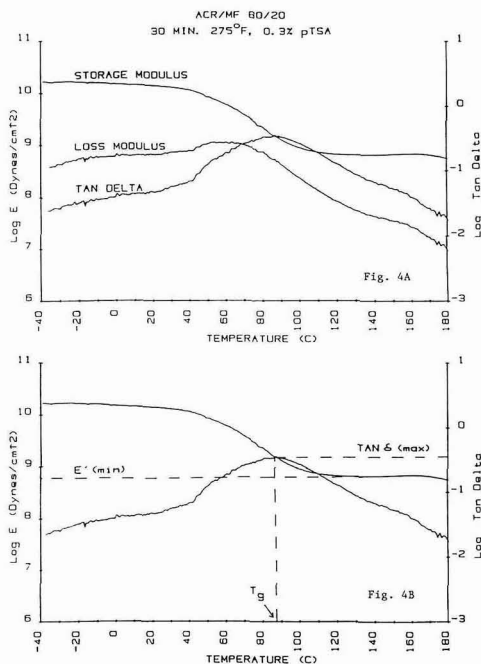


Figure 4—DMA plots for a highly crosslinked film prepared from an acrylic polyol (ACR) and an etherified melamine formaldehyde resin (MF): (A) Dynamic properties as defined in equations (4), (5), and (6); and (B) Identification of measures of cure

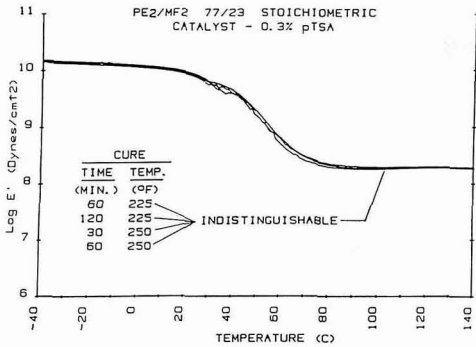


Figure 5—Storage modulus ( $E'$ ) plots for four stoichiometric films that were cured under various conditions

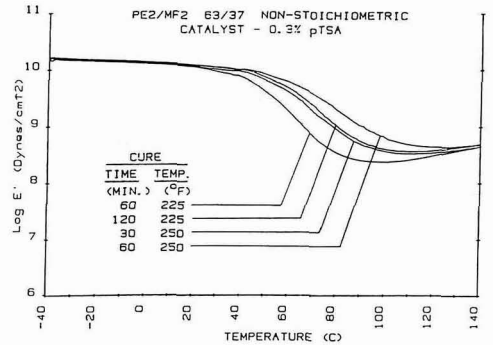


Figure 6—Storage modulus ( $E'$ ) plots for four non-stoichiometric films that were cured under various conditions

the ideal gas law. (See Appendix 2). For equation (9) to be valid,  $G$  must be determined at a temperature,  $T$ , that is in the rubbery plateau region. The instrument used in our work provided  $E'$ , not  $G$ . However, by using  $G \cong E/3$ , which is a very good approximation for small strains, and equation (8), one can convert equation (9) to

$$v_e = \frac{E'}{3RT} \quad (10)$$

Equation (10) was used to calculate  $v_e$  from experimental values of  $E'$  and  $T$ . The computer printout corresponding to Figure 4 indicated that  $E'(\text{min}) = 2.57 \times 10^8$  dynes/cm<sup>2</sup> at 135°C (408°K). Thus,  $v_e$  by equation (10) is  $6.13 \times 10^{-3}$  moles/cm<sup>3</sup>. From equation (3) and an experimental density of 1.12 g/cm<sup>3</sup>,  $M_c$  is 183 g/mole. This value indicates a very tight network.

The equation used to calculate XLD from swelling was taken from Flory's classic polymer text<sup>30</sup>

$$v_e = \frac{v_e'}{V_o} = \frac{-[\ln(1-v_2) + v_2 + \chi_1 v_2^2]/\bar{v}_1}{(v_2^{1/3} - v_2/2)} \quad (11)$$

where  $v_e'$  is the number of moles of elastically effective network chains in volume  $V_o$  of unswollen film;  $\bar{v}_1$  is the molar volume of solvent;  $\chi_1$  is the Flory-Huggins interaction parameter of solvent with network polymer; and  $v_2$  is the volume fraction of polymer in the swollen film at equilibrium swelling. Methylene chloride was used as the swelling solvent ( $\bar{v}_1 = 63.6$  cm<sup>3</sup>/mole at 25°C). Based on published values of  $\chi_1$  for various polymers in chlorinated solvents,<sup>34</sup> the value of  $\chi_1$  was estimated to be in the range of 0.4 to 0.5 for melamine cured acrylic polyol films. The only experimental value required in equation (11) was  $v_2$ . Values of  $v_2$  were determined on small, square (1 to 2 mm edge length) samples of the same free films that were used for DMA. The sample was placed on a microscope slide and covered with a thin glass coverslip. A droplet of methylene chloride from an eye dropper was contacted with the edge of the coverslip. The methylene chloride rapidly wet the glass surfaces and was drawn into the narrow gap between them. For several seconds after contact between the solvent and sample, no change in size was evident, but then in a fraction of a second the sample was observed to swell to a constant

increased size, which we took to be characteristic equilibrium swelling. Use of small samples facilitated rapid attainment of equilibrium. Swelling was observed using a microscope fitted with a TV camera. Edge length of a magnified image on a TV monitor was measured before ( $x_1$ ) and after ( $x_2$ ) swelling. Swelling was expressed as fractional increase in edge length,  $f$ , where  $f = (x_2 - x_1)/x_1$ . Geometric considerations and an assumption of isometric swelling result in

$$v_2 = 1/(1+f)^3 \quad (12)$$

Equations (11) and (12) were used to obtain  $v_e$  from swelling experiments on the sample of Figure 4. The experimental value of  $f$  was 0.16 (16% swelling). By equation (12), therefore, the value of  $v_2$  was 0.641. This value of  $v_2$  and the molar volume of methylene chloride were used in equation (11). Values of  $\chi_1$  of 0.40 or 0.50 were also inserted, giving  $v_e$  values  $6.34 \times 10^{-3}$  or  $5.14 \times 10^{-3}$  moles/cm<sup>3</sup>, respectively. This range brackets the value of  $6.13 \times 10^{-3}$  moles/cm<sup>3</sup> obtained from DMA.

### Model Network Formation

For calculation of XLD from structure, it is advantageous to study a simple reaction such as



As reported previously,<sup>26</sup> this "ideal" reaction can be approximated by using hexamethoxymethyl melamine (HMMM) for  $A_6$  and a commercial telechelic oligoester diol of uniform structure<sup>35</sup> for  $B_2$ . The experimental equivalent weights of  $A_6$  and  $B_2$  are 72 g/eq. and 244 g/eq.

Films were prepared using four cure conditions from a stoichiometric formulation  $B_2/A_6 = 77/23$  (by weight) and from a nonstoichiometric formulation,  $B_2/A_6 = 63/37$  (by weight). Storage modulus plots for the four stoichiometric films are superimposed in Figure 5. Variations in cure time and cure temperature have almost no effect on storage modulus. Storage modulus plots for the four nonstoichiometric films are superimposed in Figure 6. In this case, variations in cure time and cure temperature cause large differences in storage modulus plots. Absence of cure-condition effects (Figure 5) suggests that network



Table 1—Crosslink Density from Swelling and DMA-Stoichiometric, PE2/MF2 77/23

Cure		Swelling		DMA		
Time (min)	Temp (°F)	Percent <sup>a</sup> (%)	$\nu_e^b$ (mol/cm <sup>3</sup> )	E' at 100°C (dyns/cm <sup>2</sup> )	$\nu_e^c$ (mol/cm <sup>3</sup> )	M <sub>c</sub> <sup>d</sup> (g/mol)
60	225	25.5	$2.0 \times 10^{-3}$	$1.8 \times 10^8$	$1.9 \times 10^{-3}$	590
120	225	24.3	$2.3 \times 10^{-3}$	$1.9 \times 10^8$	$2.0 \times 10^{-3}$	560
30	250	25.1	$2.2 \times 10^{-3}$	$1.9 \times 10^8$	$2.0 \times 10^{-3}$	560
60	250	24.6	$2.3 \times 10^{-3}$	$2.0 \times 10^8$	$2.2 \times 10^{-3}$	510

(a) Change in edge length.

(b) From equation (11) with  $\chi_1 = 0.50$ .

(c) From equation (10) at 373°K.

(d) From equation (3) with  $\rho = 1.12 \text{ g/cm}^3$ .

forming reactions have gone to completion and that the desired formation of an "ideal network" may have been approximated. Observation of cure condition effects with HMMM in excess (Figure 6) suggests that network forming reactions have not gone to completion. The incomplete reaction is thought to be self-condensation of excess A<sub>6</sub>.

The XLDs for the stoichiometric films are given in Table 1. E' values at 100°C from Figure 5 were used to obtain the  $\nu_e$  results in the column headed DMA. The  $\nu_e$  values are nearly the same for all cure conditions. Values of M<sub>c</sub> in Table 1 were calculated from DMA data by

equation (3) using an experimental density of 1.12 g/cm<sup>3</sup>. The  $\nu_e$  values from swelling experiments are also given in Table 1. The fractional increases in edge length for the four samples were nearly the same,  $f = 0.24$  to 0.26. The agreement between swelling and DMA values of  $\nu_e$  is satisfactory. Selection of a slightly higher  $\chi$  value would improve this agreement.

For an ideal network, the theoretical  $\nu_e$  value can be calculated directly from definition (1). Consideration of the chemical equation and of Figure 1 indicates that in the ideal case all B<sub>2</sub> molecules form elastically effective chains. Therefore, the coefficient of 3 in the balanced equation can be placed in the numerator if the volume of 1 mole of A<sub>6</sub> plus 3 moles of B<sub>2</sub> is placed in the denominator for calculation of  $\nu_e$ . The weight of the network is  $[390 + 3(488) - 6(32)] = 1662 \text{ g}$ , where the first two figures in the bracket are based on molecular weights of A<sub>6</sub> and B<sub>2</sub> and the third figure corrects for methanol loss,  $m. wt = 32$ . For an experimental density of 1.12 g/cm<sup>3</sup>, the volume of network is 1484 cm<sup>3</sup>. Thus, the theoretical value of  $\nu_e$  from definition (1) is  $3/1484 = 2.0 \times 10^{-3} \text{ moles/cm}^3$ . The theoretical value agrees satisfactorily with both sets of experimental values. This combination of observations has given us confidence in using DMA routinely for determination of XLD of thermoset films. The DMA method is preferred in day-to-day work because it is more convenient than swelling experiments and because uncertainties and variations in  $\chi$  values detract from the feasibility of using swelling experiments routinely.

The XLD for the nonstoichiometric films are given in Table 2. E' values at 100°C from Figure 6 were used to obtain the  $\nu_e$  results in the column headed DMA. In this case,  $\nu_e$  values vary considerably for different cure conditions with 60 min at 225°F giving the lowest XLD, and 60 min at 250°F the highest XLD. The  $\nu_e$  values from swelling experiments are also given in Table 2. The fractional increases in edge length for the four samples ranged from  $f = 0.18$  to  $f = 0.24$ . The agreement between swelling and DMA values of  $\nu_e$  is satisfactory. Selection of a slightly lower  $\chi$  value would improve this agreement.

The higher XLD of films of Table 2, compared to those of Table 1, is attributed to crosslinks created by self-condensation of excess A<sub>6</sub>. The XLD with excess A<sub>6</sub> is only moderately higher at the mildest cure condition than

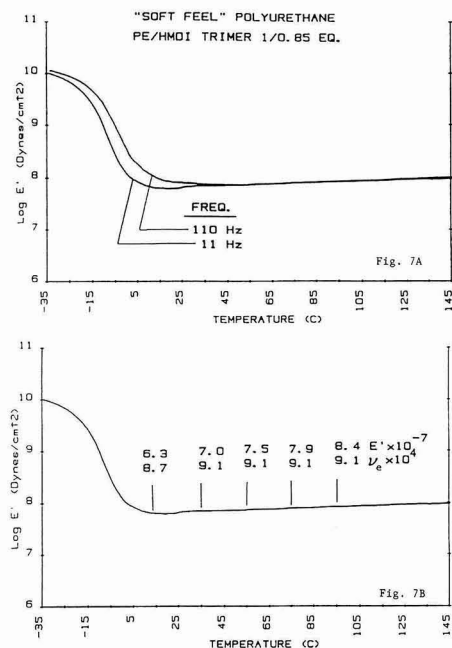


Figure 7—Storage modulus (E') plots for a two-package polyurethane film prepared from a polyester polyol (PE) and a trimer of hexamethylene diisocyanate (HMDI): (A) Effect of oscillating frequency; and (B) Experimental E' values and the corresponding values of  $\nu_e$  calculated from equation (10)

observed for the stoichiometric films. Thus, under these mild cure conditions, self-condensation must occur only to a moderate extent. In contrast, with 60 min cure at 250°F the proposed self-condensation has approximately doubled the XLD relative to that of the correspondingly cured stoichiometric film. The rise in  $E'$  plots at high temperatures for nonstoichiometric films (Figure 6) is attributed, in part, to additional self-condensation during the DMA scan. At a temperature of 140°C, the differences associated with cure schedule variations have been eliminated by additional curing during the scan; that is, the plots all come together at 140°C.

### Frequency Variation and Constancy of $E'/T$ Ratio

Further tests of equation (10) were carried out using polyurethane films that had quite low  $T_g$  values but relatively high XLD. Films having these characteristics feel like soft leather when properly pigmented. The effects of frequency change during oscillation are shown in Figure 7A. Frequencies of 11 and 110 Hz give different  $E'$  values in the transition region as expected from viscoelasticity concepts. However, in the rubbery plateau region, values of  $E'$  are exactly the same. When equation (10) is used to calculate XLD, it is evident that data obtained at either frequency give the same results. In some cases, usually with films of higher  $T_g$  or broader transitions,  $E'$  values in the rubbery plateau decrease with decreasing frequency. Extrapolation to low frequencies has been used to obtain  $E'$  values for use in equation (10) or equation (9) in some coatings studies.<sup>36</sup>

Equation (10) is not restricted with regard to the ( $E'$ ,  $T$ ) data point used for  $\nu_e$  calculation as long as this point is in the rubbery plateau. Since there are many ( $E'$ ,  $T$ ) points but only one  $\nu_e$  for a given film, equation (10) requires that all points give the same XLD result. This requirement is tested in Figure 7B.  $E'$  values at four temperatures between 35 and 95°C are noted and the  $\nu_e$  value calculated from each is also noted.  $E'$  increases from  $7.0 \times 10^7$  to  $8.4 \times 10^7$  dynes/cm<sup>2</sup>, but in calculating  $\nu_e$ , the temperature change compensates exactly so that all four points give the same XLD. This is a rather stringent and successful test of the ideal network law. A slightly lower  $\nu_e$  value is obtained at 15°C which may result from small amounts of absorbed water which is driven out of the film during later stages of the scan.

A gentle rise in  $E'$  through the rubbery plateau, as shown in Figure 7B, is expected for constant  $E'/T$  ratio and constant calculated  $\nu_e$ . If  $E'$  rises more sharply, additional cure during the scan should be suspected. If  $E'$  drops in the rubbery plateau, the cause could be thermal degradation or melting of poorly organized partially crystalline domains. This last point has not been adequately studied.

### Calculation of XLD from Structure

For stoichiometric networks obtained from a simple mixture of reactants, such as  $1A_6 + 3B_2$ , calculation of  $\nu_e$  from the definition is rather direct as previously illustrated. However, if the reactant mixture contains molecules of several different functionalities exceeding two, calculation from the definition becomes difficult. A more general approach is provided by the Scanlon equation<sup>37</sup>

$$\nu_e = \frac{3}{2}C_3 + \frac{4}{2}C_4 + \frac{5}{2}C_5 + \dots = \frac{\sum_{f=3}^{\infty} f}{2}C_f \quad (13)$$

where  $C_f$  is the concentration of polyfunctional reactants expressed in units of moles per cubic centimeter of final cured film. This equation has been under-utilized by coatings chemists perhaps because units of  $C_f$  were not carefully delineated in early publications.  $C_f$  values for various multifunctional reactants are calculated by placing their coefficient from the balanced network forming reaction in the numerator and the volume of the final film formed from the balanced equation in the denominator. Knowledge of this volume requires an experimental density (or an estimated one) and a correction for cure volatile losses if any. For the  $1A_6 + 3B_2$  example considered previously, equation (13) contains only one term, and the  $C_6$  value of this term is (1 mole ÷ 1484 cm<sup>3</sup> =  $6.74 \times 10^{-4}$  moles/cm<sup>3</sup>). Multiplication by 6/2, as indicated in equation (13), gives  $\nu_e = 2.0 \times 10^{-3}$  moles/cm<sup>3</sup> in agreement with calculation directly from the definition. Equation (13) does not include a term for difunctional reactants, such as  $B_2$ , because such reactants cannot create junction points in a network; they can only extend the network chains.

When the concentration units are clearly understood, the logic of equation (13) is apparent.<sup>37</sup> The sum  $3C_3 + 4C_4 + 5C_5 + \dots$  is the number of moles of chain ends coming into all junction points in one cubic centimeter of

Table 2—Crosslink Density from Swelling and DMA-Nonstoichiometric, PE2/MF2 63/37

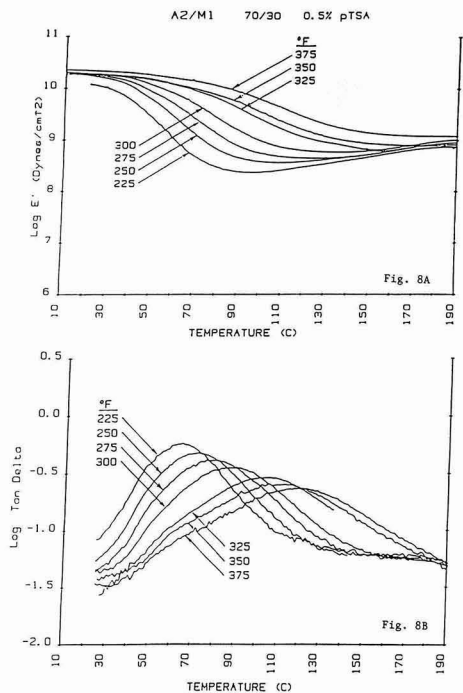
Cure		Swelling		DMA		
Time	Temp	Percent <sup>a</sup>	$\nu_e^b$	$E'$ at 100°C	$\nu_e^c$	$M_c^d$
(min)	(°F)	(%)	(mol/cm <sup>3</sup> )	(dyns/cm <sup>2</sup> )	(mol/cm <sup>3</sup> )	(g/mol)
60	225	24.4	$2.0 \times 10^{-3}$	$2.1 \times 10^8$	$2.6 \times 10^{-3}$	430
120	225	20.4	$3.3 \times 10^{-3}$	$3.1 \times 10^8$	$3.9 \times 10^{-3}$	290
30	250	21.2	$3.1 \times 10^{-3}$	$2.9 \times 10^8$	$3.5 \times 10^{-3}$	320
60	250	18.2	$4.3 \times 10^{-3}$	$4.0 \times 10^8$	$4.6 \times 10^{-3}$	240

(a) Change in edge length.

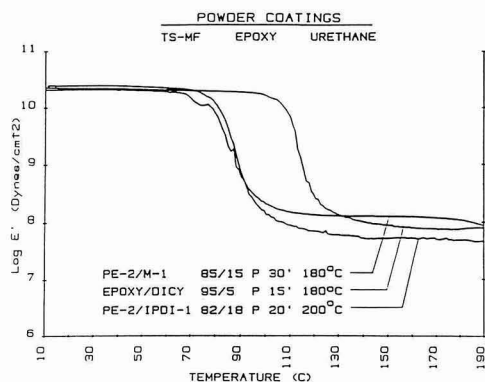
(b) From equation (11) with  $\chi_1 = 0.50$ .

(c) From equation (10) at 373°K.

(d) From equation (3) with  $\rho = 1.12$  g/cm<sup>3</sup>.



**Figure 8—Effect of cure temperature variation with a 30 min cure and para-toluene sulfonic acid (pTSA) as catalyst for a film prepared from an acrylic polyol (A2) and an etherified melamine formaldehyde resin (M1): (A) Storage modulus; and (B) Loss tangent**



**Figure 9—Storage modulus ( $E'$ ) overlay plots for powder coatings prepared from a polyester polyol (PE-2) crosslinked with either a toluene sulfonamide modified melamine resin (TS-MF = M-1) or a blocked adduct of isophorone diisocyanate and trimethylol propane (IPDI-1) and for a bisphenol A epoxy resin crosslinked with a commercial dicyandiamide (DICY) crosslinker**

network. By definition of a "chain," there must be two ends. Thus, the number of moles of chains in the network must be one half of the number of moles of chain ends coming into all junction points. The chain end versus chain argument gives rise to the divisor of two in each term of equation (13).

Although the Scanlon equation is very convenient for stoichiometric reactions and complete conversion, it does not apply to other cases. For nonstoichiometric mixtures and/or incomplete conversion, the equations of Miller and Macosko<sup>38</sup> can be used. These equations are very general. If the Miller and Macosko equations are applied to the stoichiometric case with complete conversion, the resulting XLD is exactly the same as that obtained from the Scanlon equation. Other equations for  $\nu_e$  have been proposed, as discussed by Yeo et al.<sup>23</sup> and by Graessley,<sup>24</sup> but the other equations do not give the same results as the Miller and Macosko treatment. Bauer<sup>39</sup> has given examples of applications of the Miller and Macosko method to coatings systems and has provided a computer program. Since Miller and Macosko consider many other post gel properties in addition to XLD, the equations related to XLD are somewhat difficult to pick out of the 60 equations presented.<sup>38</sup> Therefore, the Bauer paper is very useful.

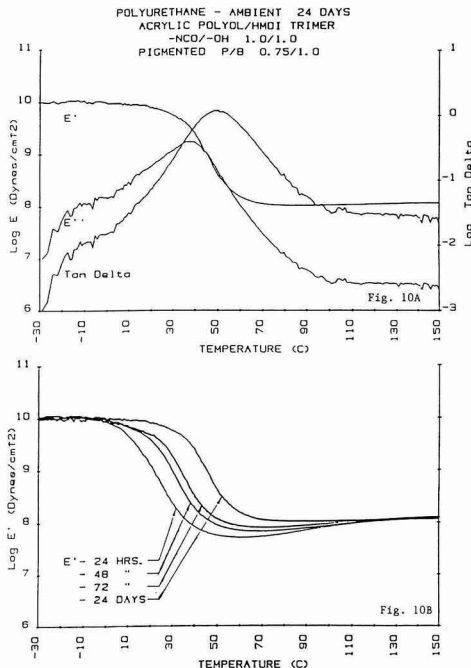
## EXAMPLES OF USE OF DMA IN STRUCTURE/PROPERTY STUDIES

### Self-Condensation vs Co-condensation<sup>27</sup>

During the approximately 50 years of use of etherified melamine formaldehyde crosslinkers (MF), a popular question has always been how much self-condensation of MF takes place along with co-condensation between MF and its polyol coreactants. Figure 8 shows examples of cure study with an acrylic polyol (A2 - OH eq. wt 400 g/eq.) and a commercial high solids methylated MF resin of the HMMM type (M1, 100% active, eq. wt of ~ 76 g/eq.). The usual cure schedule with 0.5% para-toluene sulfonic acid (pTSA) on total resin solids (TRS) is 30 min at 250°F, but cure temperatures both lower and much higher are included in this film series.

Storage modulus ( $E'$ ) plots are superimposed in Figure 8A. Although cure temperature increments are all the same (25°F), the results tend to group. The four curves for 225 to 300°F are similar to one another. The three curves from 325 to 375°F are similar to one another but different from the other group. Results suggest that for the lower group co-condensation forms most of the crosslinks and self-condensation contributes moderately. At the higher temperatures it is believed that co-condensation is complete and that continued dependence of properties on cure temperature is caused by increasing amounts of self-condensation.

Plots of  $\tan \delta$  are superimposed in Figure 8B. All of the transitions are quite broad. The ones at 325°F and above are noticeably broader than the others. We attribute this additional broadness to significant amounts of self-condensation. The  $T_g$  values taken from these maxima increase smoothly up to about 300°F. Then there is an abrupt jump followed by another smooth curve of differ-



**Figure 10**—DMA plots for an ambient cure two-package polyurethane prepared from an acrylic polyol and the biuret trimer of hexamethylene diisocyanate (HMDI). (A) Dynamic properties after 24 days; and (B) E' plots showing continued cure over several days

ent shape. These co- versus self-condensation effects depend on catalyst type and level, and steric considerations for the  $-OH$  group on the coreactant. The balance of co- and self-condensation also depends strongly on the pattern of substitution on the MF resin.<sup>27</sup>

### Crosslinkers for Powder Coatings<sup>29</sup>

Storage modulus plots for three-pigmented powder coating films are overlaid in *Figure 9*. The same polyester resin (PE-2,  $-OH$  eq. wt  $\sim 1425$  g/eq.,  $T_g \sim 54^\circ C$ ) was crosslinked with a toluene sulfonamide modified MF resin (TS-MF) and a commercial blocked trifunctional adduct prepared from trimethylol propane and isophorone diisocyanate (IPDI-1). TS modification of MF resins increases their  $T_g$  values, which helps avoid sintering of fully formulated and melt-mixed powders.<sup>29</sup> In *Figure 9*, the two polyester powder coating films are compared with a typical epoxy powder coating film crosslinked with a commercial dicyandiamide crosslinker (DICY).

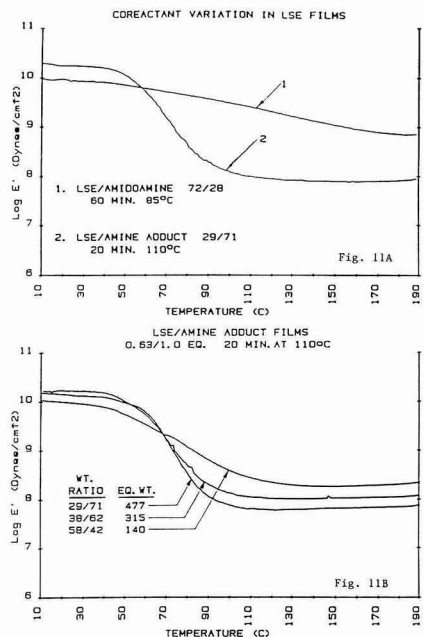
The two polyester films have very similar  $T_g$  values, which indicates that  $T_g$  is determined mainly by PE-2 with only moderate effects on  $T_g$  from the crosslinkers at the levels used here. TS-MF was expected to produce higher XLD based on its higher functionality, relative to IPDI-1, and results in *Figure 9* confirm that it does. The epoxy/DICY system has a much higher  $T_g$ , characteristic

of the BPA epoxy resin, and an XLD intermediate between those of the other two films.

Most powder coatings that we have studied have very flat modulus plots at quite high levels in the glassy region as shown in *Figure 9*. This may be related to tertiary structure, but we do not know if it should be called partial crystallinity. Once the transition starts, the modulus drops very quickly. Sometimes, under this condition, tension is lost on the sample as indicated by irregularities in the E' plot at the beginning of the transition (see *Figure 9*).

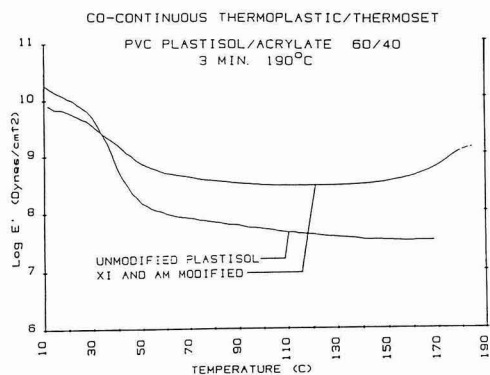
These powder coatings are pigmented at a pigment to binder weight ratio of about 0.4. At this pigment loading we see little or no increase, compared to the corresponding clears, in E' in the rubbery plateau. In contrast, pigmented solventborne coatings, pigmented at a P/B of 1.0, will often have E' values two to five times as high as that for the corresponding clear film. Van der Linde et al.<sup>36</sup> reported reductions in rubbery plateau values of modulus for pigmented versus clear films, and cure interference by  $TiO_2$  surface treatments is suspected.

More generally, for pigmented systems E' (min) is not expected to be directly proportional to XLD, as it is for clear coatings. However, if pigment content is constant in a series of films varying in the polymeric portion, the relative values of E' (min) still indicate relative XLD for the pigmented films.



**Figure 11**—Storage modulus plots for films prepared from a light stable epoxy (LSE) resin: (A) With two amine functional coreactants of widely differing equivalent weight; and (B) With amine adducts of systematically varying equivalent weight





**Figure 12**—Storage modulus plots for a polyvinylchloride (PVC) plastisol film and for a plastisol film modified by a network forming mixture of a crosslinker/initiator (XI) and an acrylated melamine (AM)

### Ambient Cure of Polyurethane Films

Results in *Figure 10* were obtained for a two-package polyurethane film prepared from an acrylic polyol and a commercial biuret trimer of hexamethylene diisocyanate. The pigment in this film increases the  $E'$  value in the rubbery plateau region very strongly. As shown in *Figure 10A*, polyurethane films give narrower transitions and higher values of  $\tan \delta$  (max) than observed for solvent-borne acrylic/MF systems or epoxy systems. The gentle rise in  $E'$  with temperature, as previously described, indicates that no additional curing occurs during the DMA scan after 24 days of ambient cure.

Films of this composition have sufficient toughness for DMA as free films after just one day ambient cure. As shown in *Figure 10B*, it is possible to follow the later stages of cure by making DMA runs each day for several days. Both XLD and  $T_g$  increase over several days, and there is a substantial change between 72 hr and 24 days. The exact cause of this longer term change is not known. Absorbed atmospheric water reacts with isocyanate groups so after several days none remain for further reaction. Long-term solvent loss may be part of these longer term changes. The close approach of the curves to one another at 140°C shows that XLD is similar when cure is all ambient or short ambient cure followed by completion of the cure during the DMA scan.

### Equivalent Weight Effects in Light Stable Epoxy Films<sup>28-40</sup>

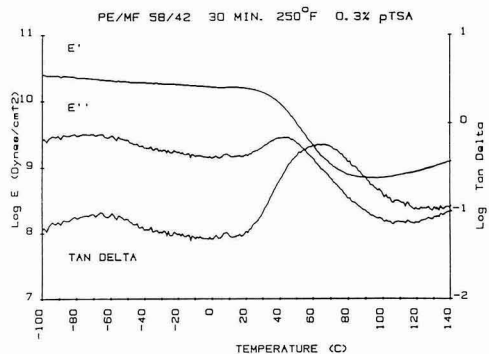
As shown in *Figure 11A*, very large differences were observed in  $E'$  plots for films prepared from a light stable epoxy (LSE) resin and two different amine functional coreactants. With the low equivalent weight amidoamine, the  $E'$  plot dropped very gradually over an extended temperature range. The high  $T_g$  and apparently high XLD, however, were not consistent with paint test results. The physical structure which produced lower than expected  $E'$  values in the glassy region and higher than

expected  $E'$  values in the rubbery plateau remained obscure during several years of study. Eventually, very similar plots were found in a review by Kamon et al.,<sup>41</sup> and these observations were attributed to the existence of many bulky ring structures. Since LSE has ring structures to begin with and self-condensation mechanisms forming other ring structures are plausible, we tentatively conclude that film 1 contains a high concentration of bulky rings. Films of this type have low values of penetration hardness, despite the high  $T_g$  and high  $E'$  (min). We have learned that low modulus in the glassy region at about 25°C often is associated with low penetration hardness, regardless of the  $T_g$  value. Selecting an adduct of much higher equivalent weight called for a 29/71 wt ratio for 0.63:1.0 equivalent ratio. For this film, a "normal" transition is observed. Films of type 2 are much harder despite a lower  $T_g$  value.

The amine adduct of film 2 in *Figure 11A* was prepared from low molecular weight BPA epoxy resins using mono(*n*-methyl)propylene diamine and blocking/unblocking techniques sensitive to primary versus secondary nature of amine groups. This approach provided good control of adduct structure and the means to vary equivalent weights through BPA epoxy selection. As shown in *Figure 11B*,  $E'$  plots in the rubbery plateau vary systematically as expected with amine hydrogen equivalent weight values of 477 and 315 g/eq. However, at 140 g/eq. the functional group ratio selected for this study called for a weight ratio of 58/42, and with the increase in epoxy group concentration, ring formation once again became significant; that is, the plot for the lowest equivalent weight amine adduct of *Figure 11B* was similar to that for the amidoamine of *Figure 11A*. This example shows that, in some cases, equivalent weight variation can alter the mechanism of cure as well as influence the XLD.

### Co-continuous Thermoplastic/Thermoset Films

Storage modulus ( $E'$ ) plots are shown in *Figure 12* for a film prepared without crosslinking from a polyvinylchloride (PVC) plastisol and for a 60/40 PVC/(XI + AM) film. The plastisol contains sufficient plasticizer, ap-



**Figure 13**—DMA plots for a flexible film prepared from a polyester polyol (PE) and a highly alkoxyated melamine resin (MF)

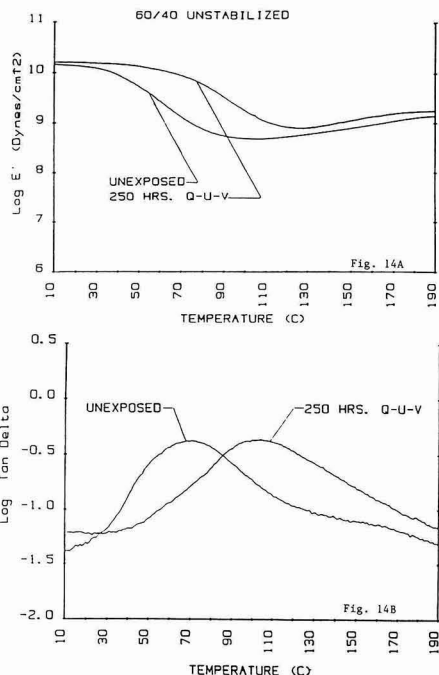
proximately 30%, to reduce  $T_g$  from about 80 to 30°C, as determined at 11 cps. PVC is reported<sup>42</sup> to be primarily an amorphous polymer that contains very small amounts of imperfect crystallites. Results in *Figure 12* indicate that these crystallites act somewhat like crosslinks in terms of increasing  $E'$  values in the rubbery plateau region. When there is no crystallinity,  $E'$  above  $T_g$  drops to approximately  $1 \times 10^6$  dynes/cm<sup>2</sup> whereas the plateau in *Figure 12* occurs at about  $2 \times 10^7$  dynes/cm<sup>2</sup>. The decrease in  $E'$  throughout the rubbery plateau is probably caused by the poorly organized nature of crystallites. Well organized crystalline regions give flat  $E'$  plots up to the melting point. The need for speculation in this discussion is a consequence of poor understanding of tertiary structure.

The 60/40 PVC/(XI + AM) film of *Figure 12* is an example of attempts to make PVC films behave like crosslinked films. The thermoset portion consists of a crosslinker/initiator (XI) and an unsaturated coreactant based on acrylated melamine (AM). Thermal cure in this system is believed to crosslink the thermoset portion by free radical reactions and simultaneously fuse the plastic. The PVC is not involved in crosslinking but exists co-continuously with the network formed by XI and AM. Efficient mixing is indicated by a single transition. Crosslinking of the thermoset portion is indicated by an increase in  $E'$  by a factor of about eight in the rubbery plateau region. The rise in  $E'$  at 170°C is attributed to additional curing during the DMA scan. The term "co-continuous" is preferred over "semi-interpenetrating polymer network" because it is more general. It is not certain that the data of *Figure 12* indicate that chains interpenetrate.

### Flexibility and Low Temperature Loss Peaks

Emphasis has been placed on the transition region and rubbery plateau in our work, but there may be a lot to learn at low temperatures as well. In thermoplastic polymer studies, low temperature peaks in the loss modulus ( $E''$ ) and  $\tan \delta$  plots are often observed for samples with good flexibility and impact resistance.<sup>43</sup> Rubber toughened plastics<sup>44</sup> and rubber toughened epoxy coatings<sup>45</sup> clearly show low temperature peaks attributable to the rubber phase. It is well known that polyester/MF films and urethane films have much better flexibility and impact resistance than do acrylic/MF films of similar  $T_g$  and XLD. A scan beginning at  $-100^\circ\text{C}$  is shown in *Figure 13* for a MF-crosslinked polyester film. A relatively weak low temperature loss peak is evident at approximately  $-70^\circ\text{C}$ . This peak might be associated with flexibility and impact resistance, but more work would be needed to confirm a relationship. Acrylic/MF films scanned over this range have no low temperature loss peaks. In *Figure 13* the low temperature peak is small in relation to the random noise, but in studies focused on the glassy region, with no attempt to observe the main transition, instrument settings could be optimized to improve the signal to noise ratio.

Flexibility of acrylic/MF films can be improved by including 10 to 15% on total resin solids of a high molecular weight, low  $T_g$ , low -OH acrylic resin, along with



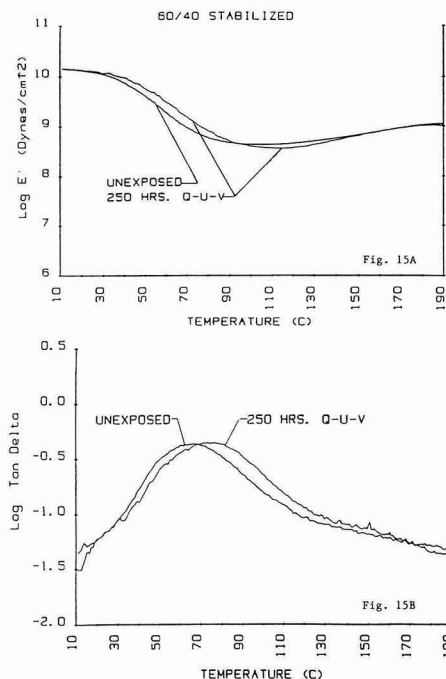
**Figure 14—Effect of Q-U-V on a 60/40 acrylic polyol/melamine film without stabilizers: (A) Storage modulus, and (B) Loss tangent**

the main acrylic coreactant.<sup>27</sup> These additives do not produce distinct low temperature loss peaks but rather result in a broad temperature interval of high loss modulus from approximately  $-30$  to  $+40^\circ\text{C}$ .

### Accelerated Weathering and Dynamic Properties<sup>46</sup>

DMA before and after accelerated weathering by Q-U-V® (Q-Panel Co.) shows that physical property changes can be determined after much shorter periods than required to observe gloss loss, cracking, or yellowing. The effects of just 250 hr Q-U-V on an unstabilized 60/40 acrylic/MF clearcoat are shown in *Figure 14*. The  $E'$  plots show that XLD increases (*Figure 14A*). The  $\tan \delta$  peak for the glass transition broadens and shifts to much higher temperatures (*Figure 14B*). Values of  $T_g$  before and after exposure are 71 and 104°C. These changes depend strongly on the acrylic/MF ratio. At 75/25, instead of 60/40, identical exposure causes a slight decrease instead of an increase in XLD, and the change in  $T_g$  is smaller, 78 to 93°C.

In commercially useful acrylic/MF clearcoats, gloss retention and crack resistance are greatly improved by hindered amine light stabilizers (HALS) and UV absorbers (UVA). The exposures of *Figure 14* were repeated on 60/40 films containing 3.0 phr UVA and 1.5 phr HALS with the results shown in *Figure 15*. It is obvious that



**Figure 15—Effect of Q-U-V on a 60/40 acrylic polyol/melamine film with 3.0 phr of UV absorber and 1.5 phr of hindered amine light stabilizer: (A) Storage modulus, and (B) Loss tangent**

stabilizers greatly reduce the effects of Q-U-V on dynamic properties. The XLD decreases slightly (Figure 15A), and  $T_g$  increases moderately, from 66 to 74°C (Figure 15B). Experiments illustrated here are very useful for adjusting acrylic/MF ratio and the type and amount of stabilizers for maintaining constant properties during weathering.

DMA and instrumental analysis by ESCA and ATR-FTIR have been carried out on the same films to provide information on relationships between physical property changes and chemical changes during accelerated weathering.<sup>47</sup> However, understanding of the multiple and complex effects of weathering is far from complete.

## SUMMARY AND CONCLUDING REMARKS

Although instrumental analysis and computer calculations were in their infancy in the 1930s and 1940s, Joseph J. Mattiello had a strong desire to apply scientific principles during his experiments carried out on production batches at Hilo Paint and Varnish Co. If he could speak to us today, how would he judge our current scientific zeal? Certainly the availability of new instrumental methods and the clarification of many structure concepts has made it easier to control coating properties rather than being controlled by and limited by available materials and methods. Despite the advances, there is still a lot that we

do not know, and few would deny that art still plays a very significant part in coatings development. Since the coatings industry is a commercial entity rather than a part of academia, judgements concerning what is known and what remains unknown are hazardous at best. Perhaps a lot more is known than is shared through publications and patents for proprietary reasons. Mattiello's inclination to communicate and interact with fellow coating scientists suggests that he would encourage us to "loosen up" and publish as much as we can short of giving away the fruits of our labor.

More specifically, this paper has shown that XLD need not be an abstract concept. Calculation of XLD is rather simple for stoichiometric networks at complete conversion. Computer programs are available for nonstoichiometric cases and/or incomplete conversion. Routine determination of XLD is possible by DMA for highly crosslinked films at least in some cases. Experimental and theoretically predicted XLDs are in agreement. Examples have been given of using DMA for understanding cure mechanisms, optimizing reactant structures, selecting coreactants, and following weathering.

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### APPENDIX 1—Joseph J. Mattiello, 1990–1948<sup>21</sup>

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As the years roll rapidly by, fewer of us know the life story of Joseph J. Mattiello. He was born in New York City in 1900, served in World War I in France receiving the Order of the Purple Heart, and years later, the Legion of Honor of France. His military service occurred between the ages of 17 to 19. He graduated from Brooklyn Polytechnic Institute in 1925 and worked for Hilo Paint and Varnish Company in New York from 1925 until he died of a heart attack in 1948. He earned his Masters Degree from Brooklyn Poly, in 1931 and his Ph.D. from Columbia University in 1936. Both degrees were earned

while on the job at Hilo. He was President of the New York Paint and Varnish Club in 1939 and President of the Federation of Paint and Varnish Clubs in 1943-44. The Federation of Varnish Clubs was, of course, the forerunner of our Federation of Societies for Coatings Technology. War hero, scholar, scientist, author, leader, organizer, promoter, communicator, and a close friend of many—he was all of these. In 1946, while acknowledging an ASTM lecture award, he was quoted as saying "I have come to realize more and more that friendship is more precious than anything else in the world."<sup>21</sup>

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**APPENDIX 2—Ideal Network and Ideal Gas Laws**


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In keeping with the “back to basics” theme of the XLD section, the analogy between the ideal network law, equation (9), and the ideal gas law is of interest. The main reason for drawing the analogy is that it aids memory. Almost everyone who took freshman chemistry can remember the ideal gas law. Development of the analogy also clarifies the circumstances under which equation (9) can be expected to be valid.

The ideal gas law can be written in the form

$$P = (n/V)RT \quad (1A)$$

where  $n$  is the number of moles of gas in volume  $V$  and  $R$  and  $T$  have the same meaning as in equation (9). The factor  $(n/V)$  from equation (1A) and  $\nu_e$  from equation (9) are similar in that both are moles of something per unit volume; gas molecules in the former and elastically effective network chains in the latter. Shear modulus,  $G$ , and pressure,  $P$ , can be expressed in the same units; e.g., dynes/cm<sup>2</sup> in the cgs system for which  $R = 8.314 \times 10^7$  ergs/°K•mole (or dynes•cm/°K•mole, which more directly shows the unit balance in equation [9] or equation [1A]).

Both equations have their basis in the effect of temperature on the motion of material. For networks, motion of chain segments is considered. For gases, motion of individual molecules is considered. Segmental motion results in various chain conformations. There are many more conformations corresponding to a balled-up chain than corresponding to an extended chain. Therefore, balled-up or compact conformations are favored statistically, i.e., by entropy. When a strain is imposed on a sample, chains must change shape from being balled-up to being slightly less balled-up. The entropy considerations of the less-random shape results in a restoring force which we can

measure and relate to  $G$ . If the temperature is increased, segments move more rapidly which results in a stronger restoring force. If a larger number of chains are participating in attempts to get more balled-up, a larger restoring force results. These last two statements are consistent with appearance of  $T$  and  $\nu_e$  on the right in equation (9). The phrase “consistent with” is pretty weak compared to the phrase “it has been shown by derivation that.” The latter is the substance of kinetic theory of rubber elasticity.<sup>22-24</sup>

We expect equation (9) to be valid if the imposed strain can be accommodated by rotations about bonds and readjustment of chain conformations. If primary bond angles must be changed, or bonds broken to accommodate the strain, we do not expect equation (9) to be valid. If the number of chains is dependent only on the concentration and functionality of junction points, we expect equation (9) to be valid. If entanglements of chains act like chemical crosslinks, we don't expect equation (9) to be valid.<sup>23,24</sup> For highly crosslinked thermosets, as discussed in this paper, the length of chains between junction points is short and entanglements are very unlikely. This may be the reason that the ideal network law is more likely to be valid for tight networks than for loose ones.

To continue with the analogy, note that pressure of a gas results from bombardment of the container walls by gas molecules. At higher temperatures the average velocity of molecules is higher and the effect of collisions with the walls is greater. If more molecules are involved in the bombardment, again the pressure is greater. These last two statements are consistent with appearance of  $T$  and  $(n/V)$  on the right in equation (1A). In this case, derivation and quantitative aspects are the substance of kinetic theory of gases.

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# Water-Reducible, Unsaturated Polyesters As Binders and Clear Coatings For UV-Curable Furniture Coatings

Michael J. Dvorchak and Bernd H. Riberi  
Miles Inc.\*

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The paper describes the development and properties of water-reducible, unsaturated polyesters (WR-UPE) which can be used to formulate UV-curable furniture coatings.

The development of WR-UPE had to overcome two problems. First, the monomer copolymerizable with the polyester, usually styrene, cannot be used. Therefore, a system needed to be developed that produced coatings with adequate properties even in the absence of copolymerizable monomers and that produced stable coatings systems with good chemical and water-resistant properties even though it was water reducible.

The incorporation of polyols which had allyl ether groups allowed the formulation of unsaturated poly-

esters that do not require styrene to cure into a film with good properties. The further incorporation of special nonionic emulsifiers into the polymer backbone made the unsaturated polyester water reducible.

Coatings formulations based on this WR-UPE have attained VOCs as low as 0.2 lb/gal without using monomer diluents or amine solubilizing agents. These formulations can be cured by a cobalt peroxide mechanism. UV-curing allows for faster curing. The curing conditions and properties of the resulting coatings are discussed. The proper selection of UV-photoinitiator makes it possible to UV-cure even pigmented systems.

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## INTRODUCTION

Currently, coatings are going through a major evolution. Low solids, solvent-containing coatings with high VOCs are decreasing in importance. Parts of California have instituted strict VOC standards and are requiring furniture companies and their coatings suppliers to develop technologies that meet these standards. Many other states are supposed to follow the lead of California. The federal government is also addressing VOC levels: Congress is currently looking at amendments to the Clean Air Act. Tougher amendments to the Clean Air Act will strain even the best coatings chemists' ability to formulate outdated coatings to lower VOCs. Truly, the decisions that

are made during the first part of this decade will determine the technology that will be used in the year 2000 and beyond.

## TRADITIONAL UNSATURATED POLYESTERS

Traditional unsaturated polyesters are polycondensation products based on saturated and unsaturated dicarboxylic acids such as maleic or fumaric acids and primary bivalent alcohols. Typically, these systems are dissolved in styrene which reacts with and crosslinks the unsaturated resin when a dryer system, that is, a cobalt salt and an organic peroxide, is used. The cobalt salt decomposes the peroxide to form free radicals,  $R^{\cdot}$ , which initiate the crosslinking of the system.<sup>1</sup> This reaction mechanism is described in *Figure 1*.

These types of systems are two-component coatings with the cobalt in the formulated product and the peroxide

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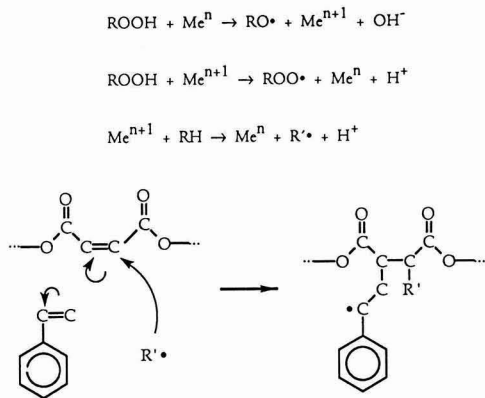


Figure 1—Free radical crosslinking of a traditional unsaturated polyester

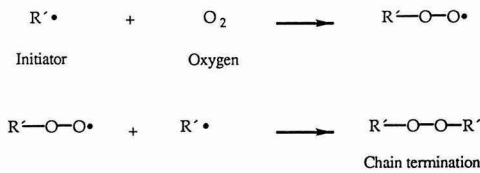


Figure 2—Chain termination of the free radical initiator by oxygen

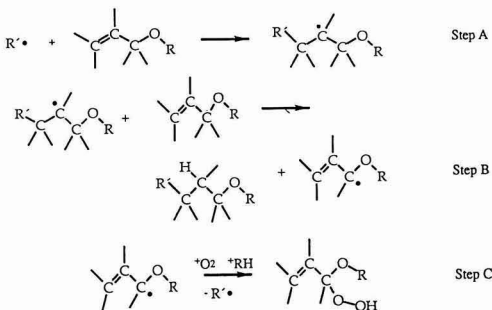


Figure 3—Reaction of a free radical with an allyl ether in the presence of oxygen to form hydroperoxides

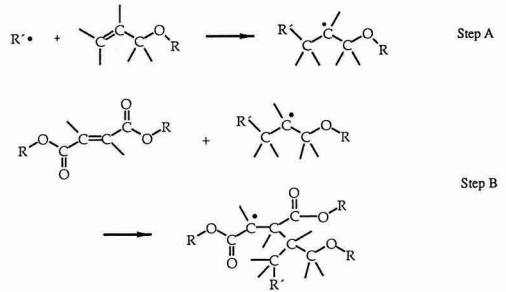


Figure 4—Reaction of the free radical with an allyl ether-modified fumarate without the presence of oxygen

Table 1—Key to Photoinitiators Used

Letter Code	Photoinitiator Used	Product Name
A	2-Hydroxy-2-methyl-1-phenyl-propan-1-one	Darocur® 1173
B	Proprietary blend of aromatic ketones	Darocur® 1664
C	Proprietary blend of aromatic ketones	Darocur® 4043
D	1-(4-Isopropylphenyl)-2-hydroxy-2-methyl-propan-1-one	Darocur® 1116
E	Oligomeric $\alpha$ -hydroxy acetophenone	Esacure® KIP
F	Proprietary blend of aromatic ketones	Darocur® 4263
G	Proprietary blend of aromatic ketones	Darocur® 4265

Darocur® is a registered trademark of EM Industries, Hawthorne, NY.  
Esacure® is a registered trademark of Fratelli Lamberti S.p.A.—Albizzate, Italy.

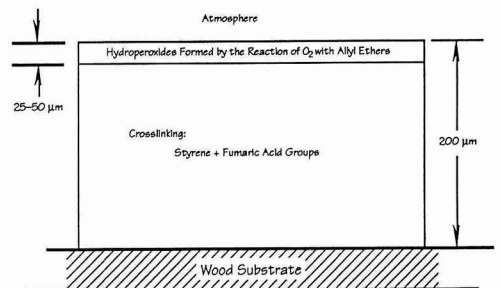
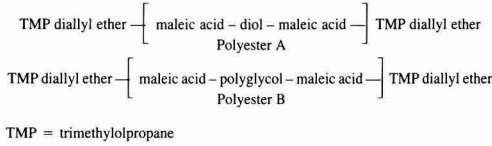


Figure 5—Curing of non-air-inhibited, unsaturated polyester

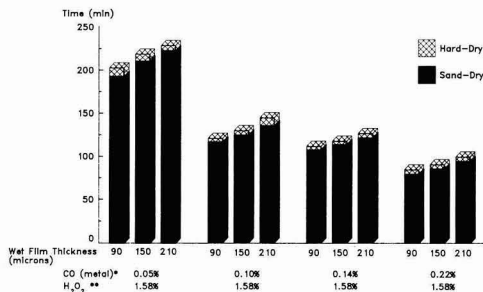


**Figure 6—Structure of the water-reducible, unsaturated polyesters**

added just prior to use. They have a short pot life which is typically about 5-10 min. They also have a problem with surface drying; the coatings surface does not cure and will be sticky because the oxygen in the air inhibits the free radical mechanism. A mechanism for oxygen termination is given in *Figure 2*.

To overcome this effect, a paraffin wax is usually added to the formulation. While the system is curing, the paraffin wax migrates to the surface. Oxygen from the atmosphere is excluded, and the chain termination is prevented. However, this technique works but requires removal of the wax since the wax leaves a low gloss appearance. The surface must then be sanded and polished to get a high gloss appearance. This additional sanding and polishing is very labor intensive.

Work in the mid-1950s<sup>2</sup> saw the development of non-air-inhibited, unsaturated polyesters that cured without the aid of waxes. These unsaturated polyesters would have the same hardness and chemical resistance of traditional unsaturated polyesters and would give a high gloss without sanding and polishing. The oxygen inhibition could be prevented through the introduction of allyl ether groups to the modified fumarates. *Figure 3* shows a reaction mechanism on the surface. The radical R<sup>·</sup> attacks the allyl ether forming the allyl ether radical (Step A). This allyl ether radical can then react with a hydrogen atom (Step B), and in conjunction with another allyl ether radical and oxygen, form hydroperoxides which will aid in the further development of new radicals in the presence of a cobalt drier (Step C).



**Figure 7—Water-reducible, unsaturated polyester influence of cobalt on air dry times. Note: \* CO based on WR-UPE; and \*\*H<sub>2</sub>O<sub>2</sub> (as supplied) based on water reducible, unsaturated polyester**

A reaction path within the film is shown in *Figure 4*. The radical R<sup>·</sup> attacks the allyl ether forming the allyl ether radical (Step A). This radical then attacks the double bond of a fumaric acid group (Step B), forming a radical that can in time form the three-dimensional network of the polymer.<sup>3</sup>

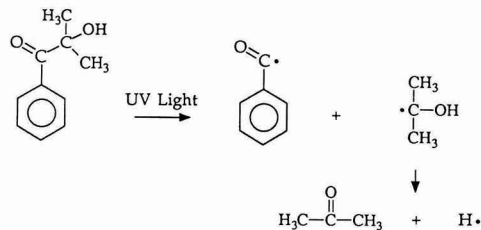
How these two separate reactions may take place within the coating is described in *Figure 5*. On the surface, the oxygen actually aids in the cure due to the allyl ether groups. Below this surface, the copolymerization of styrene and fumaric acid groups occurs without being inhibited.<sup>4</sup>

**WATER REDUCIBLE-UNSATURATED POLYESTERS (WR-UPE)**

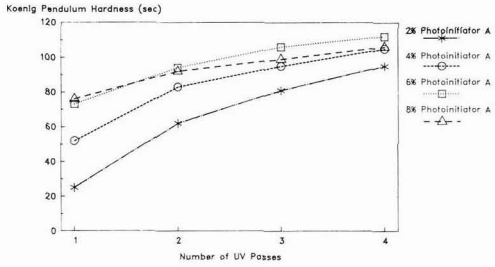
The ongoing development to reduce VOC and the desire to work with water-based coatings led to water-reducible, unsaturated polyesters (WR-UPE) with allyl ether groups. They do not require the aid of cosolvents and amines.

Early attempts modified the polyester with polyoxyalkylene glycols with varying molecular weights in order to increase the hydrophilic nature of the resins to make them water soluble. However, the dry-times of these resins were too long, the films remained sticky, and they had no resistance to water. It was found, however, that unsaturated polyesters with polyoxyalkylene glycols could act as emulsifying agents for the unmodified polyester,<sup>5</sup> and thus produce a stable oil-in-water emulsion if these two products were mixed and dispersed. The disadvantage of this approach is obvious. The two products—unmodified polyester and polyester modified with polyoxyalkylene glycol—have to be prepared separately and then mixed to give a water-reducible resin.

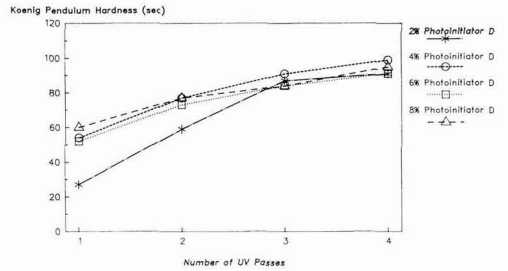
Additional work<sup>6</sup> showed that a WR-UPE could be produced in a single process. First, two moles of maleic anhydride are reacted with one mole of a mixture of low molecular weight diols/polyoxyalkylene glycols giving a mixture of half-esters that appear in the brackets of *Figure 6*. Subsequent condensation with two moles of trimethylolpropane diallyl ether leads to the formation of a mixture of Polyester A and Polyester B (*Figure 6*).



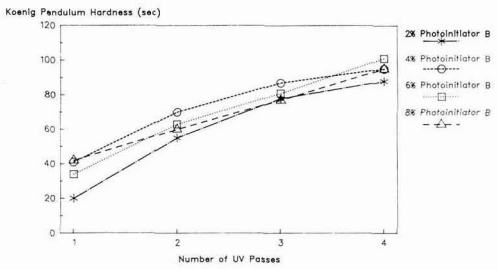
**Figure 8—Cleavage of 2-hydroxy-2-methyl-1-phenyl-propan-1-one under the influence of UV light**



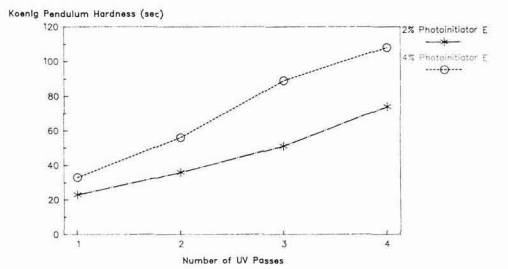
**Figure 9**—Photoinitiator A water-reducible, unsaturated polyester. Influence of the % photoinitiator on hardness. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F); and UV per pass = 0.317 J/cm<sup>2</sup>



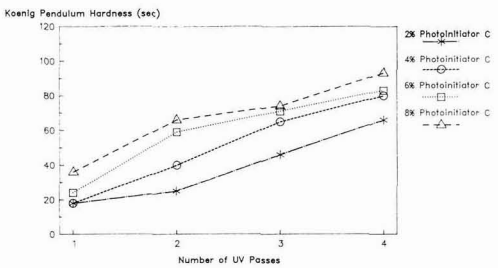
**Figure 12**—Photoinitiator D water-reducible, unsaturated polyester. Influence of the % photoinitiator on hardness. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F); and UV per pass = 0.317 J/cm<sup>2</sup>



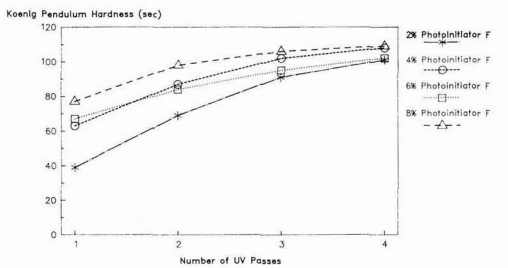
**Figure 10**—Photoinitiator B water-reducible, unsaturated polyester. Influence of the % photoinitiator on hardness. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F); and UV per pass = 0.317 J/cm<sup>2</sup>



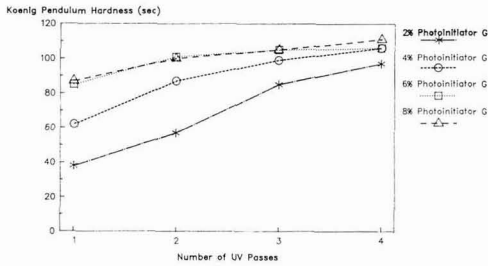
**Figure 13**—Photoinitiator E water-reducible, unsaturated polyester. Influence of the % photoinitiator on hardness. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F); and UV per pass = 0.317 J/cm<sup>2</sup>



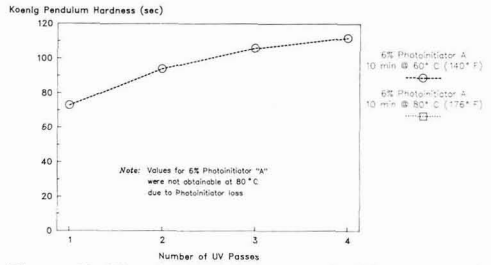
**Figure 11**—Photoinitiator C water-reducible, unsaturated polyester. Influence of the % photoinitiator on hardness. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F); and UV per pass = 0.317 J/cm<sup>2</sup>



**Figure 14**—Photoinitiator F water-reducible, unsaturated polyester. Influence of the % photoinitiator on hardness. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F); and UV per pass = 0.317 J/cm<sup>2</sup>



**Figure 15—Photoinitiator G water-reducible, unsaturated polyester. Influence of the % photoinitiator on hardness. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F); and UV per pass = 0.317 J/cm<sup>2</sup>**



**Figure 16—Photoinitiator A water-reducible, unsaturated polyester. Influence of temperature. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F) or 10 min @ 80°C (176°F); and UV per pass = 0.317 J/cm<sup>2</sup>**

This mixture can then be easily emulsified in water. No additional emulsifiers, cosolvents, or amines are needed to form a stable emulsion.

Part of the system acts as an internal emulsifier and crosslinks into the final film during the curing process. Emulsifying agents, that could lead to poor chemical and water resistance, are not needed. Also, amines that would lead to discoloration of wood substrates and cosolvents that would contribute to the VOC emissions are not required. Curing of the system can be done with hydrogen peroxide/cobalt or with UV light.

**CONVENTIONAL CURING OF WR-UPE**

This WR-UPE can be conventionally cured using a metal salt and peroxide. Dry times will vary, depending on the amount of metal salt and peroxide used. Typical dry times are given in Figure 7. Dry times at the highest level of peroxide/cobalt were between one to two hours for wet film thicknesses of 90 to 210 μm. Dry times will effectively decrease with increasing temperatures.

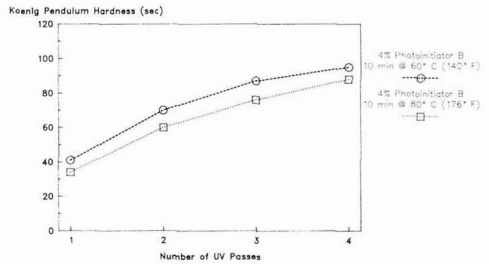
Because of the presence of the allyl ether group in the WR-UPE, it develops a hard surface typical of a UPE without the aid of the waxes or other means to exclude the oxygen. Also, as in the case of monomer containing UPE, the WR-UPE forms a two-component system but has a much longer pot life of approximately 24 hr.

**UV-CURING OF WR-UPE**

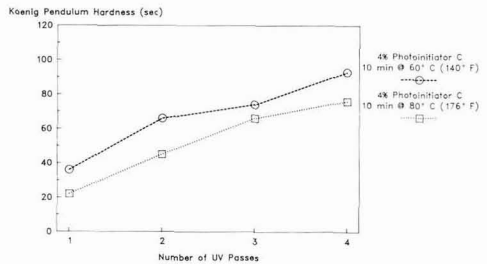
**UV-Curing Mechanism**

The most significant method of cure for this WR-UPE is through the use of UV light and photoinitiators. This radical-cure mechanism allows for instantaneous cure of the WR-UPE. It eliminates any difficulties due to ingredients in certain woods that can inhibit the peroxide/cobalt cure mechanism. It also has the added advantage of working as a one-component system. Typical photoinitiators for a WR-UPE are those based on hydroxypropiophenones which can dissolve into the oil phase of the emulsions.

A cleavage reaction of 2-hydroxy-2-methyl-1-phenylpropan-1-one when it is photolyzed is given in Figure 8. This proposed cleavage goes through a very short triplet

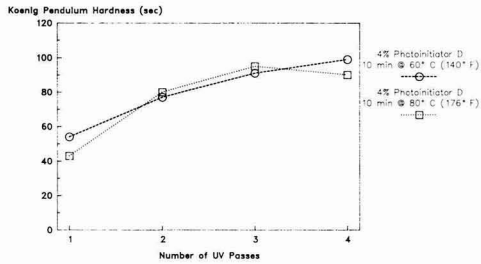


**Figure 17—Photoinitiator B water-reducible, unsaturated polyester. Influence of temperature. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F) or 10 min @ 80°C (176°F); and UV per pass = 0.317 J/cm<sup>2</sup>**

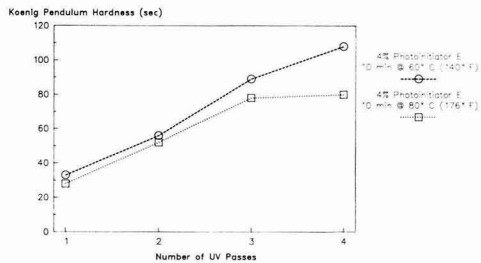


**Figure 18—Photoinitiator C water-reducible, unsaturated polyester. Influence of temperature. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F) or 10 min @ 80°C (176°F); and UV per pass = 0.317 J/cm<sup>2</sup>**

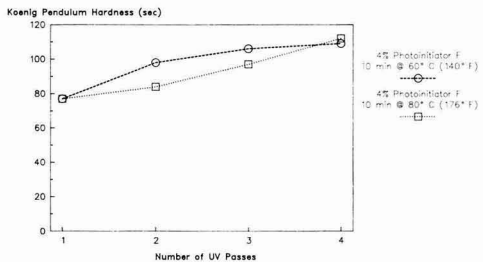




**Figure 19—Photoinitiator D water-reducible, unsaturated polyester. Influence of temperature. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F) or 10 min @ 80°C (176°F); and UV per pass = 0.317 J/cm<sup>2</sup>**



**Figure 20—Photoinitiator E water-reducible, unsaturated polyester. Influence of temperature. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F) or 10 min @ 80°C (176°F); and UV per pass = 0.317 J/cm<sup>2</sup>**



**Figure 21—Photoinitiator F water-reducible, unsaturated polyester. Influence of temperature. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F) or 10 min @ 80°C (176°F); and UV per pass = 0.317 J/cm<sup>2</sup>**

state and then decomposes by  $\alpha$ -splitting to give a benzoyl radical and a 2-hydroxy-2-propyl radical.<sup>7</sup>

### Influence of Photoinitiator Concentration

Choosing the right type of photoinitiator and the right amount of photoinitiator is very important. *Figure 9* shows an evaluation of Photoinitiator A (see *Table 1*). The old adage that “more is not necessarily better” really hits home on the percent of photoinitiator that is used. Too much photoinitiator can create an excessive number of radicals that will combine and thus terminate the chain reaction.<sup>8</sup>

The evaluations in the following were done by dispersing seven parts of WR-UPE with the photoinitiator in three parts of water for two minutes. The emulsion was reduced to 40% solids and drawn down on glass with a 210  $\mu$ m drawn down bar. The film was subjected to oven drying to remove the water at 60°C (or 80°C depending on the test) for 10 min followed by exposure to a high pressure mercury vapor lamp with a 200 W/in. at a line speed of 6 ft/min/lamp. The amount of energy that the film received was 0.317 J/cm<sup>2</sup> per pass. The Koenig pendulum hardness was used to measure the degree of cure.

### Influence of Photoinitiator Type

The type of photoinitiator used is also very important. *Table 1* shows the different photoinitiators used to evaluate the WR-UPE.

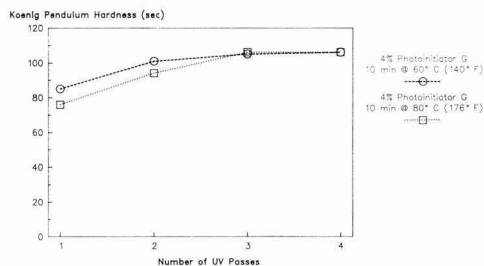
*Figures 9-15* show the influence of the different photoinitiators on the hardness of the WR-UPE after UV-curing. Obviously, Photoinitiator G (*Figure 15*) gives the best performance of all the photoinitiators studied since it gives the best hardness development. Photoinitiator A (*Figure 9*) and Photoinitiator F (*Figure 14*) at higher concentrations can also be considered. The other photoinitiators used are clearly inferior.

### Influence of Pre-Dry Temperature

Before the WR-UPE is subjected to the UV light, water has to be removed. Evaporation of water does take place at room temperature, but this would require too much time when the system is used in an industrial application. Elevated temperatures speed up the evaporation of the water; typical temperatures for the furniture industry are in the range of 60-80°C. Therefore, another aspect to consider when evaluating photoinitiators that are used in the WR-UPE systems is their ability to remain in the film during the water removal phase prior to UV-curing.

First tested was the performance of Photoinitiator A at different water removal temperatures prior to UV-curing the WR-UPE. When the system was heated for 10 min at 60°C, the film exhibited good properties (*Figure 16*). However, when the wet film was heated 10 min at 80°C, it did not cure and remained sticky. We assume that Photoinitiator A volatilized with the water. (This assumption is subject to further studies.)

Photoinitiator A has a definite problem when used at 80°C. Because an industrial finishing line can run faster at 80°C than 60°C, it was important to find a photoinitia-



**Figure 22—Photoinitiator G water-reducible, unsaturated polyester. Influence of temperature. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F) or 10 min @ 80°C (176°F); and UV per pass = 0.317 J/cm<sup>2</sup>**

tor that could be used at elevated temperatures. We found that all the photoinitiators tested generally gave an inferior performance at 80°C than at 60°C, but they all performed much better than Photoinitiator A (Figures 17-22).

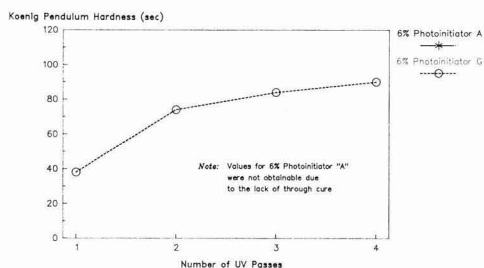
#### UV-CURING OF PIGMENTED WR-UPE

Up to this point, the UV-curing of clear coats has been described. Until recently, the UV-curing of pigmented unsaturated polyesters in film thicknesses required to coat furniture was not considered possible. However, new developments in the field of photoinitiators allow UV-curing of pigmented, unsaturated polyesters if the right resin is used.<sup>9</sup> Pigmented systems with the WR-UPE can also be UV-cured under these conditions.

Two WR-UPE coatings were prepared with 12% TiO<sub>2</sub> on the basis of resin at 40% solids: one with 6% Photoinitiator A and the other system formulated the same way but using 6% Photoinitiator G. Wet film drawdowns were done at 210 μm and cured as described in the previous section. Figure 23 shows the results. The formulation with Photoinitiator A cured at the surface but stayed liquid inside and gave a wrinkled appearance. On the other hand, Photoinitiator G gave a cured film after one pass and was almost fully cured after two passes through the UV oven.

#### CONCLUSIONS

(1) The development of WR-UPE now allows the coatings chemist to attain VOCs for furniture coatings at values as low as 0.2 lb/gal.



**Figure 23—Photoinitiator A and G pigmented water-reducible, unsaturated polyester. Influence of pigment on photoinitiator. Pendulum hardness vs # of UV-passes. Cure schedule: 10 min @ 60°C (140°F); and UV per pass = 0.317 J/cm<sup>2</sup>**

(2) Choice of UV-photoinitiator requires diligence not only in the amounts used but also the type. Water-based coating systems require additional evaluation to determine the effect of photoinitiator loss when the water is removed prior to UV-curing.

(3) With the introduction of newer UV-photoinitiators, pigmented WR-UPE is now possible.

#### ACKNOWLEDGMENT

The authors deeply appreciate the hard work and dedication that Patrick J. McFarlane and Frank C. Emanuele showed in the many tests performed for the development of this paper.

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# Fundamental Studies of Epoxy Resins For Can and Coil Coatings III. Effect of Bisphenol Structure On Resin and Coating Flexibility

R.A. Dubois and P.S. Sheih  
Dow Chemical USA\*

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Many workers have reported improvements in coating flexibility by blending and grafting flexibilizers with Bisphenol A based epoxy coatings or by using aliphatic epoxies. These techniques are often limited by the rigidity of the Bisphenol A unit or by loss of other desirable properties. Systematic variation in the structure of the groups bridging the phenol units of diphenols is correlated with flexibility of the corresponding epoxy resins and ultimately with coating performance of the cured resins. This technique, combined with optimum curing conditions, can give coatings as flexible as aliphatic epoxies while retaining the adhesion, chemical resistance, and reactivity characteristic of Bisphenol A based epoxies.

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## INTRODUCTION

This report is one of a series from this laboratory responding to a growing need for high performance epoxy resins in general and, more specifically, for more flexible coatings in the can and coil industry.<sup>1-4</sup> Typically, efforts to improve flexibility involve blending or grafting<sup>4</sup> flexibilizing agents with epoxy resins made from commonly available diphenols (i.e., those with short, relatively rigid bridging groups like Bis A, Bis F, Bis K, etc.). Some have argued that improvements in flexibility using such techniques are ultimately limited by the rigidity of the diphenol core.<sup>5-8</sup> In an effort partly to avoid this limita-

tion, a variety of aliphatic epoxy resins has been developed; but they generally suffer from loss of other properties like adhesion, chemical resistance, or reactivity.<sup>9,10</sup>

Another approach to improve flexibility is to introduce the flexibilizing agent into the spacer group between the phenol units of diphenols used to prepare epoxy resins. Although there are only a few reports using this approach for epoxy resins, it has been widely used to improve flexibility and processibility in other polymers; for example, in polyimides.<sup>11</sup> One attempt at determining the effect of such spacer groups on performance properties of cured epoxy resins failed to show any trends, apparently because the diphenols were all bridged with groups similar to the rigid isopropylidene group of Bis A.<sup>5</sup> Another report demonstrated a correlation between chain length of the spacer group and flexibility of the cured epoxy resin for a series of diphenols based on bis esters of parahydroxybenzoic acid.<sup>8</sup>

In this report, we determine the effect of using diphenols with systematic variation in alkylene and alkylene-dioxy bridging groups between the phenol units of diphenols on coating performance properties of epoxy resins made by advancing the diphenols with the diglycidyl ether of Bis A, DGEBA (*Figure 1*). The advanced resin coatings are cured with a phenol/formaldehyde crosslinking agent.

## EXPERIMENTAL

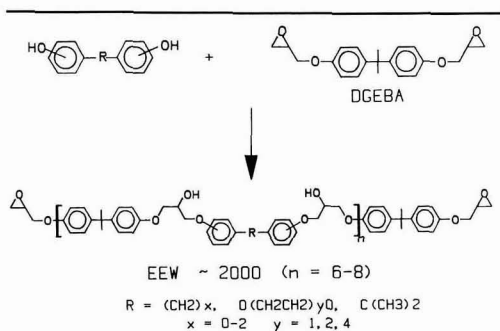
### Materials

4,4'-Biphenol was obtained internally (Dow Chemical) in >99% purity by oxidative coupling of a substituted

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**Figure 1—Advancement of the diglycidyl ether of bisphenol A with diphenols bridged with alkylenes and alkylenedioxy groups (R)**

phenol. 4,4'-Methylenediphenol and 4,4'-ethylenediphenol, p-EDP, were obtained by published methods.<sup>12,13</sup> 3,3'-(Ethylene-1,2-dioxy)diphenol, m-EDDP, was obtained from Aldrich Chemical Co. and crystallized twice from acetone to obtain a product of >97% purity by HPLC peak area. The remaining (alkylenedioxy) diphenols were obtained by a published method.<sup>14</sup> METHYLON™ 75108, a phenol/formaldehyde crosslinking agent, was obtained from BTL Specialty Resins Corp. BYK™ -361, an acrylic copolymer flow modifier, was obtained from BYK-Chemie USA. The coating substrate was tin free steel (TFS) obtained from Weirtec. The TFS is a low carbon, cold-reduced steel with a layer of chromium oxide on the surface, nominally 0.0072 in. (0.19 mm) thick, and widely used in the can manufacturing industry as a less expensive replacement for tin-plated can stock.

### Resin Advancement

All of the epoxy resins were prepared using the procedure described here for the advancement of D.E.R.™ 383 with 3,3'-(ethylene-1,2-dioxy)diphenol (m-EDDP). The molar ratio of diepoxide to diphenol was typically about 1.1 to 1.2, calculated to give resin with an epoxide equivalent weight (EEW) of about 2000. A 250 mL round bottom flask equipped with a condenser, a thermometer, and an air driven steel stirrer shaft was charged with 130.58 grams of D.E.R. 383, a Dow epoxy resin with an EEW of 180.6, and 74.57 grams (0.3028 moles) m-EDDP, then heated to about 90°C under a nitrogen purge of 200 mL/min before adding 0.286 grams of a 70% by weight solution of triphenylethyl phosphonium acetate/acetic acid in methanol as catalyst. The temperature was increased over a one-hour period to 190°C. At that point, the reaction exothermed and a temperature of up to 191°C was maintained for several minutes without external heating. The reaction mixture was heated at 185°C for an additional 45 min, then quickly heated to 210°C to facilitate pouring viscous contents onto a sheet of aluminum foil to cool. Reaction samples were removed at 15, 30, and 45 min after the reaction temperature had reached

190°C to determine the extent of reaction. The EEW of each was 1894, 1955, and 1972 respectively, and the final EEW was 2019.

### Panel Preparation

The TFS panels were rinsed first with an aromatic hydrocarbon solvent then with acetone and baked at 400°F for 10 min to degrease. Formulations were applied with wire wound drawdown rods according to a modified ASTM D-447 procedure to give cured coatings of 0.17 to 0.23 mils thickness, as measured with a Fischer Multi-scope. The coated panels were cured at 400°F in a Blue M forced air electric oven usually for 10 min but also, on occasion, 15 or 20 min.

### Coating Formulation

The advanced copolymer epoxy resin was dissolved in a blend of 2-butoxyethanol and cyclohexanone in a range of ratios from 100/0 to 60/40 respectively, to give a 40% solids solution. To a 49.34 grams portion of the resin solution an appropriate amount of METHYLON™ 75108 was added to comprise 20, 10, 5 or 2.5 wt% of the total solids, and the solution was then diluted with more of the solvent blend to attain a Gardner G viscosity. Finally, the appropriate amount of 85% phosphoric acid was added to comprise 1.00, 0.75, 0.50, or 0.30 wt% of the total solids, and BYK™ -361, an acrylic copolymer flow modifier, was also added at a 0.05 wt% level of formulation solids. The formulation was shaken at least 24 hr before applying to the TFS panels.

### Resin Characterization

EEW was determined by titration with perchloric acid and tetramethyl ammonium bromide (ASTM D-1652). Viscosity was determined by a cone and plate method at 200°C. Gardner viscosity in solvent blends of 2-butoxyethanol and cyclohexanone were obtained. Glass transition temperature ( $T_g$ ) was determined on a DuPont 912 Differential Scanning Calorimeter. Molecular weights were determined by gel permeation chromatography on a Waters Model 150-C gel permeation chromatograph equipped with a Bimodal Column Set using tetrahydrofuran eluant at 1 cc/min, calibrated against polystyrene standards.

### Coating Tests

Flexibility of the coatings was determined by the wedge bend test of ASTM D 3281-84 which specifies an impact process to produce the bend. The bend was taped and pulled with Scotch brand 610 tape and treated with acidic copper sulfate solution to highlight the exposed metal. The results were reported as millimeters of coating failure. Resistance to methyl ethyl ketone (MEK) was determined by rubbing the coating surface with the ball end of a ball-peen hammer covered with eight layers of MEK saturated cheesecloth. No force was applied to the hammer other than that needed to guide the hammer across the coating surface. Rubbing was continued until first exposure of bare metal, as indicated by dipping into



**Table 1—Epoxy Resins of the Diglycidyl Ether of Bisphenol A Advanced with Diphenols Bridged with Alkylene and Alkyl-enedioxy Groups (R)**

R <sup>a</sup>	EEW <sup>b</sup>	T <sub>g</sub> (°C)
Single Bond <sup>c</sup>	1800	87
C(CH <sub>3</sub> ) <sub>2</sub>	2183	83
p-CH <sub>2</sub>	2240	65
p-CH <sub>2</sub> CH <sub>2</sub>	2129	64
o/p-CH <sub>2</sub> <sup>d</sup>	2205	67
m-O(CH <sub>2</sub> ) <sub>2</sub> O	2019	61
m-O(CH <sub>2</sub> ) <sub>4</sub> O	2118	54
p-O(CH <sub>2</sub> ) <sub>4</sub> O	1706	53
p-O(CH <sub>2</sub> ) <sub>6</sub> O	2139	41

(a) The bridging group for the diphenol portion of the advanced resins of Figure 1.

(b) Epoxide equivalent weight.

(c) The bridging group for bisphenol.

(d) Bridging group for Bisphenol F which is a mixture of ortho and para isomers.

an acidic copper sulfate solution. The results were reported as MEK double rubs, each of which constitutes a forward and reverse stroke of the ball-peen hammer. T<sub>g</sub> of the cured coatings were determined on a DuPont 943 Thermal Mechanical Analyzer.

## RESULTS AND DISCUSSION

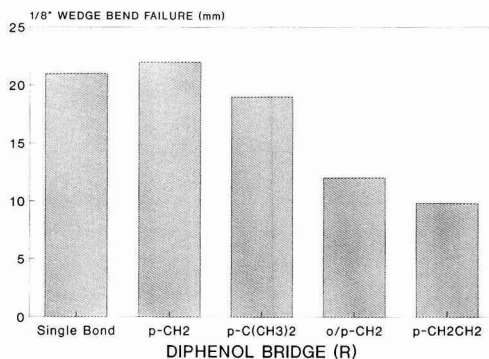
We chose to determine the effect of variations in the bridging structure of the diphenols on coating properties of epoxy resins derived from them by using the copolymers shown in Figure 1 rather than the homopolymers, which could be prepared by advancing the diphenol with its diglycidyl ether, in order to maximize the number of resins that could be screened in a given time. Since DGEBA is readily available, the copolymer approach not only obviates the need to prepare new diglycidyl ethers, but also reduces by half the amount of diphenol that has to be synthesized. The trends and correlations that develop could then be used to select homopolymers for further study.

The first series of diphenols included those bridged with short, linear alkylene chains of zero through two carbons (Figure 1). Since substitution pattern on the aromatic rings usually affects bulk properties, we chose to obtain the para substituted diphenols. Although 4,4'-biphenol (p-BP, R is a single bond) was available to us in >99% purity, 4,4'-methylenediphenol (p-Bis F, R is p-CH<sub>2</sub>) had to be recovered by a two-step crystallization of Bis F (R is o/p-CH<sub>2</sub>) that had first been distilled to remove polyphenols of greater than two functionality. Distilled Bis F is a mixture of the three ortho/para isomers of methylenediphenol (R is o/p-CH<sub>2</sub>). 4,4'-Ethylene-diphenol (p-EDP, R is CH<sub>2</sub>CH<sub>2</sub>) was prepared in >99% purity by hydrolysis of the bis diazonium salt of 4,4'-ethylenedianiline. Each of the diphenols was advanced with DGEBA to an EEW of about 2000, typical for resins used in can coatings. Bis A and the distilled, two-functional Bis F were also similarly advanced with DGEBA; the former to serve as a control and the latter to determine the effect of the presence of ortho isomers. T<sub>g</sub>s for this series of resins were obtained to determine the effect of substitution of every other Bis A unit with the diphenols

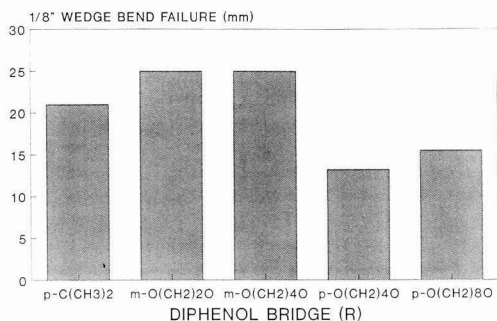
of this series on backbone flexibility of the uncured resin. Table 1 shows that all of the diphenols except p-BP cause a depression of T<sub>g</sub> by 6 to 19°C. This effect can be due to a combination of several factors including disruption in the symmetry of the backbone and changes in restrictions to rotational motion along the resin backbone.

Although T<sub>g</sub>s for the uncured resins suggest a more rigid polymer backbone for the Bis A homopolymer compared to all but one of the copolymers (the DGEBA/p-BP copolymer has a higher T<sub>g</sub>), Figure 2 shows that for the cured systems the DGEBA/Bis A homopolymer exhibits an impact wedge bend flexibility comparable to the DGEBA/p-BP and DGEBA/p-Bis F copolymers. Among the DGEBA copolymers with para substituted diphenols, it is only the one with the longer two carbon bridged, p-EDP, that shows significant improvement. These results conform to conventional wisdom on the effect of introducing longer chains into the backbone of epoxy resins, but the results for the DGEBA/p-Bis F copolymer relative to the DGEBA/Bis F copolymer and to the DGEBA/Bis A homopolymer do not. They reveal the influence of other factors on flexibility of cured systems.

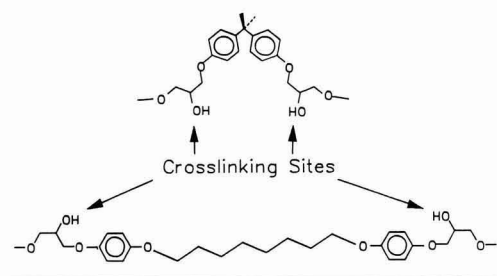
It is generally accepted that cured Bis F resins are more flexible than the corresponding cured Bis A resins. The most commonly offered explanation attributes this difference to a more restricted motion at the bridging carbon of Bis A imposed by the greater steric requirements of methyl substitution relative to the protons of Bis F.<sup>12</sup> This explanation appears to be sound for the case of the uncured resins; thus, T<sub>g</sub> for the DGEBA/p-Bis F copolymer is 18°C lower than that for the DGEBA/Bis A homopolymer. Since the Bis A is also para substituted (>97%), the only difference between the two resins is substitution at the bridging carbon. Comparison between the DGEBA/Bis F copolymer and the DGEBA/Bis A homopolymer is complicated by the fact that Bis F is a mixture of three different ortho and para isomers of methylenediphenol. On the one hand, absence of methyl substitution at the bridging carbon and the less ordered structure of the mixed isomers should contribute to backbone flexibility; but on the other hand, the presence of ortho isomers



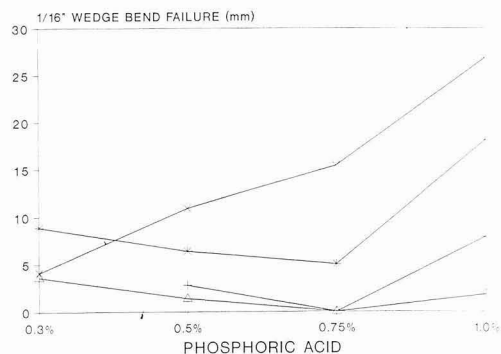
**Figure 2—Effect of the alkylene series of bridging groups, R, on coating flexibility of the advanced epoxy resins of Figure 1 cured with 1% phosphoric acid catalyst and 20% phenol/formaldehyde crosslinking agent for 10 min at 400°F**



**Figure 3**—Effect of the alkylendioxy series of bridging groups, R, on coating flexibility of the advanced epoxy resins of Figure 1 cured with 1% phosphoric acid catalyst and 20% phenol/formaldehyde crosslinking agent for 10 min at 400°F



**Figure 4**—Crosslinking sites for an advanced epoxy resin of the diglycidyl ether of bisphenol A and 4,4'-(octylene-1,8-dioxy) diphenol



**Figure 5**—Effect of concentration of acid catalyst and crosslinker on coating flexibility of the advanced epoxy resin of the diglycidyl ether of bisphenol A with 4,4'-(octylene-1,8-dioxy) diphenol cured 20 min at 400°F; (Δ) 2.5% crosslinker; (+) 5.0% crosslinker; (•) 10% crosslinker; and (×) 20% crosslinker

should restrict rotational motion around the molecular axis. The net result is a  $T_g$  nearly the same as that for the DGEBA/p-Bis F copolymer. For the cured resins, our tests show that impact wedge bend flexibility for the DGEBA/p-Bis F copolymer coating is comparable to the DGEBA/Bis A homopolymer despite the difference in methyl substitution at the bridging carbon; yet the DGEBA/Bis F copolymer coating shows a significant improvement in flexibility. These results suggest that the difference in flexibility between cured Bis F resins and cured Bis A resins is related not to the difference in substitution at the bridging carbon but to the presence of ortho/para isomers in Bis F. Other workers have also reported significant effects of isomeric structure on cured epoxy resin properties; for example, as the linearity and symmetry of the diphenol unit decreases (going from para to meta to ortho),  $T_g$  of the cured epoxy resin also decreases.<sup>15,16</sup> Also, for cured Bis F resins, Hasegawa et al., showed that the  $T_g$  decreases as the substitution pattern is changed from para,para' to ortho,para' to ortho,ortho'.<sup>17</sup>

The second series of diphenols that were evaluated in advanced copolymer resins were those bridged with alkylendioxy chains (Figure 1). All but 3,3'-(ethylene-1,2-dioxy)diphenol (m-EDDP), which is available from chemicals vendors, were prepared in a one-step process of general applicability from hydroquinone or resorcinol and the appropriate dibromoalkane. Because this process involves only one step and allows relatively easy access to either of the two isomeric forms (para or meta), evaluation of this series of diphenols is considerably more advantageous than of the comparable series of alkylene bridged diphenols. Although the ortho isomers were not prepared, they too could presumably be synthesized in a similar way from catechol.

The (alkylendioxy)diphenols were advanced with DGEBA to an EEW of approximately 2000. The meta diphenols exhibited a greater reactivity than Bis A but the para isomers were about half as reactive. The fact that the more reactive meta isomers were also more acidic is consistent with the proposed mechanism for catalyzed advancement which involves the phenate form of the diphenol.<sup>18</sup>

As we had found for the uncured alkylene bridged diphenol copolymer resins,  $T_g$ s for the uncured alkylendioxy bridged diphenol copolymer resins were all lower than the Bis A based homopolymer control (Table 1); but, in this case, they also differed from each other and in a regular fashion. Introduction of the smallest diphenol, m-EDDP (R is O(CH<sub>2</sub>)<sub>2</sub>O), produced a significant drop in  $T_g$  of about the same magnitude as that caused by introduction of the short chain alkylene bridged diphenols. Thereafter,  $T_g$  varied inversely with the square root of the length of the bridging group, in accordance with the rule of thumb for the effect of chain length on backbone flexibility.

Figure 3 shows that the effect of bridging group length on flexibility of the uncured copolymer resin does not carry over fully to the cured system. For crosslinked systems, many other factors come into play; for example, distance between crosslinking sites and flexibility of the

crosslinker. Impact wedge bend tests on the cured coatings show little difference between the DGEBA copolymers of m-EDDP and m-BDDP (3,3'-(butylene-1,4-dioxy)diphenol; R is O(CH<sub>2</sub>)<sub>4</sub>O) compared to the DGEBA/Bis A (R is C(CH<sub>3</sub>)<sub>2</sub>) homopolymer control. Only for the DGEBA copolymers of p-BDDP and p-ODDP (4,4'-(octylene-1,8-dioxy)diphenol, R is O(CH<sub>2</sub>)<sub>8</sub>O) is there a significant improvement in flexibility for a given level of crosslinker. In the cured system, crosslinking takes place at the secondary OH groups along the resin backbone. Flexibility in the case of a copolymer resin should then be severely limited by the presence of short, rigid Bis A units between every other pair of crosslinking sites. In addition to its short length and rigidity, the structure of Bis A can also play a role in holding crosslinking sites in close proximity (Figure 4). Replacement of every other Bis A unit by one that disrupts such an arrangement should improve flexibility somewhat; but large additional improvements, at least for the highly cured systems we have used to this point, may only come by replacing all the Bis A units. Thus, the approach of using copolymers of diphenols with DGEBA to evaluate the effect of the spacer group between the phenol units of the diphenols is useful, but mostly as a rapid screening tool to eliminate from further study those diphenols that exert little or no influence on the flexibility of cured systems compared to the DGEBA/Bis A homopolymer.

As discussed previously, factors other than backbone structure exert considerable influence on the flexibility of epoxy coatings cured with phenol/formaldehyde condensates; particularly those affecting crosslink density such as concentration of curing agent and acid catalyst or time and temperature of bake. Although coatings with excellent flexibility can often be obtained by minimizing one or more of those factors, for many applications the coatings must also be cured enough to exhibit some specified level of resistance to organic solvents or water or other chemical agents. In the can coating industry, relative degree of cure is often expressed as resistance to MEK double rubs.<sup>19</sup> Perhaps the levels of crosslinker used so far in this report (20%) were too high for many applications and gave such rigid networks that the effect of backbone flexibility was largely overwhelmed.

In an effort to determine how well the copolymers might perform at roughly optimum levels of acid and curing agent, a series of formulations of the p-ODDP copolymer were prepared and cured as usual. Figure 5 shows a plot of wedge bend failures against acid level for four different levels of METHYLON 75108 curing agent. A similar plot for the MEK double rubs is shown in Figure 6. One of the more prominent features of the wedge bend plot is the difference in appearance between the formulation with the highest level of curing agent, a roughly linear response of wedge bend failure to acid level, and all the others which show a pronounced dip or minimum at the 0.75% acid level. Another feature shows a substantially greater effect of acid level on flexibility than of curing agent, at least at the high acid levels. A 25% decrease in acid level (going from 1.0 down to 0.75%) results in a two- to threefold drop in wedge bend failure for the two highest levels of curing agent where

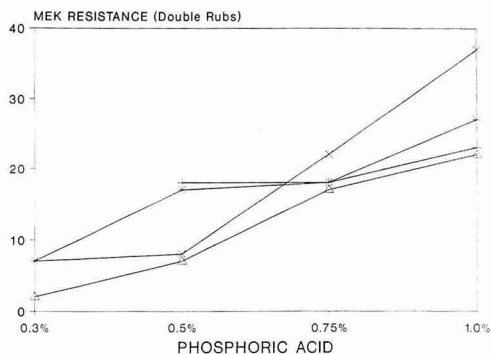


Figure 6—Effect of concentration of acid catalyst and crosslinker on MEK resistance of the advanced epoxy resin of the diglycidyl ether of bisphenol A with 4,4'-(octylene-1,8-dioxy) diphenol cured 20 min at 400°F; (Δ) 2.5% crosslinker; (+) 5.0% crosslinker; (•) 10% crosslinker; and (x) 20% crosslinker

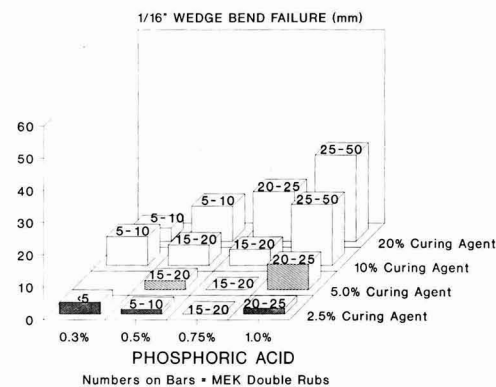


Figure 7—Effect of concentration of acid catalyst and crosslinker on coating flexibility and MEK resistance of the advanced epoxy resin of the diglycidyl ether of bisphenol A with 4,4'-(octylene-1,8-dioxy) diphenol cured 20 min at 400°F

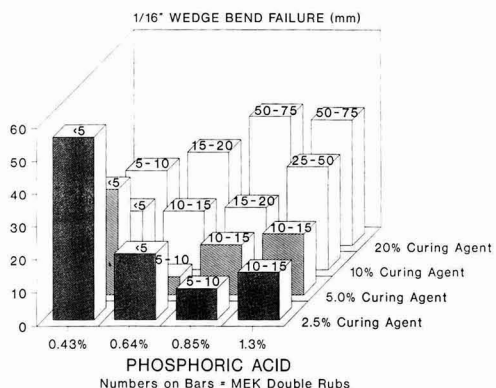


Figure 8—Effect of concentration of acid catalyst and crosslinker on coating flexibility and MEK resistance of the advanced epoxy resin of the diglycidyl ether of bisphenol A with bisphenol A cured 10 min at 400°F

the multiplication factor can be determined. On the other hand, a twofold variation in level of curing agent is required to produce a two- to threefold change in flexibility. A feature that the plot does not show may be the key to interpreting the results. The coatings exhibited two different kinds of wedge bend failure. Those to the right and above the dip in the plot gave a continuous failure along the stressed edge of the wedge bend; while those to the left and below the dip gave spotty, noncontinuous failures. Those at or near the dip sometimes gave one and at other times gave the other or both of the two kinds of failures. The dip then represents a dividing line between two different mechanisms of failure.

Inspection of the plot for MEK resistance may provide an explanation for the dip in the wedge bend plot. In this case, MEK resistance roughly parallels both acid and curing agent levels; thus, the highest levels of both acid and curing agent give the highest MEK resistance. At the 0.3% acid level, none of the formulations were cured sufficiently to give even the minimum amount of 15 to 20 MEK double rubs; whereas, at the high level of acid all the coatings were cured beyond an MEK resistance of 20 double rubs. At the 0.75% acid level, all the coatings gave MEK double rubs in the range of 15 to 25. This line corresponds closely to the dividing line between the two different kinds of wedge bend failure shown in *Figure 5*. Apparently, the wedge bend failures for those coatings to the left of the dividing line are representative of under-cured epoxy resin, and those to the right of the dividing line are representative of cured or over-cured epoxy resin. When the wedge bend and MEK resistance data were combined in one three-dimensional plot (*Figure 7*), the optimum balance of minimum wedge bend and maximum crosslinking was revealed as a well in the response curve at the 0.75% acid level and 2.5 to 5% curing agent level. This conclusion was further supported by TMA measurements of  $T_g$  on the coatings compared to  $T_g$ s obtained by DSC on the uncured resins. For example,  $T_g$ s for several samples of uncured DGEBA/Bis A homopolymer ranged between 78 and 83°C. The coatings with low acid and curing agent levels gave  $T_g$ s in the range of 79 to 87°C, remarkably close to the uncured resin, while those with high levels of acid and curing agent give  $T_g$ s in the range of 87 to 97°C.

Although an acid/curing agent study for the DGEBA/Bis A homopolymer resin exhibited a pattern similar to that of the DGEBA/p-ODDP copolymer resin (a dip or well in the plot representing optimum conditions) (*Figure 8*), the wedge bend failures are all greater than for the corresponding formulations of the DGEBA/p-ODDP copolymer resin. Comparison of *Figure 7* to *Figure 8* also reveals that the ratio of wedge bend failures for the homopolymer to the copolymer are greater at the lower levels of crosslinker and acid catalyst, suggesting a more pronounced effect of backbone flexibility on coating flexibility at the lower levels of crosslinking.

## CONCLUSIONS

The effect of variations in the structure of spacer groups bridging the phenol units of diphenols on proper-

ties of the epoxy resins made from them have been investigated. The resins were copolymers made by advancing the diphenols with the diglycidyl ether of bisphenol A. The use of copolymers rather than the homopolymers allowed for more rapid screening of systematic variations in the diphenols. With the exception of the DGEBA/p-BP copolymer, the uncured copolymer resins exhibited significantly greater backbone flexibility, as indicated by  $T_g$  measurements, than the DGEBA/Bis A homopolymer resin. Substitution of every other Bis A unit in the homopolymer backbone with diphenols that have relatively short bridging units such as p-CH<sub>2</sub>, o/p-CH<sub>2</sub>, p(CH<sub>2</sub>)<sub>2</sub>, or O(CH<sub>2</sub>)<sub>2</sub>O disrupts the symmetry of the resin backbone relative to the DGEBA/Bis A homopolymer sufficiently to produce a 16 to 22°C drop in  $T_g$ . Thereafter,  $T_g$  varies inversely with the square root of the bridging group chain length. At the relatively high level of crosslinking agent of 20%, the trend of backbone flexibility in the uncured resins is largely overwhelmed by the rigidity of the crosslinked system. Thus, although the DGEBA copolymers of p-biphenol, p-Bis F, and m-EDDP have  $T_g$ s ranging from 4°C higher to 22°C lower than the DGEBA/Bis A homopolymer, they all give coatings of comparable flexibility. It is only those copolymers with diphenols that can disrupt the regular arrangement of OH crosslinking sites characteristic of the DGEBA/Bis A homopolymer (e.g., Bis F which is a mixture of all three ortho/para isomers of methylenediphenol and p-EDP with a para-substituted two carbon bridge and the para-substituted alkylenedioxy bridged diphenols) that reveal about a twofold decrease in wedge bend failure. Additional improvements in flexibility for the copolymer resins are apparently limited both by the relative rigidity of the Bis A unit and the extent of crosslinking. However, the use of such copolymer resins even at the relatively high levels of crosslinking can still serve as a screening tool to select diphenols for further study. Large improvements in coating flexibility can be realized by optimizing the extent of crosslinking. Wedge bend failures for the DGEBA/p-ODDP copolymer plotted against the concentrations of both acid catalyst and curing agent showed a dip in the three-dimensional response curve down to or near a zero value corresponding to a level of cure that gives at least 15 to 25 MEK double rubs. For acid and curing agent levels below that point, MEK double rubs and  $T_g$  measurements reveal undercuring and wedge bend failures often increase again. Although the DGEBA/Bis A homopolymer exhibited similar behavior, it was much less flexible than the copolymer at all levels of acid and curing agent.

## ACKNOWLEDGMENTS

The authors wish to acknowledge Don Benton for analytical support and Allyson Malzman for her help in all phases of the experimental operations.

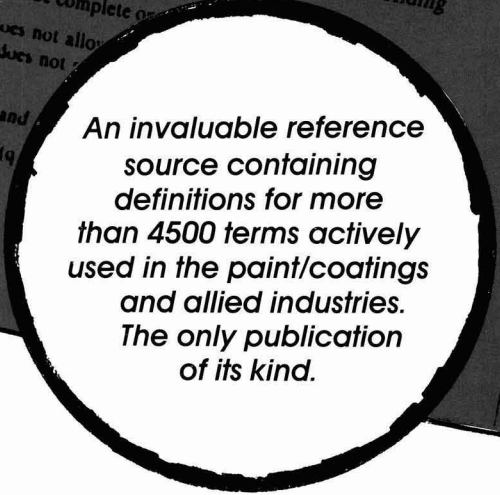
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# Strongly Adherent, Smooth Coatings Of Polypyrrole Oxalate on Iron

F. Beck and R. Michaelis  
Universität Duisburg\*

Black polypyrrole coatings on iron can be obtained through anodic electrodeposition from aqueous electrolytes containing the monomer and oxalic acid. The adherence strength of the layers is larger than  $11.5 \text{ N mm}^{-2}$  for  $1 \mu\text{m}$  coatings. Surface roughness is found to be relatively low in comparison to other polypyrrole salts, even at thicker layers. The polymer films are essentially nonporous. A pronounced leveling action for the electrodeposition was found. Overcoating by cathodic or anodic electrodeposition of paint was demonstrated. The application of these polypyrrole coatings as corrosion inhibiting polymer interlayers, eventually substituting phosphatation, is proposed.

## INTRODUCTION

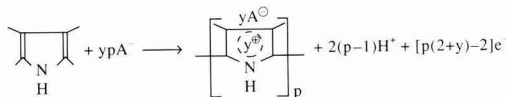
Substantial improvement over conventional methods of industrial painting of metal substrates was achieved in the 1960s with the introduction of electrodeposition of paint. The success of this method is founded on the unique properties of the electrical process. In principle, instead of starting with prefabricated waterborne polymers, it would be particularly interesting to use appropriate monomers, which undergo a film forming electropolymerization.

Synthetic metals like polypyrrole have been known for more than a decade. Electrodeposition at inert anodes like Pt, Au, GC, or stainless steel yields coherent coatings which can be peeled off to give free standing films of the conducting polymer.<sup>1</sup> The continuous fabrication of a conducting polymer band from a slowly rotating drum anode is referenced.<sup>2</sup> These methods are of great interest for the subsequent study of the material properties.

Film forming electropolymerization of pyrrole ( $\text{C}_4\text{H}_5\text{N}$ ) proceeds at an anodically polarized electrode surface. The overall reaction can be written in a simple manner as follows ( $p$  = degree of polymerization):



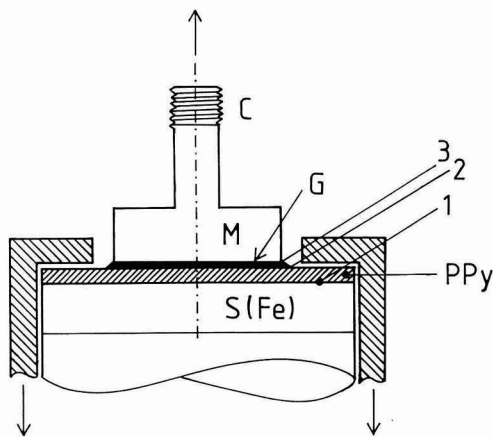
One propagation step releases two protons, which are due to the  $\alpha, \alpha'$ -hydrogens of the pyrrole molecule, and two electrons. However, this equation neglects the simultaneous insertion of anions  $\text{A}^-$  in the course of further oxidation of polymer chains to yield polymer radical cations, and it is only valid for  $p \longrightarrow \infty$ . Thus, a complete description is given in the following formulation:



where  $y$  is the degree of insertion of the anions, the degree of oxidation of the monomer units. The theoretical electrochemical equivalent  $m_{e,\text{th}}$ , used in equation (1) is derived from this equation, but again for  $p \longrightarrow \infty$ .

It was discovered recently that it is also possible to electrodeposit polypyrrole, etc., on un-noble commodity metals. In this case, the bath and the conditions for electrodeposition must be thoroughly considered to avoid the anodic dissolution of the substrate. Only a few systems are known to meet these criteria for polypyrrole electrodeposition on iron, namely, propylenecarbonate with quarternary ammonium salts,<sup>3,4</sup> aqueous nitrate,<sup>5</sup> and aqueous oxalic acid.<sup>6</sup> The aqueous electrolytes are preferable from a practical point of view. A possible application of these coatings could be direct corrosion protection, for these materials are insoluble in all solvents. Moreover, a functional device as a corrosion inhibiting polymer (inter)layer (CIPL) can be envisaged, for

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**Figure 1**—Experimental design for the measurement of adherence strength  $\tau$ . M = steel cylinder, A = 0.5 cm<sup>2</sup> (sandblasted), C = connection to the draw-up spindle, G = cyanacrylate glue, PPy = polypyrrole layer, S = substrate; phase boundaries: (1) iron/PPy, (2) PPy/G, and (3) G/M

the heterocyclic monomer units have essentially the same structures as many efficient corrosion inhibitors.

However, such an application depends on the realization of strong adhesion of the polymer layer, with a thickness of typically 0.1–30  $\mu\text{m}$ . We have discovered that this quality is extremely good with the previously mentioned oxalate system on iron.<sup>6</sup> Moreover, the surface roughness is extraordinarily low, even for thicker coatings. In this paper, we will provide more quantitative data with regard to these two technical properties. Polypyrrole was chosen because it is a nontoxic material which can be electrodeposited under a wide variety of conditions.<sup>7–9</sup>

### Experimental

The electrolytes were oxalic acid and  $\text{KNO}_3$ , both from Merck, analytical grade, dissolved in bidistilled water. Pyrrole (Merck, for synthesis) was distilled under Ar just before use. The iron substrate was a 1 mm sheet of low carbon content steel, St 37 from Thyssen AG, Duisburg.

The coated surface area of the iron strips was  $2 \times 6 \times 2.5 = 30 \text{ cm}^2$ . Pretreatment of the iron surface was as follows: wet grinding with Artifex SC 250 (Artifex, Hamburg, grain size of SiC: 45  $\mu\text{m}$ ), then degreasing with tetrachloroethene.

Electrodeposition of polypyrrole on iron was performed galvanostatically under conditions described in the following paragraph. Adherence strength,  $\tau$ , of the polymer coatings was measured with a homemade machine. A cylindrical body made of St 37 with a sandblasted front surface area of 0.5 cm<sup>2</sup> was glued to the polymer surface with the aid of a cyanacrylate glue (No. 380 from Loctite). After 72 hr hardening at 23°C, the probe was mounted in the test machine. The upper part of the

device, which is depicted schematically in *Figure 1*, was connected to a draw-up spindle, which was able to apply exactly coaxial forces up to 1000 N perpendicular to the sheet plane. The forces were measured with dynamometers of MWT Mess- und Wiegetechnik, Typ 127, with measuring ranges of 1–100 and 10–1000 Newton, respectively.

Surface roughness, R, was measured with a commercial mechanical profilometer from Feinprüf GmbH, Göttingen (Perthometer S6P). A tracing system was used throughout, which was optimum for the present problem. A total of three to six measurements were performed on the same probe along a line of about 1 cm at various locations on the polypyrrole surface.

## RESULTS

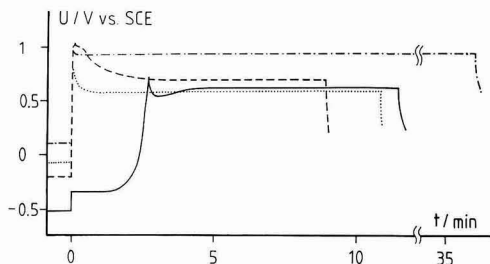
### Electrodeposition

Polypyrrole layers were electrodeposited galvanostatically on iron substrates. In some cases, a smooth platinum sheet was employed for comparison. The electrodeposition was performed at 20°C, in a nonstirred bath, up to a nominal thickness of  $d_n = 3 \mu\text{m}$ .<sup>10</sup> The general definition of this quality is given in reference (10).

$$d_n = \frac{\gamma Q_A m_{c,th}}{\rho} = \frac{\gamma Q_A (M_M + y M_A)}{\rho F (2 + y)} \quad (1)$$

where  $Q_A$  is the surface area specific charge for electropolymerization,  $\rho$  is the density of the compact material,  $M_M$  and  $M_A$  are the molecular weights of the monomer unit and the anion, respectively,  $F$  is the Faraday constant, and  $y$  is the degree of insertion. The real thickness is larger than  $d_n$  for porous layers, but it is below  $d_n$  for nonquantitative current efficiency  $\gamma$ . Other thicknesses in the range of 0.3–30  $\mu\text{m}$  were studied as well. The standard conditions are listed in *Table 1*. Primarily two aqueous electrolytes were used, both described in previous work: oxalic acid<sup>6</sup> and  $\text{KNO}_3$ .<sup>5</sup> The doped polypyrroles arising from this are denoted in the following by PPy-Ox and PPy-Nt, respectively. In both cases, coherent polypyrrole layers are accomplished, but they differ widely in many aspects, as shown in the following. For comparison, the "classical" bath with wet acetonitrile<sup>1,7–9</sup> was used. It should be noted that the current efficiencies for system No. 1, PPy-Ox from  $\text{H}_2\text{C}_2\text{O}_4$ , were calculated on the basis of the real charge flowing into the film forming electropolymerization, and the real polymer mass, corrected by the initial anodic dissolution of iron as cited in reference (28).

*Figure 2* shows typical potential/time-curves for four different electrodepositions according to *Table 1*. The most positive steady state potential is found for the "Diaz-system" No. 4 at platinum, the conditions of which could not be applied to iron.<sup>5</sup> The aqueous systems Nos. 2 and 3, sodium oxalate and potassium nitrate, display a more negative steady state potential, but both lead, as No. 4, to an instantaneous passivation of the iron after switching on the current. Unfortunately, this is not the case for oxalic acid (No. 1), and it is only after an induction period that passivation is established. This very promising system therefore has a drawback in that the bath is contaminated by iron, which is dissolving anodi-



**Figure 2—Potential/time-curves for the galvanostatic electrodeposition of polypyrrole, (Table 1),  $d_n = 3 \mu\text{m}$ . 0.1 M  $\text{H}_2\text{C}_2\text{O}_4$ ,  $j = 2 \text{ mA/cm}^2$ , at Fe; — — — 0.1 M  $\text{Na}_2\text{C}_2\text{O}_4$ ,  $j = 2 \text{ mA/cm}^2$ , at Fe; ······ 0.1 M  $\text{KNO}_3$ ,  $j = 2 \text{ mA/cm}^2$ , at Fe; and - · - · - 0.1 M  $\text{NBu}_4\text{BF}_4(\text{MeCN})$ ,  $j = 0.5 \text{ mA/cm}^2$ , at Pt**

**Table 1—Galvanostatic Electrodeposition of Polypyrrole under Various Conditions. Standard Data. Pyrrole Concentration was 0.1M Throughout**

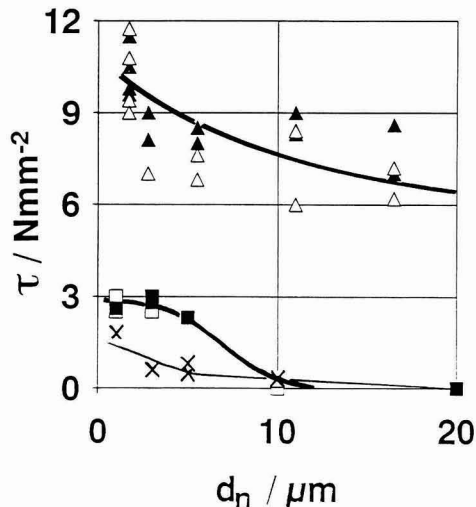
No.	Electrolyte Composition	$j/\text{mAcm}^{-2}$	PPy-type	CE/%	S
1	0.1M $\text{H}_2\text{C}_2\text{O}_4$ in $\text{H}_2\text{O}$	2	PPy-Ox	70 <sup>a</sup>	Fe (Pt)
2	0.1M $\text{Na}_2\text{C}_2\text{O}_4$ in $\text{H}_2\text{O}$	2	—	80	Fe
3	0.1M $\text{KNO}_3$ in $\text{H}_2\text{O}$	2	PPy-Nt	90	Fe (Pt)
4	0.1M $\text{NBu}_4\text{BF}_4$ (TBAT) in acetonitrile, 0.05M $\text{H}_2\text{O}$	0.5	PPy- $\text{BF}_4^-$ (Diaz)	90	Pt

(a) Calculated with respect to the charge after the induction period.

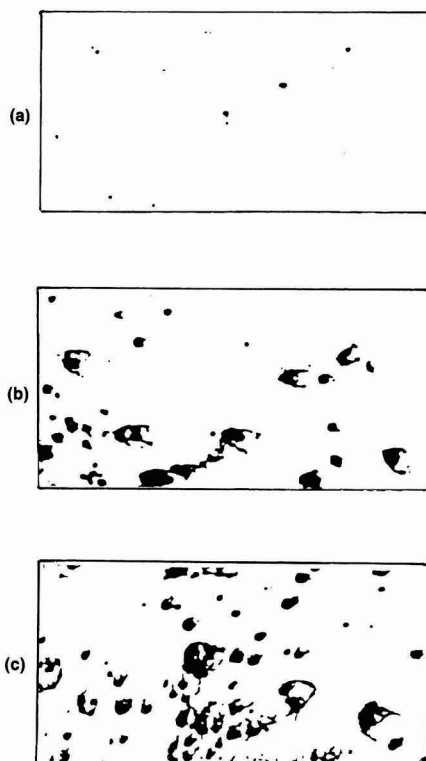
**Table 2—Adherence Strength of Polypyrrole Layers at Steel Surfaces (St 37). Standard:  $T = 23^\circ\text{C}$ ,  $d_n = 3 \mu\text{m}$ , and electrolyte concentration = 0.1M**

Parameters	$\tau / \text{Nmm}^{-2}$ (MPa)
Variation of electrolyte composition at 0.1M Py	
0.1M $\text{KNO}_3$	1
0.1M $\text{Na}_2\text{C}_2\text{O}_4$	0.2
0.1M $\text{H}_2\text{C}_2\text{O}_4$	>11.5
0.1M $\text{H}_2\text{C}_2\text{O}_4^a$	4
Variation of pyrrole concentration at 0.1M $\text{H}_2\text{C}_2\text{O}_4$	
0.1M Py	>11.5
0.2M Py	11
0.4M Py	5
0.8M Py	3.3
Variation of oxalic acid concentration of 0.8M Py	
0.1M $\text{H}_2\text{C}_2\text{O}_4$	3.3
0.05M $\text{H}_2\text{C}_2\text{O}_4$	2.2
0.02M $\text{H}_2\text{C}_2\text{O}_4$	5
0.01M $\text{H}_2\text{C}_2\text{O}_4$	6
Variation of temperature at 0.4M Py and 0.1M $\text{H}_2\text{C}_2\text{O}_4$	
0°C	4.5
23°C	5
40°C	6.2

(a) Prepassivation of iron with manganese oxide.



**Figure 3—Adherence strength,  $\tau$ , vs nominal thickness,  $d_n$ , of polypyrrole layers on various substrates.  $\Delta$ ,  $\blacktriangle$ : PPy-Ox at Fe;  $\square$ ,  $\blacksquare$ : PPy-Nt at Fe; X: PPy- $\text{BF}_4^-$ , at Pt (Diaz,  $\text{Br}^-$ -catalysis); and  $\Delta$ ,  $\square$ : after leaching 30 min, boiling MeOH**



**Figure 4—FerroxyI-porettest, as described in the text (after ISO/DIS-Standard, No. 4527, p 16). Elongations: (a)  $\epsilon = 0$ ; (b)  $\epsilon = 0.5\%$ ; and (c)  $\epsilon = 2.0\%$**

**Table 3—Roughness Factor,  $R_z$ , of Various Metal Surfaces as Test Systems**

Entry	System	$R_z / \mu\text{m}$
1.....	Smooth platinum sheet	0.9..... 1.5
2.....	Stainless steel, wet polishing with Artifex SC250 (SiC, grain size 45 $\mu\text{m}$ )	1 ..... 1.6
3.....	Iron, polished with diamond spray 1 $\mu\text{m}$	1.3..... 2.4
4.....	Iron, wet polishing with Artifex SC250 (see No. 2)	7.5..... 8.1
5.....	Sandblasted stainless steel, SiO <sub>2</sub> particles, 70-100 $\mu\text{m}$	14.2.....16.3

cally in the course of the induction period. The problem can be overcome by pre-passivation using a thin manganese oxide layer.<sup>11</sup>

**Adherence Strength  $\tau$**

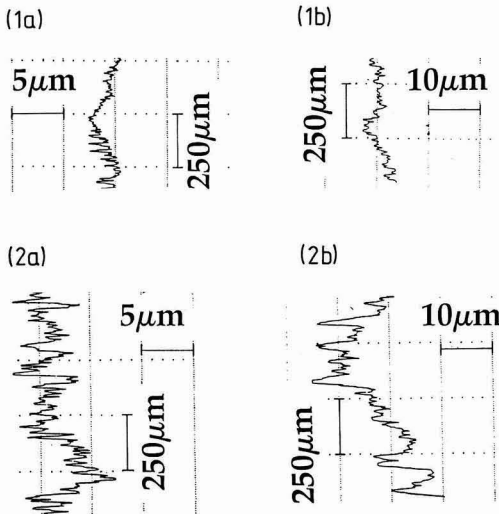
A measure of the adhesion of the electrodeposited polymer layer to the substrate,  $\tau$ , was determined by a force applied perpendicularly to the substrate to detach the coating. Table 2 provides some characteristic data. They show the influence of the electrolyte, the concentrations of pyrrole and of oxalic acid, and the temperature during electrodeposition. Clearly, the highest  $\tau$  can be obtained with the oxalic acid electrolyte, while the other electrolytes are much inferior. In most cases, breakoff occurred at the phase boundary (No. 1 in Figure 1), and a blank, circular round spot of the metal substrate was left behind. The PPy surface originally contacting the iron looked very smooth. Interestingly, polypyrrole oxalate/iron with a maximum  $\tau$  even exhibited in some cases a

breakoff at the phase boundary (No. 3 in Figure 1), thus demonstrating the similarity of both values, the latter being typical for the high standard of metal/epoxide or cyanacrylate resin adhesion. Table 2 shows that  $\tau$  decreases with increasing concentrations of the monomer and of the oxalic acid. It increases slightly with increasing temperature. It should be noted that the previously mentioned measurement could only be performed with PPy-layers deposited at higher monomer concentrations, for in this case a measurable range of  $\tau$  values would not have been possible.

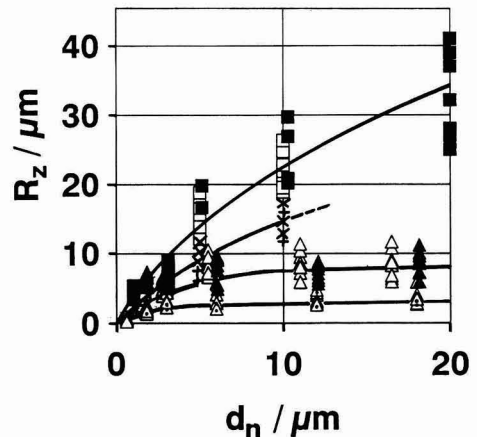
The exceptional behavior of the oxalic acid electrolyte is also demonstrated in Figure 3, where the dependency of  $\tau$  on the thickness of the coating is shown. The polypyrrole oxalate on iron coatings not only has this high  $\tau$  at thin layers, but  $\tau$  values decrease only slightly up to 20  $\mu\text{m}$ . There are only minor effects of a leaching for 30 min in boiling methanol. In contrast, the same coating on platinum has only a low  $\tau$ , namely about 1 N mm<sup>-2</sup> at 3  $\mu\text{m}$ , which decreases to about 0.4 N mm<sup>-2</sup> at 30  $\mu\text{m}$ . The other electrolytes yield only very low values for  $\tau$ , and the initial value decreases rapidly with increasing nominal thickness,  $d_n$ . There is practically no difference in  $\tau$  for iron or platinum in the case of the nitrate system. The Diaz coatings in electrolyte 4 in Table 1 have an even lower  $\tau$ , and it decreases to virtually zero at medium thicknesses. In this case, foils can be easily peeled off from the substrate.

The strong adherence of polypyrrole oxalate to iron is of great importance for any application in terms of corrosion protection. It would then play the same role as the paint coatings in conventional systems, where this was repeatedly demonstrated.<sup>12-15</sup>

If the iron strip, which is coated by a strongly adherent PPy-Ox layer of  $d_n = 6 \mu\text{m}$ , is drawn in a tensile test machine around the apparent limit of elasticity, the coating is able essentially to follow this deformation without



**Figure 5—Direct surface profiles, measured with a Perthometer, for polypyrrole layers on iron. (1) PPy-Ox—(a)  $d_n = 3 \mu\text{m}$ ; and (b)  $d_n = 11 \mu\text{m}$ . (2) PPy-Nt—(a)  $d_n = 3 \mu\text{m}$ ; and (b)  $d_n = 10 \mu\text{m}$**



**Figure 6—Averaged roughness depth,  $R_z$ , vs nominal thickness,  $d_n$ , of polypyrrole layers on various substrates. ■, □: PPy-Nt, at Fe; X, +: PPy-BF<sub>4</sub><sup>-</sup>, at Pt (Diaz); ▲, △: PPy-Ox, at Fe; △, △, +: after leaching, 30 min, boiling MeOH**



cracking. *Figure 4* demonstrates that no cracks developed, as indicated by the "ferroxyl test." A wet filter paper, impregnated with hexacyanoferrate-III, is pressed against the layer. Any contact with iron would be detected by the development of an intensive blue spot of Berlin blue. The only effect is a slight increase in the number of pores, going down to the PPy/Fe-interface. This holds for up to 2% elongation. It seems that the coating is lengthened, together with the steel, and no detachment occurs. The adhesion is so excellent that no detachment is observable after bending a PPy coated iron strip by about 180°. Even cooling down in liquid nitrogen and then bending by about 180° does not lead to any cracks or detachment.

### Surface Roughness

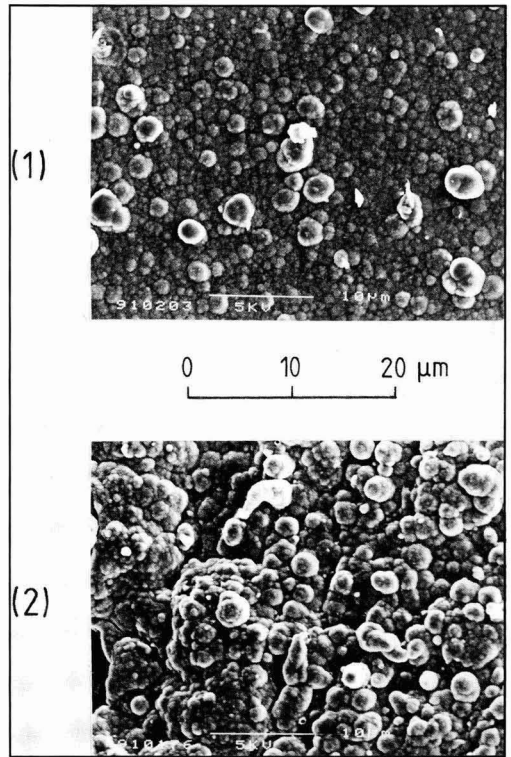
The PPy-Ox/Fe-system was found to develop relatively smooth surfaces, even at higher thicknesses of the polypyrrole layer. This is demonstrated in *Figure 5* by the "Perthometer" profiles for two different thicknesses. They were measured by the mechanical profile stylus tracing method. For comparison, the same procedure is shown for polypyrrole on iron with a nitrate doping. In this case, surface roughness is increased by a factor of four for the  $d_n = 3 \mu\text{m}$ -layer, and it is furthermore enhanced for the thicker coating.

*Figure 6* summarizes the results for the average roughness depth,  $R_z$ , as a function of the nominal thickness,  $d_n$ , for various systems.  $R_z$  is the average of five individual roughness depths,  $\Delta R$ , measured along five single lines, typically  $l = 1 \text{ cm}$  in five measurements.  $\Delta R$  is the difference between the highest and the lowest peaks, that is, the largest peak to valley height for each individual measurement. Thus,  $R_z$  is defined as

$$R_z = 1/n \sum_{i=1}^n \Delta R_i \quad (n=5) \quad (2)$$

The measurement was performed with a mechanical free tracing system where a skid was also used. The tip radius was  $5 \mu\text{m}$ , with a tip angle of  $90^\circ$  and a force of 80-100 mN. The measuring unit was calibrated with various systems with a roughness which can be partially defined (*Table 3*). These results demonstrate the feasibility of the method.

As shown in *Figure 6*, the polypyrrole layers are in general relatively rough. While  $R_z$  extends to the true thickness in the case of PPy-Nt, where it is about 200-300% of  $d_n$  (see *Figure 9*), it is about 50-100% of the true thickness with PPy-Ox. However, with the nitrate system, it increases continuously with increasing nominal



**Figure 7**—SEM micrograph for polypyrrole on iron  $d_n = 3 \mu\text{m}$ , 2700 fold magnification. (1)—PPy-Ox; and (2)—PPy-Nt

thickness, whereas it attains a constant value at higher  $d_n$ s for the oxalate system. As expected, this is virtually unchanged if the iron substrate is substituted with a smooth platinum foil, as shown for the oxalate samples in *Figure 6*. The conventional Diaz system (see Entry 4 in *Table 1*) is nearly as rough as the nitrate system (*Figure 6*).

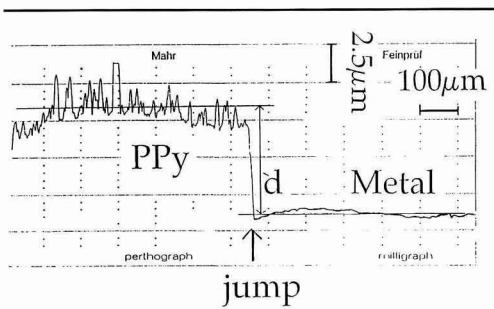
Another convenient method for expressing surface roughnesses is the so-called average roughness,  $R_a$ . It averages *all* roughness elongations, starting from an average straight line, where the micro elongations rise in positive and negative directions. *Table 4* presents typical  $R_z$ - and  $R_a$ -data.  $R_a$ -values are much lower. They provide an overall view of the surface. However, information regarding the extremes is totally lost. Therefore *both*

**Table 4**—Surface Roughness Data for Polypyrrole Coatings Without Solvent Extraction. Average of Five Measurements

No.	System (Electrolyte)	Anode	$d_n / \mu\text{m}$	$R_z / \mu\text{m}$	$R_a / \mu\text{m}$
1. ....	0.1M $\text{H}_2\text{C}_2\text{O}_4$	Fe	16.5	9.3	1.1
	0.1M $\text{H}_2\text{C}_2\text{O}_4$	Fe	2.8	6.5	0.7
2. ....	0.1M $\text{KNO}_3$	Fe	20	33	7.2
	0.1M $\text{KNO}_3$	Fe	3	7.3	1.0
3. ....	0.1M TBAT in MeCN	Pt	10	18	2.2
	0.1M TBAT in MeCN	Pt	3	6	0.5

**Table 5**—Porosities, P (%), Derived from Equation (3) (True and Nominal Thickness) for  $\text{BF}_4^-$ -Doped Diaz-Polypyrrole Layers

Conditions	$d_n / \mu\text{m}$		
	1	5	20
Uncatalyzed, no leaching	29	46	69
Uncatalyzed, leaching	16	29	44
Catalyzed ( $\text{Br}^-$ ), no leaching	25	42	56
Catalyzed ( $\text{Br}^-$ ), leaching	5	10	30



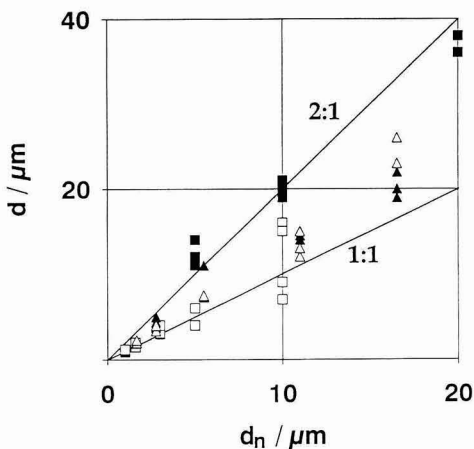
**Figure 8—Determination of the true thickness, *d*, of a polypyrrole layer on polished iron according to the jump method. PPy-Ox, *d<sub>n</sub>* = 5.5 μm, and *d* = 7.4 μm**

data must be used to obtain a complete characterization of surface roughnesses.

These findings are confirmed by the SEM micrographs shown in Figure 7. They display the typical cauliflower textures found frequently for polypyrrole layers. However, the nodular structures are much coarser for the nitrate system than for the oxalate system. The latter emerge initially from a very smooth polypyrrole first layer.

**True Thickness, Porosity, and Leveling Action**

The nominal thickness, *d<sub>n</sub>*, was calculated with the density  $\rho = 1.5 \text{ g/cm}^3$  of the compact material, using equation (1). The true thickness, *d*, of the layers can be measured with the “jump method.” In this case, the same tracing system was applied, but without a skid. Figure 8 shows the result of such a measurement. Prior to electrodeposition, a part of the polished metal surface was shielded by a PTFE-ribbon. A step arises, and the tracing systems “jump” from the polypyrrole surface



**Figure 9—Correlation of nominal thickness, *d<sub>n</sub>*, to real thickness, *d*, for polypyrrole layers on iron. ■, □: PPy-Nt; ▲, △: PPy-Ox; and □, △: after leaching, 30 min, boiling MeOH**

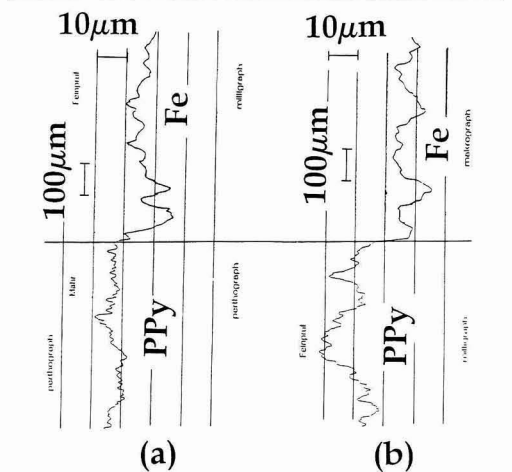
down to the blank metal surface. The reliability of the jump method was controlled by various layers of exactly defined thickness, for example, Mylar foils in the range of 1-10 μm thickness. The result was a very close 1:1 correlation between the true and the measured thickness parameters.

Figure 9 depicts a correlation of these true thickness data, *d*, versus the nominal thickness, *d<sub>n</sub>*. The PPy-Ox layers follow essentially the 1:1 correlation, and from this we conclude that the porosity of these coatings must be low. However, the opposite is the case for PPy-Nt. Here the 2:1 correlation is approached, and this means a porosity of 50%. Interestingly, this is only true for the pristine layer (after a short rinse). If the coating is leached in boiling methanol for 30 min it seems to shrink, and appreciably lower *d*-values were found. It is possible to derive the porosity, *P*, of the layers with the aid of the simple relation

$$P = 1 - \frac{d_n}{d} \tag{3}$$

Some data are compiled in Table 5 for polypyrrole layers after Diaz, evaluated by this method. Three effects can be easily identified: (1) *P* increases with increasing *d<sub>n</sub>*. The new data agree well with other values derived formerly with an optical method.<sup>16</sup> (2) *P* decreases after leaching in boiling methanol for 30 min, and this must be discussed in terms of a shrinkage after the elimination of occluded electrolytes, predominantly acids. (3) Electrocatalytic deposition of polypyrrole, in the presence of small concentrations of bromide,<sup>17-19</sup> leads to a more compact material.

To our surprise, we were able to identify a pronounced leveling action. Figure 10 demonstrates that this was found for the PPy-Ox system on iron, but not for PPy-Nt. The effect resembles phenomenon, the well known leveling in electrodeposition of metals, where the transport of



**Figure 10—Leveling action in the course of electrodeposition of polypyrrole PPy-Ox at iron, abraded with “Artifex 250.” (a)—*d<sub>n</sub>* = 5.5 μm, PPy-Ox. For comparison: (b)—*d<sub>n</sub>* = 10 μm, PPy-Nt**

inhibitors plays the decisive role,<sup>20</sup> but actually, we know nothing about the present mechanism.

### Bulk Properties

**Electronic conductivity** of hydrogenoxalate doped polypyrrole is relatively low, as shown in Table 6. It is four orders of magnitude below the value for Diaz-polypyrrole. The nitrate doped material exhibits a medium level of  $\kappa$ . A strong decrease of  $\kappa$  for the oxalate system with time of exposure was observed.<sup>21</sup> The  $\kappa$ -measurement was performed by the four probe method on free standing films originally electrodeposited on smooth platinum electrodes. It should be noted that such low values are not a limiting factor for overcoating by electrodeposition of paint, as shown in the Discussion section.

**Tensile strength** of free standing PPy-Ox films (16.5  $\mu\text{m}$ ) was found to be about 10  $\text{Nmm}^{-2}$  (MPa). This is of the order of other results for polypyrrole films reported in the literature.<sup>22-25</sup> The films adhered so strongly to the iron substrate that the only way to remove the iron (in this case a 0.1 mm thin foil of pure iron) was by dissolution in warm half-concentrated HCl.

The **infrared spectrum**, measured by the IRRAS-method<sup>26,27</sup> shows a pronounced effect for the PPy-Ox system on iron (Figure 11). It should be noted that the same system on platinum agrees well with the standard, which is also found for PPy-Nt on platinum as well as on iron. The standard is distinguished by the total absence of any bands characteristic of the dopant anion. This is attributed to steric hindrance of the corresponding atomic oscillations. The effect of the iron substrate may be connected with the presence of iron oxalate at the interface, and this point will be further examined in the Discussion section. However, the main absorption band of Fe-II-oxalate cannot be identified again in the IRRAS profile, as shown in Figure 11.

### DISCUSSION

Polypyrrole layers can be electrodeposited on iron from two aqueous electrolytes. One contains nitrate and was the result of a screening of more than 20 inorganic electrolytes.<sup>5</sup> The other electrolyte was oxalic acid, and this was the only successful one emerging from a screening of about 20 organic acids and their salts.<sup>6,28</sup> Only the latter system, PPy-Ox/Fe, exhibited the unique coating properties described in this paper. It should be stressed that the layers consist of doped polypyrrole. The degree of insertion of nitrate ions is  $y = 0.22$ ,<sup>5</sup> while  $y = 0.30$  was found for oxalic acid.<sup>29</sup> Presumably, the hydrogen oxalate monoanion must be regarded as the dopant.<sup>30</sup>

The state of the interface, PPy-Ox/Fe (No. 1 in Figure 1), is responsible for the adherence of the polymer layer. We identified very high values for the adherence strength, which is of the same order as typical metal adhesives, epoxide, and cyanoacrylate resins. An interlayer of iron oxalate was identified as a thin interlayer at the interphase, Fe/PPy-Ox, using the XPS method,<sup>28</sup> and this may be the reason for the strong adherence. It can be compared to zinc/iron-phosphates, which improve adherence of paint coatings on iron in conventional sys-

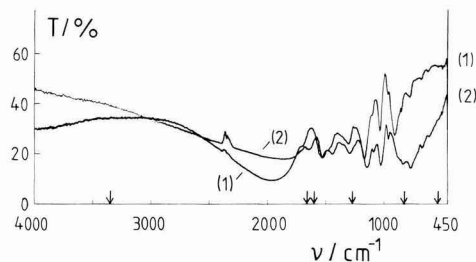


Figure 11—IRRAS measurements (80° fixed angle) for polypyrrole layers ( $d_n = 1 \mu\text{m}$ ) on iron. (1)—Fe/PPy-Nt,  $d_n = 1 \mu\text{m}$ ; and (2)—Fe/PPy-Ox,  $d_n = 0.6 \text{ mm}$ . The arrows indicate strong absorption bands of Fe-II-oxalate (anhydrous and with 2 H<sub>2</sub>O)

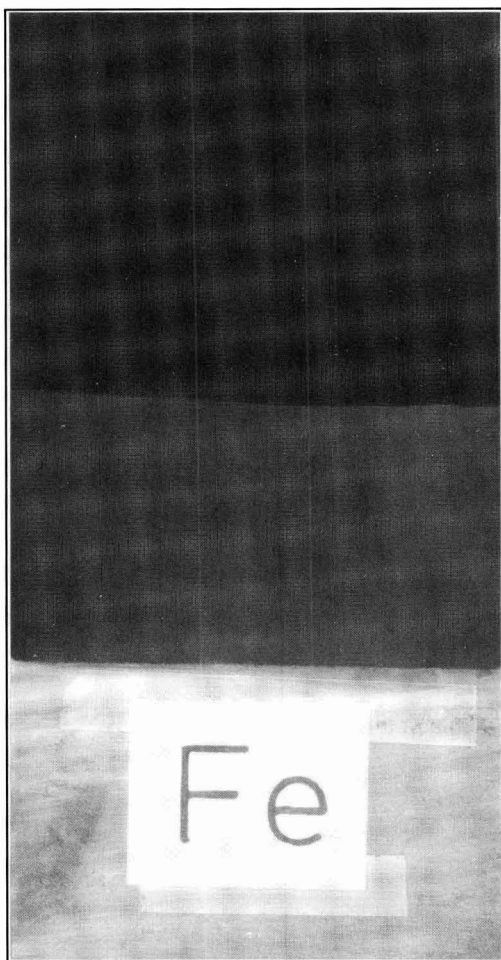


Figure 12—Iron panel (10 × 20 cm) with a polypyrrole layer ( $d_n = 1.5 \mu\text{m}$ ) (PPy-Ox) partially overcoated by a cathodically electrodeposited paint (83 - 0270, Cathodip) at 250 V, 2 min

tems.<sup>31,32</sup> The needle-like crystal texture in both cases is an important factor. In addition, our XPS results<sup>28</sup> revealed that no iron was found at the surface of a polypyrrole layer,  $d_n = 1 \mu\text{m}$ ; thus, the iron compound was not distributed in the polymer. On the other hand, polypyrrole was not found in samples with  $d_n = 10\text{-}100 \text{ nm}$ , as calculated from the initial peak in *Figure 2*; thus, PPY forms a stable layer only in the later phases of electrodeposition. It should also be noted that polypyrrole layers, which are electrodeposited from nonaqueous electrolytes at platinum, adhere poorly, but the presence of water<sup>1,33</sup> greatly improves the adherence. This may be attributed to the anodic formation of a platinum oxide interlayer. The electrolyte also influences adherence of polypyrrole at platinum.<sup>34</sup>

We also have shown that the Fe/PPy-Ox system leads to relatively smooth polypyrrole layers. This may be a consequence of just this iron oxalate interlayer. It provides a high density of nuclei for electrocrystallization of polypyrrole. Two models are known, namely, surface polymerization for polypyrrole,<sup>35</sup> and an oligomer precipitation for polythiophene.<sup>36</sup> A three-dimensional nucleation was identified for polyaniline.<sup>37</sup>

Our novel conclusions are expected to find further application in steel coating technologies for corrosion protection. Polypyrrole provides a very strongly adherent interlayer. For a thickness of  $1 \mu\text{m}$  and a typical current density of  $2 \text{ mA cm}^{-2}$ , a deposition time of 4.5 min is derived from equation (1), using  $\gamma = 0.70$  and  $y = 0.30$ . As the current efficiency can be improved to about 0.90, the current densities can be doubled and the induction period can be omitted,<sup>11,28</sup> the cycle time can be adjusted to that of electrodeposition of paint. Catalysis by bromide<sup>17-19</sup> to achieve higher current densities is not compatible with the iron substrate, but quinone as an electrocatalyst<sup>38</sup> may be an adequate solution. We were able to overcoat the Fe/PPy-Ox system by cathodic electrodeposition of paint, and *Figure 12* shows the result (after the baking procedure). It should be noted that the last step must be accompanied by cathodic undoping of the polypyrrole layer. However, this did not result in any loss of adherence to the steel or to the polypyrrole. Anodic overcoating by electrodeposition of paint is also possible. Similar results were obtained on aluminum,<sup>39</sup> where PPy-Ox was also found to be the optimum system.<sup>29</sup> In conclusion, this may provide a way to substitute the conventional phosphate interlayers used hitherto in industrial practice with polymer layers, making use of the CIPL principle mentioned in the Introduction section.

## SUMMARY

Only a few bath compositions are known to allow for the electropolymerization of pyrrole on iron to yield a polypyrrole coating. The exceptional performance of the aqueous oxalic acid electrolyte is demonstrated with respect to the following important technical properties:

(1) Adherence strength,  $\tau$ , is larger than  $11.5 \text{ N mm}^{-2}$  for  $1 \mu\text{m}$  films, and it decreases only slowly with increasing thickness. This is attributed to the formation of an iron oxalate interlayer.

**Table 6—Specific Electronic Conductivities of Doped Freestanding Polypyrrole Layers of  $10 \mu\text{m}$  Thickness at  $25^\circ\text{C}$**

Anions in PPy	$\kappa / \text{Scm}^{-1}$
$\text{ClO}_4^-$	120
$\text{BF}_4^-$	80
$\text{NO}_3^-$	3
$\text{HC}_2\text{O}_4^-$	0.01

(2) Surface roughness,  $R$ , measured with a profilometer, is found to be relatively low in comparison to other polypyrrole salts. It tends early to a steady state with increasing thickness of the layer, while normally it increases strongly.

(3) The polymer films are essentially nonporous. Porosity increases only slightly on elongation of the coating in parallel to the iron substrate by 1-2%.

(4) A pronounced leveling action for the electrodeposition was found, resembling the effects in galvanotechnology.

Overcoating by cathodic or anodic electrodeposition of paint was found to be feasible without severe losses in the previously mentioned qualities. This finding opens the possibility to apply these polypyrrole coatings as corrosion inhibiting polymer (inter)layers (CIPL). A substitution of phosphatation is expected, and a cycle time of about two minutes can be adjusted.

## ACKNOWLEDGMENTS

Financial support by Arbeitsgemeinschaft industrieller Forschungsvereinigungen (AIF) and by the German Israeli Foundation (GIF) is gratefully acknowledged. We are obliged to Professor Molt, of Universität Duisburg for the IRRAS measurements; to BASF Lacke und Farben AG, Hiltrup, for supply of the electro paints; and to Thyssen Stahl AG, Duisburg, for provision of the steel panels.

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## January 1992 Subcommittee Reports of ASTM Committee D-1

The January meeting of ASTM Committee D-1 was held on January 19-22, 1992 at the Crown Sterling, in Fort Lauderdale, FL. In the three-and-one-half days preceding the final session and general meeting of Committee D-1, 224 members and guests met in 166 scheduled meetings of D-1 and working task groups. The current membership of Committee D-1 is 580.

The officers elected to serve two-year terms starting January 1992 are: K.A. Trimmer—Chairman; K.H. Fujimoto—First Vice Chairman; J.J. Brezinski—Second Vice Chairman; J.C. Weaver—Third Vice Chairman; M.E. McKnight—Recording Secretary; and F. Gale—Membership Secretary and Treasurer.

During the Tuesday meeting of D-1, J.C. Weaver presented a special award to S.B. Levinson for his nearly 40 years of dedication and service to ASTM D-1. Following the award presentation, a Mini symposium on Hardness Testing was held in honor of G.G. Sward. P.R. Guevin chaired the symposium and papers were presented by J.C. Weaver, C.K. Schoff, and M.R. Euverard.

### Highlights

*D01.08*—Recent Federal actions have led to studies to measure the rate of VOC emission from paints.

*D01.15*—D-1 and G-3 will jointly sponsor a symposium on Accelerated and Outdoor Durability Testing in January 1993 in Fort Lauderdale.

*D01.21*—A new method for determining the water content for waterborne coatings, for VOC calculation purposes, will be developed in *D01.21.54*.

*D01.46*—A new task group was formed to develop standards for field sampling of paint, soil, and debris for laboratory analysis of heavy metals.

### Future Meetings

June 28-July 1, 1992—Marriott City Center, Minneapolis, MN.

January 17-20, 1993—Crown Sterling, Fort Lauderdale, FL.

June 27-30, 1993—Franklin Wyndam, Philadelphia, PA.

January 23-26, 1994—Crown Sterling, Fort Lauderdale, FL.

June 26-29, 1994—Marriott Crab Tree Valley, Raleigh, NC.

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### DIVISION 1 ADMINISTRATION

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#### SUBCOMMITTEE D01.08 ENVIRONMENTAL CONCERNS

**J.J. Brezinski, Chairman**

*D01.08.01*—*Durability and VOC*—M.M. Gaske, Chairman, reported that a survey conducted by the Steel Structures Painting Council provides data that relate the performance of professionally applied industrial maintenance coatings with relative VOC content. The survey results will be presented to EPA for consideration in the

development of a national rule on architectural and industrial maintenance coatings.

*D01.08*—New regulations to implement the revised Clean Air Act and the pending Indoor Air Quality Act will limit VOC emissions from paints and coating processes not presently controlled and also create a need for new and revised test standards.

Standards that address these needs under development or in the planning stage in D-1 include:

- Determination of volatile content of water- and solvent-borne aerosol spray paints.
- Volatile content of radiation-cured materials.
- Formaldehyde content of waterborne architectural paints containing a preserva-

tive and of industrial paints with melamine resins.

- Revision of the standard on solvents in paint, the study to include solvents designated as hazardous air pollutants under the Clean Air Act.

- Ammonia content in waterborne systems (task group participants are needed). Exemption of ammonia from the VOC content calculation will likely be available after a consensus standard is developed.

- The revision of ASTM Manual #4, Determination of VOC Content of Paints, Inks and Related Coating Materials, will update comment and include VOC-related standards developed or revised since 1988. Target for publication is late 1992 or early 1993.

- Studies related to indoor air quality in progress in D22.05 use a small scale environmental chamber (D 5116) to measure the rate of VOC emission from adhesives, caulks, paints, etc.

Pending Federal legislation on abatement of hazards from lead in buildings suggests ASTM focus on standards applicable to in-place management; additionally, standard sampling procedures are needed relating to the removal of lead paint from industrial structures.

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

**G.Y. Moore, III, Chairman**

ASTM Committees D-1 and G-3 will jointly sponsor a symposium on "Accelerated and Outdoor Durability Testing" in January 1993, in Fort Lauderdale. Co-moderators for the symposium are W.D. Ketola and D.M. Grossman. A call for papers was sent out in October 1991, and ten proposals have already been received. At least two more and maybe several more are expected. The symposium will begin on Tuesday afternoon, January 19, and will continue that evening and all of the next day.

D-1 will sponsor a mini-symposium on "Slip Resistance" in January 1994, in Fort Lauderdale. T.J. Sliva will be moderator and P.R. Guevin will be one of the speakers.

G.Y. Moore has resigned as Chairman of D01.15, and T.J. Sliva has agreed to assume this responsibility.

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## **DIVISION 20 RESEARCH AND GENERAL MEETING**

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

**G.Y. Moore, III, Chairman**

R.K. Morrison made a presentation relating SCM Chemicals' experience in obtaining ISO 9002 registration. Additional comments were made by R.M. Hopkins. SCM achieved ISO 9002 registration from a British company. Two major words of advice for those seeking such registration are "Documentation" and "Consistency." Mr. Morrison suggested that a company seeking ISO 9002 registration should ask

itself the following three questions and carefully document the answers:

- (1) What does your company do?
- (2) How does your company do it?
- (3) How does your company know?

Mr. Hopkins added that from a laboratory point of view, technician training and qualification records were critical.

G.Y. Moore discussed the Aqualon Company's experience as it has begun seeking ISO 9002 registration from Underwriters Laboratories. ISO 9002 is a basic set of quality elements with which a plant's quality manual must deal. It is not Total Quality Management or Good Manufacturing Practices. Aqualon management has mandated achievement of ISO registration by all of its facilities, using Underwriters Laboratories in the U.S. and Lloyd's Register in Europe. The Hopewell, Virginia Plant will have its first audit inspection in the summer of 1992.

G.Y. Moore has resigned as Chairman of D01.20, and R.K. Morrison has agreed to assume this responsibility.

### **SUBCOMMITTEE D01.21 CHEMICAL ANALYSIS OF PAINTS AND PAINT MATERIALS**

**K.H. Fujimoto, Chairman**

*D01.21.10—Lead-in-Paint Policy*—J.C. Weaver, Chairman. Fast changes in policies of detection, abatement, and disposal of lead in dwellings and public structures make difficult any reporting here of progress in quantitative analysis for lead in diverse scenarios.

E06.23 on lead abatement in structures has meetings of its five sets of task groups on detection, abatement, removal, accreditation, and terminology on October 14-16, 1991, January 22-23, 1992 and April 8-9, 1992 to develop its dozen task subgroups. Coordination of these with D01.08 and D01.21 is difficult unless the paint/pigment community volunteers much time.

D01.57 on Artists' Materials and its task group D01.57.12, under the leadership of toxicologist Dr. Woodhall Stopford, of Duke Medical Center, continues to adapt the European concept of limited biologic availability of lead and other toxic metals in artists' materials and their exemption from regulation.

D-22 on Atmospheres and its Sub. D22.05 on Indoor Air has its D 3112 method for lead in atmospheres.

A base line for lead in street and yard soil and dusts higher than the 600 ppm of lead-in-paint in the Lead Based Paint Poison Prevention Act is in prospect from EPA from its sponsorship of the Three City (Boston, Baltimore, and Cincinnati) Study to be completed in 1992.

Lead Free Kids, Inc., 100 E. 31st St., Box 8595, Minneapolis, MN 55408, (612) 377-4304 is selling three booklets and accessories: (1) family guide, (2) workers guide, and (3) owner/contractors, on the theme "Lead Poisoning is the Number One Most Preventable Disease Effecting Children in the United States Today", i.e., keep small children scrupulously clean in their lead soiled homes.

*D01.21.13—Update on Regulatory Action*—J.J. Brezinski, Chairman, reviewed, correspondence received from Jim Berry, of EPA, at the task group meeting. The document covered the planned exploratory meeting at Research Triangle Park dealing with the "reg-neg" process (negotiated rulemaking) being considered for the development of a future national rule on VOC emission control for architectural and industrial maintenance coatings.

Gene Praschan, of the Motor Vehicle Manufacturers Association, summarized the discussions of the November 1991 meeting of the National Air Pollution Control Technical Advisory Committee (NAPCTAC) on the ongoing development of Control Technique Guidelines on VOC Control for automobile refinishing, painting of plastics (for automotive), and wood furniture finishing. Draft copies of the CTG documents have since been distributed by EPA.

The ongoing standard activity in ASTM Sub. D22.05 on Indoor Air includes the use of a small environmental chamber to characterize VOC emissions from architectural paints as well as from adhesives, caulks, etc. Close liaison between D-1 and D22.05 should be established.

The draft of the revision of EPA's Federal Reference Method 24 was briefly reviewed. Changes are compatible to those recently adapted in ASTM Standards D 2369 and D 3960. EPA will be asked to recommend D 3960 as a guide for the calculation of VOC content of paints and coatings.

*D01.21.14—VOC Publication and Workshops*—J.J. Brezinski, Chairman, stated that the revision of ASTM Manual #4 on Determination of VOC in Paints, Inks and Related Coatings has been initiated. The new version of Federal Reference Method 24, ASTM standards D 2369 and D 3960 will be incorporated, as well as other VOC-related standards finalized since 1989. Comments about VOC-standards development in progress will also be included. Assistance from members of various subcommittees of D-1 will be sought, including those from Sub. 21, 24, 47, 55, and 56. Publication target date is the end of 1992 or early 1993.

H. Fujimoto discussed the VOC workshops. The workshop scheduled for November in Toronto, in conjunction with the Annual Meeting and Paint Industries' Show, was cancelled due to low registration. A workshop is scheduled for June 10-11, 1992

in Detroit, MI. The host laboratory will be The Du Pont Company, in Troy, MI.

The fall workshop is scheduled for Philadelphia on November 5-6, 1992. The host laboratory will be The Du Pont Company, Marshall Laboratory.

The purpose of the workshops is to instruct in the proper techniques of running test methods needed to determine VOC of Paints and Coatings using ASTM Standards referenced in EPA Method 24, and to understand the calculations used to obtain VOC content. In addition, participants receive the latest standards and pertinent information available on determination of VOC.

*D01.21.24—Revision of D 2369*—M.E. Sites, Chairman, reviewed the recent ballot results on the revision of D 2369. This revision was done for three purposes;

(1) To incorporate additional directions and instructions in all sections, based on experience gained in VOC workshops while instructing persons on use of D 2369.

(2) To broaden the scope of the method to include multicomponent paint systems based on results of round-robin testing on multicomponent systems by D01.21.24.

(3) To remove Note 2 and the appendix which refer to the original D 2369 method which was a 20' at 110°C bake. The U.S. EPA references D 2369 with the 60' at 110°C bake in its documents and we need to remove all references to 20' at 110°C to eliminate possible confusion.

One negative was received and three persons had comments. The negative was withdrawn and the comments were incorporated where appropriate.

Although this revision will not be in the 1992 book, separates will be available after this passes society ballot, and will be available to be placed in the revised Manual on Determination of VOC.

*D01.21.24A—Ion Chromatography, Electrocoat Samples*—M.J. Mahon, Chairman, reported the initiation of cooperative work with Dionex Corporation, in California, to develop methods for measuring the concentration of anions in E-coat baths by IC. Past methods have been plagued with technical difficulties and Dionex assistance is welcome.

Chairman Mahon reported excellent agreement between two laboratories analyzing NIST Standard Reference Rainwater Samples using Ion Chromatography. Further work on measuring anion concentrations in E-coat ultrafilters will commence.

*D01.21.25A—VOC Release from Applied Coatings*—J.S. Komathy, Chairman, reviewed the three negative votes received on the balloted method. A negative received for the lack of a footnote on the title page was withdrawn by telephone with the correction to be made. A negative received for two undefined symbols was withdrawn by

telephone with the correction to be made. A negative received because of possible inherent errors has not been resolved. It was decided to withdraw the method, to make some changes, and to reballot. Before it is reballoted, the updated version will be sent to EPA personnel for comment. It was also suggested at this meeting that an improved technique be used to remove the wet coating from the panel by covering the panel with aluminum foil and removing the foil for coating analysis. This change will be incorporated. A new round-robin has been organized, which will commence in a few weeks after receipt of the revised method.

*D01.21.27A—VOC Content of Aerosols*—R. Osterman, Chairman, reported that methods for the VOC content of solventborne and waterborne aerosols were submitted for concurrent D-1/D01.21 ballot since the last meeting held in Ottawa, Canada in June 1991. Two negative votes on item #1 of Main Committee Ballot D-1 (91-03) were received. The negatives were withdrawn by phone due to editorial changes and the following: (1) addition of keywords; and (2) that a research report will be sent to ASTM headquarters for filing.

Two negative votes on item #2 of Main Committee Ballot D-1 (91-03) were received. Item #2 was removed from the ballot by action of the subcommittee which agreed to the appropriate changes to satisfy the negatives. The standard will be submitted for committee/subcommittee balloting concurrently.

*D01.21.27B—New Approach to VOC*—R.K.M. Jayanty, Chairman, was absent and H.K. Fujimoto acted as temporary chairman. A quick review of the technique used to determine VOC was made. The method consists of placing an aluminum pan, which contains the test paint specimen plus a glycol, such as trimethylene glycol (to keep the paint film open), into a special air tight glass chamber into which dried nitrogen gas flows. The whole glass chamber is placed into a 110°C forced draft over and baked for one hour per D 2369.

The vapors emitted are collected on two large tubes filled with activated charcoal and which are in tandem. Water absorbing tubes have been attached to the second charcoal tube to collect the moisture or water emitted. Water can be calculated by difference. Preliminary results on controlled panel samples have shown good VOC precision.

Although a successful round-robin has been run, some negative comments have been made by some of the collaborators.

(1) Activated charcoal does not readily absorb polar compounds such as methanol and other low molecular weight alcohols and amines.

(2) The glycol used absorbs moisture which could affect the results.

(3) There are too many connections, and it is difficult to check for leaks during a test run.

(4) Alternative absorbents could be used such as Carbpak or Carboseive (Supelco) which would do away with packing the tubes with charcoal.

(5) The apparatus has to be revised and simplified.

(6) Carbon tubes are too large and bulky for the amount of VOC collected (i.e., weight differential). R.K.M. Jayanty will be contacted to ascertain the status of this project.

*D01.21.41—Field Methods for Detection of Lead in Paint Films*—M.E. McKnight, Chairman, proposed that a standard for evaluating spot tests for lead in paint be developed. Although limitations in the use of laboratory prepared paint film were acknowledged, it was felt that a useful screening procedure could be developed. After some "hands-on" use of two spot tests with laboratory prepared lead-containing paint films, the group agreed to proceed with development of a standard. The Chairman agreed to draft a procedure and conduct a preliminary round-robin.

*D01.21.46—X-Ray Analysis of Pigments*—M.J. Mahon, Chairman, reported the revised method for "Determination of Phosphate in Electrocoat Baths by XRF" has been distributed and a round-robin study initiated. Chairman Mahon also reported that he and A.M. Snider, of PPG Industries, plan to issue a "Standard Guide for Qualitative Identification of Pigments by XRF" within three months. All Task Group 46 members will receive copies for comment.

*D01.21.51—Determination of Formaldehyde in Paints*—K. Plecity, Chairman, has resigned due to a change in job responsibilities; H.K. Fujimoto acted as Chairman.

J. Benga reviewed the various formaldehyde methods he had evaluated unsuccessfully for the task group; Bisulfite reduction; HPLC—derivatization/UV detection; head space/GC with methanization; and GC with mass selective detection. After a lengthy discussion on the complexity of analyzing for formaldehyde in paints, it was decided two approaches should be used. The first is to develop a test method to analyze for the formaldehyde placed in water reducible paints as a preservative. The second approach will be to determine the free formaldehyde in paint systems which can generate the gas such as acrylic/melamine-formaldehyde resin systems.

Darwin McCunn, of Glidden, has volunteered to chair this task group. He has asked for copies of methods, in use, to determine free formaldehyde. As soon as he is satisfied he has a viable technique, he plans to run a mini-round-robin to evaluate the method. Detection will be either GC or GC/MS.

*D01.21.52—Replacement of D 2349—*J.G. Lambertson, Chairman, announced the task group was renamed "Paint Solvent Analysis by GC" in order to better indicate its activities and improve participation. D 2349 is to be placed on a letter ballot for discontinuance or removal. The new method proposed is to utilize the following approaches:

(1) Utilize capillary columns possibly of the wide bore design.

(2) Utilize AFFAP, Carbowax, and/or DB-5 type liquid phase.

(3) Utilize an FID as the detector on possibly the MSD/ion trap.

(4) Quantify and identify those chemicals identified as hazardous by the Clean Air Act.

A two-part method including an initial screening procedure to determine whether a direct injection or extraction type of analysis should be performed was suggested for a first draft of the method. Both the direct injection and extraction procedures are to be written and distributed for an initial round-robin evaluation. Comments concerning this evaluation are to be brought to the next meeting for consideration at that time.

*D01.21.56—Revision of D 3960—Calculation of VOC—*M.E. Sites, Chairman, announced the latest revision of D 3960, balloted on the D01.91.02 ballot and on the September 1991 society ballot will be included in volume 6.01 of the 1992 *ASTM Book of Standards*. This standard provides a guide to appropriate ASTM test methods and calculations used for determining VOC according to U.S. EPA Reference Method 24.

This revision was undertaken when concerns were expressed that the calculation section was confusing and should be clarified and be compatible with symbols and equations used in EPA Reference Method 24. This would make D 3960 easier to use and we hope this guide would be referenced by the U.S. EPA as an appropriate document. J. Brezinski will write a formal letter to the U.S. EPA once this document is published. In addition, this revision now includes reference to multicomponent systems which will be included in the new revision of EPA Method 24.

Task Group D01.47 on Masonry Treatments met with D01.21.56 in June 1991 and requested that D 5095-90, Standard Test Method for Determination of Non-Volatile Content in Silanes, Siloxanes, and Silane/Siloxane Blends used in Masonry Water Repellent Treatments, be referenced in D 3960. This provides a method for correctly determining the nonvolatile content of penetrating sealers and their corresponding VOC levels. Since this method for nonvolatiles of silanes is not approved by U.S. EPA at this time, it was agreed to reference this in D 2832, "Standard Guide for Determining

Volatile and Nonvolatile Content of Paint and Related Coatings." This revision was balloted on D01.91.04 and passed with no negatives or comments. If D 5095 is approved by U.S. EPA in the future, it will be referenced in D 3960.

If it was agreed to keep this task group on an active status, since VOC issues are in constant change and review.

*D01.21.80—Exploratory Analytical—*M.J. Mahon, Chairman, reported continuing work on a new method of wet digestion for the determination of trace metal concentrations in coatings. The collaborating laboratories of PPG, ICI Glidden, and Du Pont will evaluate this new procedure in a newly created Task Group D01.21.49. Bruce Neff, of The Du Pont Company, will chair this new task group.

M. Mahon also introduced a newly developed method for determining the water content, for VOC calculation purposes, of waterborne coatings. Several laboratories will participate in studies to determine this procedure's applicability to the wide-variety of waterborne coatings. Task Group D01.21.54 will continue this work.

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

**J.J. Brezinski, Chairman**

ISO 9038, "Determination of the Ability of Liquid Paints to Sustain Combustion," is being considered for round-robin testing. Data developed using this standard will, it is believed, demonstrate that many latex-based paints though they yield a defined flash point are incapable of sustaining combustion. Such data could be instrumental in convincing OSHA and DOT to change the flammability classification (for shipment) of latex-based paint.

A new standard, "Sampling for Flash Point and Related Tests," being developed by the Coordinating Committee on Flash Point will, when finalized, be considered for adoption in D-1. M.K. Harding has accepted the appointment as Chairman of Task Group 22.01 on Flammability and Flash Point.

Jurisdiction for E 848, "Standard Practice for Safety and Health Requirements Relating to Occupational Exposure to Water-Insoluble Chromates," has been accepted by D-1. E 848, first issued by E-34 in 1982, was last revised in 1985. Task group members are needed to revise/update this health and safety standard.

The chairman will continue to encourage D-2 on Petroleum Products and Lubricants to delete partial chemical-specific precautionary hazard statements in flash point standards.

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

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

*D01.23.10—Adhesion—*G. Nelson, Chairman, reported that the revision of D 3395-90, "Test Methods for Measuring Adhesion by Tape Test," had received a negative vote on the society ballot. The task group found the negative nonpersuasive and not relevant to the method under ballot.

Representatives from 3M will be invited to the next meeting to further discuss their concerns about the tapes currently specified in the method. It was further recommended that an appendix be developed for D 3359 citing various issues relating to the tapes currently specified in the method and that the task group evaluate the relative adherability of tapes to various coating surfaces.

The chairman reported that the new standard, D 5179-91, "Test Method for Measuring Adhesion of Organic Coatings to Plastic Substrates by Direct Tensile Testing," and the reapproval of D 2197-86, "Test Method for Adhesion of Organic Coatings by Scrape Adhesion," had successfully completed society ballot. D 3002-81, "Practice for Evaluation of Coatings for Plastics," is currently under revision.

*D01.23.12—Dry Film Thickness—*K.A. Trimmer, Chairman, reported that two negatives were received on the D01.23 ballot to revise D 1186-87, "Test Methods for Non-Destructive Measurement of Dry-Film Thickness of Non-Magnetic Coatings Applied to Ferrous Base," and D 1400-87, "Test Methods for Non-Destructive Measurement of Dry Film Thickness of Non-Conductive Coatings Applied to a Non-Ferrous Base." One negative was found nonpersuasive based upon its lack of specificity. The second negative submitted by M. Morse stated that the bias statements in both methods were unsatisfactory. The voter will be contacted to see if he will withdraw his negatives after suggested revisions are included.

P. Guevin reported on the use of ultrasonic instrumentation to measure the dry film thickness of organic coatings and will determine its applicability to measure organic coating thickness over concrete.

*D01.23.14—Hardness, Abrasion and Mar Resistance—*D.J. Wilverding, Chairman, reported on progress on the proposed "Test Method for Mar Resistance of High Gloss Coatings." The method uses a Taber Abraser and measures gloss retention. B. Gregorovich, of Du Pont Company, explained the origin of the test and how it shows advantages over other typically used methods which would measure haze development and DOI (Distinctness of Image). Further discussion centered on specifying

instrumentation, the positioning of the meter and panels, and the use of the Taber refacer versus the discs when preparing the wheels before and during test. Editorial comments on the method by T. Sliva were reviewed and will be incorporated into a revision of the method. It was the decision of the task group to initiate a round-robin of the method.

The chairman reported that the revisions to D 1474-85, "Test Methods for Indentation Hardness of Organic Coatings," D 3363-74, "Test Method for Film Hardness by Pencil Test," and D 4366-87, "Test Methods for Hardness of Organic Coatings by Pendulum Dampening Tests," have completed D-1 letter ballot.

P. Guevin will submit a proposed revision of D 2134, "Test Method for Determining the Hardness of Organic Coatings with a Hardness Rocker," for review by the task group at the next meeting.

*D01.23.15—Slip Resistance*—P.R. Guevin, Chairman, read a report from M. Morse on the status of round-robin activity to develop a precision statement for D 4518-91, "Test Methods for Measuring Static Friction of Coating Surfaces," under wet conditions.

The chairman reported on the workshop on Slip Testers held at Bucknell University in July 1991. He showed a chart which compared the various slip testers on a medium friction tile under dry conditions and the typical curves that are generated using the force plate. M. Euverard discussed the importance of dwell time and its effect on the Coefficient of Friction (COF) measurement and the need for measuring "breakaway" force, as this is when slip occurs. He also discussed the effects of scratches on the test surface on its COF reading.

The chairman reported that Underwriters Laboratory has issued UL-410, "Proposed First Edition of the Standard for Slip Resistance of Floor Surface Materials," for comment.

*D01.23.16—Water Vapor Transmission*—T.J. Sliva, Chairman, reported that the revision to D 1653-85, "Test Method for Water Vapor Transmission of Organic Coating Films," had completed society balloting. The chairman discussed additional proposed revisions to the method. These revisions will include introducing the terms "wet" and "dry" perms to the method and include a statement in the Appendix outlining differences expected between methods and the applicability and limitations of both methods under typical use.

*D01.23.18—Flexibility*—P.R. Guevin, Chairman, reported that M. Morse has revised the precision statement for D 522-88, "Test Methods for Mandrel Bend Test of Attached Organic Coatings," and the Significance and Use Section of D 2794, "Test Method for Resistance of Organic Coatings

to the Effects of Rapid Deformation (Impact)." The chairman distributed these revisions and additional editorial changes were discussed.

M. Burgess, of AMTL, reported on comments from H. Moore that indicated revisions to D 522 do not define panel thickness and magnification power to be used when examining for cracks and she would like to see the method as outlined in Federal Test Method Standard 141 incorporated into D 522. J. Peters and P. Guevin will contact the voter and determine what changes can be made without detracting from the intent of D 522.

*D01.23.19—Drying Time*—T.J. Sliva, Chairman, discussed the scope and purpose of this new task group. D01.23 has assumed stewardship and responsibility for D 1640, "Standard Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature," since the last meeting. The method will be reviewed for editorial and technical revisions and the task group will begin looking to incorporate various mechanical methods of determining drying time into the method.

At the present time, the task group will look to evaluate both straight line and circular drying time recorders and evaluate their applicability to D 1640. The chairman reported that data using the circular drying time was available and would be reviewed to see if it could be used to develop a precision and bias statement. The chairman intends to begin round-robin testing using straight line drying time recorders at that time.

*D01.23.10—Exploratory Research*—P.R. Guevin, Jr., Chairman. This new task group was set up to make members aware of new instrumentations and technologies that are being developed to determine the physical properties of applied films. Task group members will present and report relevant literature and articles. Manufacturers will be invited to give demonstrations of new technology. Liaison with other ASTM and professional committees will be established. At this initial task group meeting, the chairman listed possible projects involving the use of ultrasonics and X-ray fluorescence spectrometry to measure paint thickness and hardness.

## **SUBCOMMITTEE D01.24 PHYSICAL PROPERTIES OF LIQUID PAINTS**

**C.K. Schoff, Chairman**

*D01.24.18—Dispersion Phenomena*—R.K. Morrison, Chairman, discussed D 1210, "Fineness of Dispersion of Pigment-Vehicle Systems," which is due for revision or reapproval. There also was considerable

discussion regarding a new cleanliness or texture gauge that appears to give superior precision for cleanliness readings compared to a Hegman gauge. The new gauge was developed by SCM and is a step gauge with one mil and three mil steps giving one square inch reading areas. The chairman agreed to revise D 1210 to include a uniform light source and allow a one-inch width gauge to be used. He also will write a draft for a procedure B for cleanliness using the new step gauge.

*D01.24.19—Efflux Cups*—C.K. Schoff, Chairman, discussed D 1200, "Viscosity by Ford Cup," which is due for reapproval or revision. R.E. Manning, of Cannon Instrument Company, had offered to make a major revision of D 1200. He suggested including ISO and DIN cups as well as Ford cups, changing the form of the calibration equations, and generally revising the method to make it similar to ISO 2431. The task group voted to take him up on his offer, but would like him to add the Fisher cup as well, since it is used, along with the Ford cup, in the automotive industry. D 4212, "Viscosity by Dip-Type Viscosity Cups," was discussed very briefly and will be reviewed before and during the June D-1 meeting for possible revision.

*D01.24.26—Electrical Properties of Liquid Paints*—The proposed Method for Electrical Resistivity of Liquid Paint and Related Materials was reviewed, and further revisions due to negatives and comments on the latest D-1 ballot are needed. Questions had been raised regarding test conditions, particularly humidity and temperature. Humidity was not considered a problem since air was not the dielectric in the test. There were no known humidity problems, although specimens may pick up moisture and change conductivity. It was felt that this was a storage concern rather than a measurement problem. The question of the test temperature was not so easy to answer and will be looked into more carefully. The method now specifies measurement at 25°C, but gives no range or limits. Editorial and other changes will be made and the method will be rebalotted, if necessary.

*D01.24.33—Odor Evaluation*—D.E. Darr, Chairman, discussed a possible guide or practice on odor evaluation. This task group has had difficulty deciding on exactly what it should do and how it should go about doing it. There is considerable interest in odor and odor evaluation in our industry, but the attack on the problem is highly fragmented with many different organizations, ASTM Committees, etc., being involved. Before we put a lot of effort into writing a guide or practice, we need to find out what already is available and who is actively working on odor evaluation. The



chairman will circulate such information before the June meeting, so that we can start writing at the meeting.

*D01.24.34—Viscosity by Falling Needle Viscometer*—J. Hartnett, Chairman, reviewed the latest draft of the proposed Standard Test Method for Viscosity by Falling Needle Viscometer. The method has received two negatives and a number of comments on the most recent D-1 ballot. It was decided to withdraw the method from ballot and do a considerable amount of re-writing in order to simplify and clarify the method and make it more readable and easier to use. The revised method will be submitted for concurrent D-1/D01.24 balloting.

*D01.24*—The possibility of developing a guide or practice for determining the cleanliness of paint including detecting foreign materials, particularly dirt, was discussed. It was thought that such a guide or practice would be useful for automobile paints, furniture coatings, architectural gloss enamels, and other high gloss coatings. A.F. Rutkiewicz volunteered to write an outline for a procedure and to present it at the June D-1 meeting.

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

### **R. Kumar, Chairman**

*D01.26.02—Color Measurement*—R.T. Marcus, Chairman. Because the chairman was unable to attend this meeting, C.J. Sherman presided. The subcommittee ballot of the "Standard Guide for the Preparation, Maintenance and Distribution of Physical Product Standards for Color of Paints" received negatives from Messrs. Peters, Hammond, and Hale. Time did not allow to review all of the changes and suggestions of the negatives and comments from members at the meeting. Therefore, Mr. Sherman will make the remaining changes to the guide and submit it for D01.26 ballot.

D 1544-80, "Standard Test Method for Color of Transparent Liquids (Gardner Color Scale)," received a negative from Mr. Peters and comments from Mr. Hammond. The method will be revised accordingly by Mr. Billmeyer and submitted for D-1 ballot.

*D01.26.06—Hiding Power*—L.E. Schaeffer, Chairman, reviewed the efforts of the group to develop a hiding power method for powder coatings in cooperation with D01.51 on Powder Coatings. A round-robin has been run in which hiding power, expressed as the spreading rate in square meters per kilogram, was determined by the same weight area method as in D 2805. The results indicated very satisfactory correla-

tion among four cooperators. The Powder Coating cooperators prefer film thickness measurements instead of the weight area procedure and to obtain the film thickness at 0.98 contrast ratio rather than spreading rate. A round-robin will be run for that purpose and the results should be available at the next meeting.

*D01.26.11—Gloss and Goniophotometry*—A.F. Rutkiewicz, Chairman, reported the revision of D 4039-87, "Standard Test Method for the Reflection Haze of High Gloss Surfaces," was discussed and with the addition of key words and reference to E 430 as an alternate method for measuring haze will be submitted for concurrent D-1/D01.26 ballot.

The last draft of a new, "Test Method for the Instrumental Measurement of Distinctness of Image Gloss of Coating Surfaces," was discussed. A number of minor changes were made in addition to a revision of the primary and working standards refractive index to 1.527 in Method B (Suga HA-NSIC). The corresponding Fresnel values of  $G_s = 89.2$  for  $20^\circ$  and  $G_s = 93.6$  for  $60^\circ$  were added. The method will be submitted for concurrent D-1/D01.26 ballot. A. Rutkiewicz will gather what round-robin data is available and request M. Morse to analyze the data for precision and bias.

The question of developing a new standard method for the use of various visual-mechanical devices to measure distinctness of image was not generally supported, mostly because of lack of knowledge. However, if a draft were developed, it would be considered.

The task group took note of the many years of fine work in the field of gloss evaluation contributed by M.P. Morse and appreciate his continued interest and support.

*D01.26.24—Tinting Strength*—R. Morrison, Chairman, reported the responses of negatives and comments on D 387, "Test Method for Color and Strength of Color Pigments with a Mechanical Muller," have been completed and will be submitted for concurrent D-1/D01.26 ballot.

Suggested changes to the proposed method, "Test for Evaluating the Tint Undertone of Titanium Dioxide Pigments," have been made and the method will be submitted for D01.26 ballot. A round-robin on this proposed method will be initiated shortly.

Test method D 332, "Test Method for Tinting Strength of White Pigments by Visual Observation," is ready to go to printing.

D 2745, "Test Method for Relative Tinting Strength of White Pigments by Reflectance Measurements," needs to have the tinting strength equation in section 8.1 corrected. This will be done and submitted for concurrent D-1/D01.26 ballot.

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

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

*D01.27.02—Water Tests*—D. Grossman, Chairman. All four water tests passed D-1 ballot. They are: D 870, Immersion; D 1735, Water Fog; D 2247, 100% Humidity; and D 4585, Controlled Condensation. They will go to society ballot with editorial changes. However, a separate D01.27 ballot will be made on a new Method B for D 2247, 100% Humidity. The practice now specifies that cabinets shall generate humidity by heating a bath of water in the chamber bottom. However, most of the cabinets being used for this type of humidity test generate humidity by bubbling compressed air through an un-heated water bath, while maintaining chamber temperature by a heated water jacket around the walls. This design often fails to produce adequate condensation on the specimens, unless the water jacket is drained to below the level of the specimens. F. Lutz will draft a revision with a Method B specifying the proper operation of this design.

*D01.27.04—Light and Water Exposure Apparatus*—L.E. Thieben, Chairman. The recent balloting of D 3361, "Unfiltered Open-Flame Carbon-Arc Dew Cycle," was discussed. A negative from S. Suga was withdrawn on condition of making an editorial addition to footnote #6, which lists manufacturers of equipment for this test. Other comments were discussed, but no other changes will be required. This revision will be sent to D-1 ballot.

Draft #6 of a new standard practice on Xenon-Arc test, was circulated and any comments on this draft should be sent to the task force chairman by March 1, 1992. If no significant comments are received by this deadline, then this draft will be submitted to D01.27 ballot. The development of this standard was tabled at the last January meeting because of a lack of interest and the time required to review other standards. Since the docket of this committee is clearing, it was decided to continue the development of this standard.

*D01.27.09—Evaluation of Corroded Specimens*—G. Rommal, Chairman, reviewed the results of the D01.27 ballot for revision of D 1654, "Evaluating Corroded Specimens." The ballot drew two negatives and one comment. A negative from D. MacGregor requested clarification of the definition of a "style E" lathe cutting tool. J. Robbins, III, voted negatively, requesting the addition of three more exposure testing standards to the list of referenced documents. Editorial changes were made to the proposed draft to address these concerns and the negatives were withdrawn. Then a new draft will be sent for the next D-1 ballot.

*D01.27.10—Accelerated Outdoor Weathering*—J. Robbins, III, Chairman. The negative comments were reviewed of the D01.27 ballot of D 4141, "Standard Practice for Conducting Accelerated Outdoor Exposure Tests of Coatings." After revising the standard in the meeting, a decision was made to reballoon this standard at the subcommittee level.

*D01.27.12—Detergent*—F. Lutze, Chairman. D 2248 on detergent resistance was reviewed as a result of difficulty in finding sources of sodium salt of a linear alkylaryl sulfonate. It was suggested that a note directing the user to a reference manual of surfactants be added to the standard. An effort will be made to determine if this standard reflects the actual tests in use today. Appropriate changes will be made to the standard and it will be sent to D01.27 ballot.

*D01.27.17—Evaluation of Weathering Effects*—J. Martin, Chairman. The results of D-1 ballots on D 772, "Evaluation of Flaking," D 1006, "Conducting Outdoor Weathering Tests on Wood Coatings," and D 1150, "Multi-Panel Forms for Exposure Records," were discussed. D 1150 was balloted for withdrawal and passed without negatives. D 772 received no negatives and two comments. An editorial change will be made in section 8.2 changing global radiation to total solar radiation per R. Kinmonth's comment. D 772 will be sent to society ballot. D 1006 received no negatives and seven comments. A comment by J. Hinkle was discussed concerning sections 9.2 and 9.4 in the panel size to allow for three test sections on each 36-inch board. Section 9.2 and 9.4 will be rewritten and reballoon by D01.27. The current D 1006 draft will be sent to society ballot with only editorial changes.

Revisions of D 660, "Evaluation of Checking," and D 661, "Evaluation of Cracking," and D 662, "Evaluation of Erosion," were not available for discussion, but will be completed by D. Dietz and J. Martin and sent to D01.27 ballot as soon as possible.

An "instant round-robin" was conducted at the meeting to establish precision and bias statements for D 714, "Evaluation of Blistering," and D 772, "Flaking." Also, R. Morrison conducted a round-robin test for D 4214, "Evaluation of Chalking," to identify a replacement tape (3M #480) to Sellotape 1401, which the TNO method requires but is not easily available in the U.S. Approximately 25 people participated in these round-robin tests. The task group thanks all of those who participated. The results will be discussed at the next meeting.

*D01.27.28—Filiform Corrosion*—F. Lutze, Chairman. D 2803, "Filiform Corrosion Resistance of Organic Coatings on Metal," was reviewed. Proposed changes include adding another procedure and changing the standard to a guide. Changes

will be made to the draft and it will be sent to D01.27 ballot.

*D01.27.29—Test Substrates*—D. Grossman, Chairman. D 2201, "Zinc Coated Steel Panels for Paint Tests," passed D-1 ballot and will go on to society ballot. A negative by P. Guevin was withdrawn with editorial changes. However, F. Lutz noticed that Appendix X1, Characterization of Zinc Coating, is missing many of the newer metric designations used in the auto industry. He will draft another revision for concurrent D-1/D01.27 ballot.

*D01.27.30—Corrosion Tests—Automotive*—F. Lutze, Chairman. Results of the accelerated outdoor cosmetic corrosion test were presented. Although acceleration rates varied widely, rank ordering was consistent among the exposure sites. A general discussion took place concerning the papers presented at the Automotive Corrosion & Prevention Conference (SAE) held in October 1991. A draft of a new standard for conducting accelerated outdoor cosmetic corrosion tests will be developed, involving outdoor exposure with intermittent spray with salt solution.

*D01.27.31—Corrosion Tests—Non-Automotive*—S. Boocock, Chairman. The round-robin on cyclic corrosion tests is progressing. The outdoor sites have been narrowed to SFTS Netherlands (industrial), Kure Beach 25m from ocean, Kure Beach 250m, and SSPC Neville Island (mild industrial). There is a possibility of also including one of the following sites: Rohm & Haas, Texas (Gulf/industrial); Sherwin-Williams, Cleveland (mild industrial); and SSPC, Pittsburgh Coke Plant (severe industrial). The final selection will be made prior to the June meeting. The coil coating specimens have been cut to size, and will be scribed and impact dimpled by D. Grossman before the next meeting. The industrial maintenance paint specimens present a much larger investment to prepare, and S. Boocock is pursuing funding through SSPC and other avenues. It is hoped that the specimens can be prepared to start exposure this summer. S.Boocock will submit requirements for evaluation of the industrial specimens and will prepare standard reporting forms. An estimate of the spread in the data was given derived from prior exposure studies by SSPC. Based on this, the task group has decided to expose a compromise number of five replicate specimens of each coating for each outdoor site.

## **SUBCOMMITTEE D01.28 BIODETERIORATION**

**M.C. McLaurin, Chairman**

*D01.28.01—Package Stability*—M.C. McLaurin, Chairman. The proposed method

was again reviewed by the group, and it was agreed that the following amendments would be incorporated: specifying the incubation temperature as 32°C (rather than 30°C); and requiring a count on the number of organisms in the inoculum. These amendments, along with those proposed at the June 1991 meeting, will be made to the method. The revised version will be circulated to members for comment prior to our June meeting.

*D01.28.02—Rapid Determination of Enzymes*—C.W. Vanderslice, Chairman, briefly reviewed the scope of the task group and problems associated with the current test method based on a single grade of sodium carboxymethylcellulose. New data showing significantly different viscosity responses by aqueous solutions of conventional and enzyme-resistant types of hydroxyethylcellulose (HEC) to a fixed level of enzyme (0.1 ppm) in a vinyl-acrylic flat paint were discussed. No differentiation was evident between the same test solutions for a common reducing agent added to a second sample of the same paint.

It was agreed that a new test method based on these two types of HEC be written up in ASTM format for use in a second round-robin before the June meeting. The chairman will supply all of the necessary materials for conducting the round-robin. Two vinyl-acrylic flat paints containing 0.1 and 1.0 ppm enzyme, and two vinyl-acrylic latexes containing differing levels of an oxidizing agent (to be determined) will be used for evaluating the ability of the proposed test method to distinguish between the two types of contaminants.

*D01.28.03—Microbial Quality of Raw Materials*—J. Hinkle, Chairman, had reviewed the proposed practice with his group, as well as a non-microbiologist, and made numerous clarifications based on their comments since the June meeting. A revised draft was distributed incorporating these clarifications as well as most of the changes proposed in the June 1991 meeting (notes on TTC indicator and sources of purchased plates were not yet added). Any other feedback from the attendees after further review of the practice was requested to be sent directly to J. Hinkle. A final draft will then be prepared for D01.28 ballot in time for the results to be reviewed at the June meeting.

*D01.28.04—Resistance of Paint Films to Algae Attack*—J. Hinkle, Chairman, circulated a draft of the procedure in ASTM format based on the outline presented by L. Rossmore in June. During discussion of the method, it was indicated that drawdown paper might be a more appropriate substrate than the glass filter disks. L. Rossmore agreed that this change could actually help the results by increasing the coating's susceptibility to algal growth. It was also mentioned that the algal species used were pri-

marily aquatic and that terrestrial species should be tested in the method. Again, feedback will be submitted to the chairman and a new draft prepared for possible future round-robin testing. O. Lesceiko agreed to provide data on algal test methods, including species used, from Abbot's old files if they could be located. These might be useful for the proposed drafts and round-robins.

**D01.28**—The minutes from June's meeting were approved without comments or changes. It was noted that attendance at the meeting was excellent with several new membership requests.

Discussions of the proposed new task groups for a fungal agar plate assay based on G 21, and for a revision of D 3273 to include exterior performance was held. M.C. McLaurin noted that their D 3273 chamber does not support *Aureobasidium pullulans* (*A.p.*), and that it has an opaque cover. J. Hinkle found that their chamber, which has a clear cover and is located under UV room lighting and near lighting from windows, does support *A.p.* The method does not specify materials of construction for the chamber cover. Specifications for UV transparency may be required to maintain this exterior fungal species in the chamber. J. Hinkle also reported he had found a company which can build chambers to meet the D 3273 method. This company suggested some updates to the antiquated control apparatus still described in the current revision. It was suggested that these proposed new task groups meet informally and concurrently with D01.28.03 and D01.28.04 sessions in June. J. Hinkle will chair these sessions prior to actually establishing new task group numbers.

#### **SUBCOMMITTEE D01.28 JUNE 1991 MINUTES**

**D01.28.01—Package Stability**—M.C. McLaurin, Chairman. The revised method was reviewed and accepted with the following amendments. All named organisms should have ATCC numbers to standardize the testing. Notation that the use of actual in-plant spoilage isolates when available (as agreed upon by supplier and vendor) would be very beneficial as additional inocula. Discussion of the use of spoiled paint as the inoculum led to proposals for allowing this as an option put into an Appendix for use as agreed upon by supplier and vendor. Potential concerns raised about this option included problems of test reproducibility. Also, contamination to paint is generally from external (nonpaint) sources rather than from preconditioned, spoiled paint additions. The committee agreed that it is not a good practice to attempt to recover spoiled paint by working it off into fresh batches. These and a few other proposed changes will be incorporated in a draft for discussion in January.

**D01.28.02—Rapid Determination of Enzymes**—C.W. Vanderslice, Chairman. The current proposed test method employing a specialized grade of sodium carboxymethylcellulose (CMC) to detect degradants in paints was reviewed. In light of a recent subcommittee ballot in which two negative votes were cast concerning this method, it was agreed that using only this substrate did not permit the proposed method to distinguish between enzymes and chemical degradants. Accordingly, both negative votes were unanimously voted to be "persuasive," resulting in substantial changes in the method.

It was agreed that the proposed method be rewritten to evaluate the suggestion that two hydroxyethylcelluloses (HEC) of differing uniformity of substitution be used as potential indicators of the source of such paint viscosity losses. Other procedural comments made by the negative voters will be incorporated as well.

A second round-robin will be conducted using latex samples inoculated with two levels of a commercial fungally-derived cellulose and a common reducing agent ("Formapon") often used in "finishing" latexes.

**D01.28.03—Microbial Quality of Raw Materials**—J. Hinkle, Chairman. Two revised drafts of the proposed practice were disseminated at the meeting. Several changes were suggested, including adding notes to the apparatus section as to using Tryptic soy agar plates with TTC indicator dyes, and for sources of purchased plates, etc. Several other minor changes were proposed. It was decided that this practice could be balloted without round-robin testing, since it was basically standard microbiological techniques. It was agreed that a non-microbiologist should attempt to follow the procedure as written to suggest any possible changes.

**D01.28.04—Resistance of Paint Films to Algae Attack**—J. Hinkle, Chairman. L. Rossmore circulated a draft of the procedure he developed based on G 21, and his testing results using a number of biocides following that procedure. It was generally agreed that this draft and test results looked very good and should be written into ASTM format for subcommittee balloting. While this is underway, examination of a few variables such as the length of time for incubation will be examined. Since insufficient copies of the draft were available at the meeting, they will be distributed by M. McLaurin based on the attendance list.

**D01.28—Biodegradation**—M.C. McLaurin, Chairman. The minutes from January's meeting were approved without comments or changes. It was noted that attendance at the meeting was excellent. Two ballot items were addressed.

A negative vote pertaining to reapproval of D 4610 was received, since it does not

specify a means for washing. Upon discussion, the negative was found persuasive. The reapproval of D 4610 was withdrawn and it is now under revision to include methods for "washing" as clarification. Also included will be alternate choices for washing to distinguish between those using the practice to repaint, and those using it to evaluate other properties (chalking, etc.) of the film beneath the microbial growth. Also included will be a special hazards warning for the washing procedures. A negative vote pertaining to the withdrawal of D 2574-86 was received, since there would be no interim method on the books until the revision is completed. After discussion, the negative was found persuasive, so D 2574 will not be withdrawn. D 2574-86 will also not be balloted for reapproval since it is currently under major revision. D 2574-86 will be allowed to remain on the books as is until the revision is approved.

The following topics were discussed:

(1) A new task group will be established to draft a standard practice on an agar plate method for determining the resistance of a dry film to fungal attack based on G 21. J. Hinkle will chair this task group. This should be applicable to paint and coatings (including caulks, sealants, etc.) on draw-down paper, wood, glass, etc., for interior or exterior exposure.

(2) A new task group will be established to propose a D 3273 revision to include applicability to exterior as well as interior performance. Items such as equipment specifications (e.g., clear top versus dark tank) should also be clarified or differences noted. M. McLaurin will chair this task group.

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## **DIVISION 30 PAINT MATERIALS**

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### **SUBCOMMITTEE D01.31 PIGMENT SPECIFICATION**

**D. Kesatie, Vice Chairman**

**D01.31**—The vice chairman stated that no volunteers have been found to champion a number of pigment standards. Thus, several standards will be submitted to D-1/D01.31 ballot for approval as written, including: D 081 (basic carbonate of white lead); D 261 (chrome green); D 262 (ultramarine blue); D 263 (chrome oxide green); D 267 (gold bronze powder); D 607 (wet ground mica); and D 656 (pure toluidine red toner). Several standards will be submitted to D-1/D01.21 ballot with intent to withdraw, including: D 912 (cuprous oxide); D 963 (copper phthalocyanine blue); and D 3021 (copper phthalocyanine green).

D 1649 on strontium chromate will be submitted to D01.31 ballot prior to the June 1992 meeting. A decision on D 867, pumice, was held in abeyance until the June 1992 meeting as a champion for it may have been found.

Two task group reports were submitted. W. Spangenberg chaired the discussion on a proposed standard for zinc phosphate. In terms of current published supplier specifications, the only consensus of opinion was for color (white) and specific gravity. Other reported characteristics of this pigment varied because of the pigment producers, the test method used, differences in operator, etc.

An ISO method is available as a guide and it differentiates between three types of zinc phosphate pigments, 1:  $Zn_3(PO_4)_2 \cdot 2H_2O$ ; 2:  $Zn_3(PO_4)_2 \cdot 4H_2O$ ; and 3: a blend of types 1 and 2. A fourth type may be needed in the ASTM standard as the ISO method only applies to untreated zinc phosphates and several producers are now modifying their pigments.

The ISO method sets a minimum specific resistance value (percent water solubles may be an equally valid characteristic). The percent chemical composition based on an ignited sample, pH, density, and percent plus 325 mesh should also be included in the standard. Oil absorption should be agreed upon between supplier and user, but this value should not vary more than - 10% for a given product once agreement is reached. The particle size distribution for a given product may also be specified.

M. Hendry chaired the discussion for the proposed standard on micaceous iron oxide. Because of its descriptive nature, micaceous iron oxide will be used for this pigment instead of lamellar iron oxide. ISO method 10603 is also in the process of being approved for micaceous iron oxide. Currently, this method only applies to the naturally occurring pigment. Synthetic products are being developed, possibly commercialized in the UK and Japan. Photographic standards are available to aid in the determination of Grade 1 (> 65% lamellar); Grade 2 (50-60% lamellar); and nonlamellar/nonmicaceous iron oxides (< 50% lamellar).

W. Spangenberg recommended that paragraphs 2 and 3 under the Scope section of the document be incorporated into the Significance and Use section. He also recommends that the term "grade" be changed to "type" in the finished document. Both recommendations were found to be persuasive. Other editorial comments were agreed upon, and a revised document should be available for balloting at the subcommittee level on the next appropriate ballot.

In new business, it was recommended by the group that the chairman of D01.31 contact the chairman of D-1 regarding improved relations and communications with ISO as D01.31 and the ISO committees are developing similar documents. This could

be accomplished either through D-1 or possibly through an ISO task group set up as part of D01.31.

## SUBCOMMITTEE D01.33 POLYMERS AND RESINS

### M.J. Mahon, Chairman

*D01.33.12—Urethanes*—M.J. Mahon announced that R. Briselli will be the new chairman of this task group following the resignation of P. Guevin, Jr. The new chairman will continue work on methods for analysis of residual isocyanate monomers in urethane polymers. Round-robin testing is expected to commence in the near future.

*D01.33—Polymers and Resins*—M. Mahon, Chairman, discussed recent efforts in the drive to obtain new D01.33 members. He reported he will continue asking resin manufacturers to more effectively support the subcommittee's activities.

A negative vote from D. MacGregor, on the recent society ballot of new standard D 5180, "Test Method for Quantitative Test for Turbidity in Clear Liquids," was discussed. D. MacGregor agreed to withdraw the negative vote if the word "transparent" were substituted for "clear" in the method's title and scope. The subcommittee approved this action and the editorially revised method will continue to the Committee on Standards.

## SUBCOMMITTEE D01.34 NAVAL STORES

### J. Russell, Chairman

*D01.34.01—Capillary Gas Chromatography of Rosin and Fatty Acids*—R. Raney, Chairman, distributed a draft of a proposed standard method to serve as a basis for discussion. It was agreed that before the chromatographic conditions could be established, it was necessary to establish the preferred methods for converting rosin and fatty acids to methyl esters. It was agreed that members would send copies of their methylating procedures to J. Russell and he would compile them and distribute them to D01.34. Members could then evaluate these methods using their own chromatographic conditions prior to the next meeting.

*D01.34.2—Capillary Gas Chromatography of Turpentine*—D. Stewart, Chairman, had developed the outline of a standard method. It was agreed to distribute this method for comment to interested laboratories with expertise in this area and incorporate the comments into the method prior to the next D01.34 meeting.

*D01.34.3—Softening Point of Rosin and Rosin Derivatives by the Automatic Mettler Apparatus*—P. Zawislak, Chairman, stated that she would prepare a draft of a standard method prior to the next meeting. Such a method is required as the Mettler method is replacing the established Ring and Ball Method (E 28) in some laboratories.

*D01.34*—The committee continued its review of the 21 standards that require revision. Of these standards, 15 had been sent to D01.34 ballot and, of these, six had also been sent to a D-1 ballot. At the present time, only one method, D 464, "Saponification Number of Naval Stores Products, Including Tall Oil Related Products," had passed all ballots. This method has passed final editing and will be published as a 1992 standard method. J. Russell reviewed the results of the D-1 ballot. Three methods, D 465, "Acid Number of Rosin," D 1065, "Unsapinifiable Matter in Rosin," and D 3009, "Composition of Turpentine by Gas Chromatography," all passed D-1 ballot without any negative votes and will be included in the next society ballot.

Three negative votes were received on E 28, "Softening Point of Rosin by Ring and Ball Apparatus." One voter recommended that this method be withdrawn and replaced by D 36, "Softening Point of Bitumen by Ring and Ball Apparatus." D01.34 voted unanimously to find this negative vote nonpersuasive because:

(1) D 36 does not require stirring of the heating medium and, therefore, requires corrections when changing from one heating medium to another.

(2) D 36 does not include any information on the preparation of rosin and rosin derivative test samples.

(3) E 28 is a widely used, well established, and widely cited standard method for testing rosin and rosin derivatives.

The steward of E 28, P. Zawislak, reported on the second negative vote. She reported that many of this voters' comments were justified and that the method should be corrected prior to further balloting. She also recommended that the precision and bias statement in the method be updated by including the results of the recently completed round-robin. P. Zawislak also reported that the third negative vote had been withdrawn after a phone conversation with the voter.

The following comments were made on methods that had been submitted for D01.34 ballot but no D-1 ballot.

D 1240, "Rosin Acids in Fatty Acid"—W. Mark reported that the D01.34 ballot had approved the method with no negative votes. However, a question remained about the reflux time required for the methylation of samples containing more than 15% rosin. He reported that various D01.34 members were currently carrying out trials to establish the required reaction time. The method will be resubmitted for D01.34 ballot when the results become available.



D 1585, "Fatty Acid Content of Rosin"—W. Mark reported that this revised method was approved in the D01.34 ballot with no negative votes and will be submitted for a D-1 ballot.

D 269, "Toluene Insoluble Matter in Rosin"—J. Daust reported that this revised method was approved in D01.34 ballot with no negative votes and can now be submitted for D-1 ballot. The method has been revised to include rosin derivatives in addition to rosin and now permits the use of solvents other than toluene.

D 801, "Sampling and Testing Pinene" and D 802, "Sampling and Testing Pine Oil"—D. Stewart reported that these two methods were approved without any negative votes in a recent D01.34 ballot. These methods will be submitted for a D-1 ballot.

Mr. Russell reported that drafts of the final six methods to be reviewed had been circulated prior to the meeting and these drafts were discussed.

D 803, "Testing of Tall Oil"—J. Russell pointed out that this method has undergone a major revision with most of the chemical methods being transferred from the original D 803 to other specific methods. For example, the test method for acid number had been deleted from D 803 and the reader referred to D 465. Such transfers eliminated duplication and also eliminated any differences that existed between duplicate methods. The method was approved as submitted with minor changes. It was noted that D 803 is a widely used and widely cited standard for testing tall oil and tall-oil products and so it was agreed that a paragraph be added to the Significance and Use section, stating that these changes had been made and why they had been made. Other changes to be made were to reference the Karl Fischer method for testing for water content, to use the "bubble time" method for measuring viscosity, and to recommend the use of an electric mantle rather than an oil bath for heating to dissolve rosin acid crystals. This method will be submitted for D01.34 ballot in February 1992.

D 509, "Sampling and Grading Rosin"—J. Russell had revised this method and circulated drafts prior to the meeting. This method was approved as written but it was mentioned that this standard no longer describes all the industry practices used for the sampling and grading of rosin. It was agreed that the D01.34 should address this problem at a later date and, in particular, develop a new standard for the measurement of the color of rosin. This method will be submitted for D01.34 February 1992 ballot.

D 889—"Volatile Oil in Rosin"—P. Zawislak had revised this standard and drafts had been circulated prior to the meeting. This draft was approved as written and will be submitted for D01.34 February 1992 ballot.

D 1131, "Testing Rosin Oils"—P. Zawislak had revised this standard and drafts had been circulated prior to the meeting.

Several minor suggestions to up-date the method were made. For instance, it was recommended that viscosity be measured by the "bubble time" method and that reference to the use of a Brookfield viscometer should also be included. These changes will be incorporated into the method and it will be submitted for D01.34 February 1992 ballot.

D 809, "Water in Liquid Naval Stores"—D. Stewart had circulated a draft copy of the revised method prior to the meeting. Several comments were made on this method and they will be incorporated into the method prior to D01.34 ballot in February 1992. For example, it was agreed that the Karl Fischer method was applicable at high levels of moisture in addition to low levels. Also, pyridine free Karl Fischer reagent is now commercially available and is generally preferred to the standard Karl Fischer reagent described in this standard.

D 1064, "Iron in Rosin"—W. Trainor had circulated a draft of the revised method prior to the meeting. After discussion on the need for a standard method for measuring trace metals in addition to iron in naval stores products other than rosin, it was decided to proceed with approval of this standard and to discuss the need for a broader standard at a later date.

A series of round-robins were scheduled with a completion date of September 30, 1992. Three materials, rosin, distilled tall oil and tall oil fatty acids, will be the test materials and the five methods to be included in this study will be: D 464 (saponification number); D 465 (acid number); D 1065 (unsaponifiable matter); D 1240 (rosin acid content); and D 1585 (fatty acid content). It was also agreed that future round-robins would be held on two automated methods for the determination of the softening point of rosin and rosin derivatives. A round-robin has been carried out on the manual Ring and Ball method (E 28) and the resulting precision statement will be included in the revised method currently under development.

The subcommittee also discussed new methods which included improved color standards for fatty acid and rosin and the crystallization resistance of rosin. No actions were taken on these topics.

It was agreed that the next meeting of D01.34 would be in Panama City, FL, on May 1, 1992. The subcommittee will not meet with D-1 in Minneapolis in June.

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

**G.Y. Moore, III, Vice Chairman**

J.S. De Wit reported the results of a round-robin test of falling ball viscosity of cellulose acetate and cellulose acetate pro-

ponate. Eight laboratories participated, and the statistical data were generated using ASTM E 691. Material with an average viscosity of 20.35 seconds was found to have a repeatability (r) of 1.28 seconds and a reproducibility (R) of 2.64 seconds. Material with an average viscosity of 57.13 seconds was found to have a repeatability (r) of 4.91 seconds and a reproducibility (R) of 8.92 seconds. This data will be incorporated into a rewrite of D 1343, "Standard Test Method for Viscosity of Cellulose Derivatives by Ball-Drop Method," and submitted for D01.36 ballot before the June meeting in Minneapolis.

The only method this subcommittee is responsible for which is due for review in 1992 is D 4085, "Standard Test Method for Metals in Cellulose by Atomic Absorption Spectrophotometry." J.H. May is steward for this method, but he was not present at this meeting. G.Y. Moore will contact him to see if he can conduct the review, make the necessary revisions, and submit the method for D01.36 ballot before the June meeting in Minneapolis.

Participation in D01.36 has decreased significantly in the past several years. G.Y. Moore and J.S. De Wit will jointly author a letter to be sent to users of cellulose and cellulose derivatives to invite them to join ASTM and specifically to attend the June meeting in Minneapolis.

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## **DIVISION 40 PAINT PRODUCTS APPLIED ON SITE**

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### **SUBCOMMITTEE D01.41 PURCHASE OF PAINTS AND RELATED COATINGS**

**C. Metz, Chairman**

J. Weaver presided in the absence of L. Rogers, Chairman, who has resigned. The subcommittee decided that no further action will be taken on D 4717 which failed to pass an earlier ballot. The subcommittee approved a new scope at the June 1991 meeting. J. Weaver agreed to present the following new scope for D01.41 to the executive committee: Scope—To develop guides for the selection of finished products for individual consumers, and for government and general industry use; and to serve as a forum to address the needs and concerns of these users for matters under the jurisdiction of Committee D-1. D01.41 recommended that D01.31 on Consumer Affairs and D01.07 on Government Standards be combined into D01.41. J. Weaver agreed to present this proposal to D01.93 on Bylaws and Scopes.



D01.41 agreed to recommend to D01.90 that C. Metz of the Department of Defense, be appointed chairman. (D-1 Chairman K. Trimmer announced her as chairman during the D01.90 Wednesday meeting. She agreed to research and report back to the group on potential activities for D01.41 at the upcoming meeting in Minneapolis.)

## **SUBCOMMITTEE D01.42 ARCHITECTURAL FINISHES**

### **L. Schaeffer, Chairman**

*D01.42.03—Porosity of Paint Films—C. Tatman, Chairman.* The meeting was chaired by R. Hopkins in the absence of the regular chairman. A brief review of the previous round-robin was presented and copies of the current procedure were handed out. Several problems with the technique were uncovered during that round-robin. They were related to the film thickness of the test drawdowns, the removal of excess saturants from the surface of the film, and the drying conditions. New data was introduced in these connections to help define what changes should be made in the next round of testing. It was agreed that a 4-mil clearance applicator would be used for waterborne coatings and a 3-mil clearance for solventborne coatings. Two odorless mineral spirit wipes followed by dry wipes would be incorporated as the washing procedure. Insufficient data was available to evaluate the effect of drying conditions. The USP designation of the mineral oil will be specified. Another round-robin is planned before the next meeting with the help of four cooperators.

*D01.42.04—Wet Adhesion of Latex Paints—C.W. Vanderslice, Chairman,* reviewed problems concerning loss of adhesion of the alkyd gloss enamel test surface to the unfinished western red cedar panels used in the method. The chairman discussed test results done in his lab to address this problem. Changing the water contact time from 2 hours to 30 minutes appeared to correct the problem, except in cases where the wet adhesion of a test paint was so high that even the alkyd disbonded from the panel. The results also showed that the use of a latex primer beneath the gloss alkyd gave wet adhesion values which were more reproducible in duplicate trails. The present method will be revised and another round-robin will be run using shorter water contact time. Two exterior flat paints, two trim paints, and a fail control will be tested and six cooperators will participate.

*D01.42.09—Color Development in Tinted Latex Paints—F. Marshall, Chairman,* handed out a copy of Draft #7 of the proposed method, which had just gone through D-1/D01.42 concurrent ballot and received four negatives, mostly pertaining

to colorimetric concepts and definitions. The negatives were considered persuasive and various revisions in response to them were discussed. The title of the standard was discussed with suggestions for revising this too. The members concluded that the draft should be withdrawn and a revised draft submitted again to D-1/D01.42 ballot. The question of the title revision was left to the judgement of the task group chairman.

*D01.42.16—Hiding Power by Roller Application—J. Price, Chairman,* reported that minor revisions in D 5150 had gone through D-1 ballot with one negative received from P. Guevin that was subsequently withdrawn. This related to the absence of a research report, and was not relevant to the material being balloted. The chairman will address P. Guevin's negative later by submitting a research report to the subcommittee for approval prior to filing it with headquarters. Editorial comments were received from H. Ashton and the revisions to address them will be included in the final text. The revision just approved by D-1 will now proceed to society ballot.

*D01.42.40—Water Repellents on Wood—V. Scarborough, Chairman,* presented the group with comments received from the last D-1/D01.42 concurrent ballot and each comment was discussed. Most of them were found to be editorial in nature, but an extensive negative from J. Berry contained a number of valid points requiring technical revisions. It was therefore decided to withdraw the whole document for another concurrent ballot.

*D01.42.21—New Project Planning—S. LeSota, Chairman,* reported that this group has thus far made two proposals, one for developing a test for Burnish Resistance and another for modifying Method D 2354, "Minimum Film Formation Temperature." Both proposals are now in the process of implementation, with Burnish Resistance as a new task group. Other ideas under consideration are heat stability tests and alkali resistance tests. Anyone having ideas with regard to useful test methods for ASTM development should contact the chairman.

*D01.42.22—Guides for Testing Architectural Coatings—H. Ashton, Chairman,* reported that the fourth draft of the combined Guide for Testing Waterborne Architectural Coatings had not been submitted to concurrent D-1/D01.42 ballot because there were several points on which a consensus had not been reached. The meeting was devoted to reviewing the comments and results from the D01.42 ballot and from a later supplementary ballot. The chairman will revise Draft #4 for D-1/D01.42 spring ballot. He announced that he would take the same approach as with D 5146 in that only clarifications approved in the last subcommittee ballot would be included in the revision.

Suggestions for new methods or properties would be held for the first subsequent revision, to be started when the current document goes to society ballot. The chairman acknowledged the special efforts on the work of the combined guide by the following members: R. Schiller, R. Rydewski, D. MacGregor, C. Tatman, D. Vanderweide, T. Sliva, B. Jenkins, J. Brezinski, L. Newman, F. Winkleman, T. Rieth, and J. Robbins.

*D01.42.24—Application Properties of Paints—J. Price, Chairman,* discussed the concept of Natural Spreading Rate, and whether it is possible to measure such a property with a reasonable degree of objectivity and reproducibility. Five cooperators were obtained for a research round-robin to try to determine a suitable approach to this problem. The chairman will provide a suggested procedure and materials for this purpose. H. Ashton will submit methods for consideration that he has on file from other organizations.

*D01.42.26—Burnish Resistance of Coatings—C.W. Vanderslice, Chairman,* distributed a test method for burnish resistance proposed by S. LeSota. This included both an instrumental and a manual procedure. Preliminary data was shown for wet and dry burnishing tests, performed using the instrumental procedure. From a discussion of this data, a round-robin was planned in which both wet and dry burnishing would be measured instrumentally on a series of three wall paints ranging from eggshell to contractor type flats. The paints will be supplied by a paint producing member of the task group. The chairman will write a working draft for the round-robin after further preliminary tests to decide whether the 200 cycles of abrasion called for in the originally proposed method should be reduced to avoid excessive film erosion of high PVC paints. Four cooperators will participate in the first round-robin.

*D01.42—Balloting actions* included successful D-1 ballots on revisions of D 1849-91, "Test Method for Package Stability of Paint," D 5150-91, "Hiding Power by Roller Application," D 3129-91, "Guide for Testing Latex House Paints," and D 4540-91, "Guide for Testing Latex Semi-Gloss Paints." A new standard, D 2064-91, "Test Method for Print Resistance," passed society ballot. Two proposed new standards, "Color Development in Tinted Latex Paints" and "Evaluation of Water Repellents for Wood," received numerous negative comments on D-1/D01.42 ballot and were withdrawn for revisions and subsequent rebaloting. It was agreed to hold for discussion at the June meeting, a proposal to withdraw the four solventborne coating guides that were combined into D 5146, namely D 2932, D 3323, D 3383, and D 3425. Waterborne types, D 2931 and D 3358, will be

submitted for reapproval without change while the combined Guide for Testing Waterborne Architectural Coatings is being completed. A new task group chaired by V. Scarborough was formed, namely D01.42.28, Efficiency of Paint and Varnish Removers, to develop a method for evaluating products of this type. All those interested in participating should contact the task group or subcommittee chairman.

## **SUBCOMMITTEE D01.45 MARINE COATINGS**

### **C. Stanley, Chairman**

*D01.45.08—Organotin Leach Rate*—C. Biter, Chairman. Method D 5108 has been published which is restricted to organotin compounds. The chairman raised the question of the scope of this method and whether it should be useable for measuring the leach rate of other antifouling toxicants since both U.S. and Canadian governments are starting to investigate the expansion of the scope of D 5108.

*D01.45.13—Biofouling of Treated Surfaces*—C. Perez, Chairman, presented a draft method he had prepared. The draft caused some confusion in its scope as it covered both biofouling resistance and physical performance. Also, the physical performance ratings in some cases were not to standard ASTM methods. It was proposed that two separate methods be worked on, the first on biofouling evaluation and the second on physical performance. The committee would like to acknowledge the contribution made by C. Perez to these methods as he has in effect written both methods and provided us with excellent starting points to complete these tasks.

*D01.45.12—Intermittent Antifouling Immersion*—R. Gangi, Chairman, has asked for more input from the committee on this method. C. Perez has volunteered to act as Vice Chairman for this task group. Discussions were held on the title of this method and a new title was approved, Standard Practice for Testing Marine Coating Systems in Air/Water Cycling.

*D01.45*—Discussion of a review of proposed marine paint methods developed by F.25.01 by selected Washington Paint Technical Group (WPTG) personnel occurred with WTPG on an informal basis. WPTG will make an effort to obtain a review of a single method provided the proposed draft is submitted to the president of WPTG. Reviewers for a chapter on Marine Paints in the *Gardener Handbook* were requested. B. Allanach and C. Stanley volunteered and H. Stoner and F. Winkleman were suggested. A new task group to develop a method for testing the adhesion of fouling

to coated surfaces was formed. G. Swain agreed to chair this task group.

## **SUBCOMMITTEE D01.46 INDUSTRIAL PROTECTIVE PAINTING**

### **K.A. Trimmer, Chairman**

*D01.46.02—Surface Preparation*—K.A. Trimmer, Chairman, reported that ASTM D 2200, "Standard Pictorial Surface Preparation Standards for Painting Steel Surfaces," has successfully completed the 1991 society ballot and has been reissued.

D 4417, "Standard Methods for Field Measurement of Surface Profile of Blast Cleaned Steel," received a negative on the society ballot. The negative voter, K.A. Trimmer, indicated that there were errors in the new Precision and Bias statement. The negative was found persuasive and the appropriate changes made. The revised standard will be submitted to a concurrent D-1/D01.46 spring 1992 ballot.

D 2092, "Standard Practice for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting," is due for reapproval. D01.46 has approached D01.53 "Coil Coated Metal" to determine their interest in assuming custody of the practice since many of the methods are performed as a shop operation rather than as part of a field painting operation. D01.53 agreed that some of the methods are shop-related, but indicated that D01.53 expertise is limited to coil coated steel and may not be applicable to hot dip galvanized parts or steel members. They agreed to participate in a review of the document, but decided that it would not be appropriate to assume custody.

In order to proceed with the reapproval process, B.D. Flowers agreed to update the standard and to add field methods of preparation such as abrasive blast cleaning and water washing/solvent cleaning. The revised standard will be sent to a D01.46 spring 1992 ballot. While the reapproval is underway, D01.46 will continue contacting other subcommittees that may have greater expertise in this area. One suggestion is B-8, "Metallic and Inorganic Coatings."

The chairman also reported that the Department of Defense adopted D 2092 on July 28, 1991 to replace MIL-T-12879A (MR). The custodian is Army-MR.

*D01.46.04—Adhesion*—M.E. McKnight, Chairman, reported that the revision to D 4541, "Standard Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers," received two negatives (B.R. Appleman and R.K. Fisher) in the D01.46 fall 1991 ballot. Both negatives questioned the need and validity of the sentences added to the scope. The task group found the negatives to be persuasive and

made appropriate changes. The Appleman negative also identified an error in the equation found in X.2.2 ( $R_0$  appears missing to the left of the parentheses), and questioned the need for all of the columns of data found in Table I. The table will remain as is, however, as it is consistent with ASTM format, although the values in the third column will be rounded off. The revised method will be submitted to a concurrent D-1/D01.46 spring 1991 ballot.

*D01.46.03—Repainting*—G.W. Gardner, Chairman, reported that Draft #4 of the proposed "Standard Practice for Assessing the Solvent Resistance of Organic Coatings Using Solvent Rubs" had been submitted to a D01.46 fall 1991 ballot. One negative was received (M.E. McKnight) with comments from J.M. Nielson. The negative stated that by requiring the coating manufacturer to specify the solvent to be used, it implies that the procedure is only done for coating work in progress. Because the method may also be used for determining the solvent sensitivity of an old unknown coating, 4.4 should be rewritten to state that the manufacturer "may" specify the solvent. The reference to the solvent used in Appendix A will also be changed from "manufacturer specified solvent" to "solvent used." Section 7.1 will also be rewritten because the information to be reported in Appendix A is more complete than listed in 7.1.

The comment from J.M. Nielson suggested that a better definition of the cloth is necessary. The task group recommended that no changes be made at this time, but that the influence of the type and texture of the cloth be determined in a round-robin test. It is hoped that the round-robin can be conducted during the June 1992 meeting. This would eliminate any variables associated with the cure of the coatings should the round-robin be conducted through the mail.

D 4752, "Standard Test Method for Measuring MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub," was submitted to a D01.46 fall 1991 reapproval ballot with a few modifications. No negatives were received, but comments were issued by J.G. Raska and M.E. McKnight. J.G. Raska indicated that the use of water in 7.2 to clean the surface prior to testing may influence the cure of the zinc, and as a result, effect its solvent resistance. The task group agreed and will add the use of cheese cloth without water as an option, and add a sentence "Note that the use of water may influence the cure of the zinc-rich primer." M.A. McKnight recommended deleting "Immediately" from 7.4 and adding it to 7.5. The task group agreed. The task group recommended that both standards be submitted to a concurrent D-1/D01.46 spring 1992 ballot.

*D01.46.07—Inspection*—B.D. Flowers, Chairman, reported that he is compiling the changes from previous ballots of D 3276,

"Standard Guide for Painting Inspectors (Metal Substrates)," and will have a revised copy ready for a D01.46 spring 1992 ballot. It was also recommended that D-33 be contacted for possible liaison due to their interest in this area.

A parallel guide, "Standard Guide for Painting Inspectors (Concrete and Masonry Substrates)," has been under development for a few years. It will be resubmitted with a goal of submitting it to a D01.46 ballot prior to the June 1992 meeting.

*D01.46*—K.A. Trimmer, Chairman, reported that he is resigning as Chairman of D01.46 at the completion of the January 1992 meeting because of his new position as Chairman of D-1. The new D01.46 Chairman is G.W. Gardner.

Because of the problems associated with the removal of hazardous paints (e.g., lead-containing), D01.46 has formed new task groups to develop procedures for the field collection of samples for laboratory analysis. The tentative titles of the standards and task group identifications are as follows:

— Field Sampling of Paint for Laboratory Analysis of Heavy Metal  
(Task Group D01.46.12—Field Paint Sampling)

— Field Sampling of Soil for Laboratory Analysis of Heavy Metals

(Task Group D01.46.13—Field Soil Sampling)

— Field Sampling of Paint Removal Debris for Laboratory Analysis of Heavy Metals

(Task Group D01.46.14—Field Debris Sampling)

A single organizational meeting of the task groups will be held at the June 1992 meeting. This will be handled under D01.46.12. Prior to the meeting, a draft of the paint and soil sampling methods will be circulated for subcommittee review. After the June meeting, it is intended that the task groups begin meeting separately. New members are being actively solicited to participate in the development of these practices. The task group Chairman is K.A. Trimmer.

D 1014, "Standard Method for Conducting Exterior Exposure Tests of Paints on Steel," is due for reapproval in 1993. It will be submitted to a D01.46 spring 1992 reapproval ballot.

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

**F. Gale, Chairman**

*D01.47.01—Water Repellency of Treated Masonry*—V. Scarborough, Chairman, reviewed the work previously conducted by the task group using brick as the test substrate. It was the decision of the task group to revise its title and scope to evaluate

the efficiency of water repellent materials using masonry units rather than brick as the test substrate. Because of the poor interlaboratory correlation of the method using common brick, it was felt that by specifying the preparation and composition of the 4" x 4" masonry unit some of the variables inherent in the test substrate may be minimized.

Draft #1 of the "Proposed Test Method for the Determination of the Water Repellency of Treated Masonry" was distributed and reviewed in part by the group. It was the decision of the task group to initiate a round-robin of the new method using three materials and two methods of application; namely, a 10 second immersion and a brush application at a specified spreading rate. The chairman will report the results of the round-robin and submit Draft #2 of the method for review at the next meeting.

*D01.47.02—Chemical Resistance of Treated Masonry*—P. Thibaudeau, Chairman. In the absence of the task group chairman, the meeting was chaired by F. Gale. F. Gale reviewed Draft #3 of the "Proposed Test Method for Determination of the Chemical Resistance of Concrete and Masonry Treated with Water Repellent Penetrating or Surface Coating Sealers." A discussion followed on the suggested immersion solutions in the method. It was felt that some oils do not effect the efficiency of water repellents, but do have an affect on concrete substrate. It was suggested that a separate test was needed to determine whether oils penetrate through the sealer into the concrete.

It was the decision of the task group to initiate a round-robin using only salts and atmospheric contaminants as the immersion solutions. Two-inch mortar cubes will be used as the test substrate and treatments will be applied by a 10 second immersion. The chairman will report on the results of this round-robin and distribute Draft #4 of the method at the next meeting.

*D01.47.03—Nonvolatile Content of Silanes, Siloxanes and Silane/Siloxane Blends Used in Masonry Water Repellent Treatments*—T. Sliva, Chairman, updated task group members on the status of D 5095, "Determination of the Nonvolatile Content of Silanes, Siloxanes and Silane/Siloxane Blends Used in Masonry Water Repellent Treatments." The chairman reported that the revision to D 2832, "Standard Guide for Determining Volatile and Nonvolatile Content of Paints and Related Coatings," to include D 5095 into the guide had successfully completed concurrent D-1/D01.21 balloting.

The revision to D 5095 to specify the use of triplicate rather than duplicate determinations had completed society ballot and the research report number had been included. The chairman also discussed the inclusion of the method into the MNL-4

VOC reference manual currently under revision.

*D01.47.05—Permeability of Treated Masonry Substrates*—V. Mertz, Chairman, distributed Draft #3 of the proposed "Test Method for Permeability of Treated Masonry Substrates" and reviewed the results of recent screening tests. Water uptake was evaluated by immersing the brick longitudinally into the test solution except for the bottom 2 inches, which was left uncoated. The brick was placed longitudinally into a 2-inch head of water. Water absorption was accomplished by capillary action over 24 hours. The other bricks were coated on all four sides and one face. After dry, they were submerged in water with a 1/2-inch head for 24 hours.

It was the decision of the task group to initiate a round-robin with the method. The coatings will be applied to all four sides and one face and then immersed in water with a 1/2 inch head. The chairman will submit Draft #4 of the method and the results of this round-robin at the next meeting.

*D01.47.06—Freeze/Thaw Resistance of Treated Masonry*—L. Stark, Chairman, distributed the results of a screening of the two suggested preconditioning conditions, namely, the oven (minimum moisture content) and water soak (maximum moisture content) precondition methods. All samples had completed 30 freeze/thaw cycles at the time of the meeting.

After review of the data, it was the decision of the task group to initiate round-robin testing of the method using the oven preconditioning method. The chairman will incorporate suggested editorial and technical changes into Draft #3 of the method and distribute the revised method and the results of the round-robin testing at the next meeting.

*D01.47.07—Alkali Resistance of Masonry Treatments*—V. Repkin, Chairman, passed out copies of Draft #4 of the proposed "Test Method for Determining the Alkali Resistance of Treated Masonry Surfaces" and the results of the second round-robin. Each test material was applied to 10 blocks. Five blocks were immersed in water and five blocks in 0.1N KOH. Weight changes were recorded after 1, 7, 14, and 21 days immersion.

The test results showed a need to specify additional preconditioning of the test substrates and a significant variation in the amount of test material absorbed by each test substrate from lab to lab. The next round-robin testing will include a step to specify the amount (weight) of test material to be absorbed by each test substrate, rather than specifying just a time for dipping. There was further discussion on the effects of different types of substrates, the need for a more severe test medium, and the effect of the age of the test substrates on the results of the method.

It was the decision of the task group to initiate another round-robin of the method. The chairman will revise the test method and report the results of the round-robin at the next meeting.

*D01.47.08—Rapid Chloride Ion Intrusion*—C. Colleta, Chairman. Did not meet in the absence of the chairman.

*D01.47.09—Crystalline Treatments*—B. Harrill, Chairman, began the meeting with a review of crystalline treatments and a discussion on the focus of the task group. At the present time, there are not many task group members with an interest in these materials. It was the decision of the task group to contact other manufacturers and users of crystalline treatments and initiate publicity to increase interest.

The chairman has agreed to evaluate the existing test method for Wind Driven Rain as outlined in Federal Specification TT-C-555B and determine if it could be updated and used as an ASTM procedure for masonry treatments. In addition, the chairman will evaluate methods for treatments applied to below grade masonry for resistance to hydrostatic pressure. The chairman will report his findings at the next meeting.

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## **DIVISION 50 PAINTS FOR FACTORY APPLICATION**

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### **SUBCOMMITTEE D01.51 POWDER COATINGS**

**N.D. Emily, Chairman**

*D01.51.01—Polymeric Powders and Powder Coatings*—E. Waddles, Chairman. The results of concurrent balloting for the revision of D 3451, "Standard Practices for Testing Polymeric Powders and Powder Coatings," were the primary subject for the task group. Three negative votes and two affirmative with comments were received on the D-1 ballot. The three negatives were withdrawn after consultation with the voters. The two comments were discussed with two parties preceding and during the task group session to address these concerns. D 3451 will be presented for society ballot in 1992.

*D01.51.02—Hiding Power of Powder Coatings*—M.P. Sharma, Chairman. A new test method for determining powder hiding characteristics was discussed in the task group with L. Schaeffer (Chairman, Task Group D01.26, Optical Hiding Power), in an attempt to improve techniques for developing powder film thickness uniformity on

metal Leneta black and white contrasting substrate test panels. Another round-robin will be conducted during early 1992 to address specific procedures for powder application to the Leneta test panels.

*D01.51.03—Spray Characteristics of Powder*—C. Merritt, Chairman. This task group will be investigating methods for measuring first pass transfer efficiency of organic powder coatings with the objective of generating a standard method for this determination. First pass transfer efficiency of powder is recognized by industry as a separate and significant application characteristic from that of "utilization" efficiency. Interested persons, associated with the powder industry, are encouraged to join in this task group investigation.

*D01.51.04—Optical Properties of Powder Coatings*—H. Fairman, Chairman. This new task group was created to develop a guide for the determination of appearance properties of gloss, color, and color difference of powder coatings.

*D01.51.05—Test Method for Specific Gravity of Coating Powders*—S. Hart, Chairman. This new task group was formed to develop a method for the determination of specific gravity of Coatings Powders. Individuals interested in this investigation should contact either the task group or subcommittee chairman through ASTM Headquarters.

*D01.51.06—Review of Particle Size Measuring Techniques*—D. Pont, Chairman. Preliminary investigation will focus on a review of current industry methods of determining particle size and distribution. The first formal discussion will occur at the D-1 summer 1992 meeting.

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

**S.B. Schroeder, Chairman**

*D01.52B—Hardboard*—S.B. Schroeder, Chairman, reported that the new "Cobb Ring" water permeability method and a much revised D 2793, "Standard Test Method for Block Resistance of Organic Coatings on Wood Substrates," are both ready for D01.52 ballot. There was discussion of the less than desired repeatability and reproducibility of the data from the first Cobb Ring round-robin, and, although it was decided to go forward with balloting, a future, additional round-robin was proposed. T. Rieth prepared the D 2793 revision.

*D01.52.13—Prefinished Hardboard*—T. Reith, Chairman, reported that, based on recent comparative exposure data showing useful correlation, and acceleration factors

of approximately 10 fold relative to 45 South exposure fences, a proposed standard practice has been drafted. This practice for a concentrated sunlight/soak/freeze/thaw accelerated weathering test will be submitted to D01.52 ballot. Additional data using this new accelerated weathering test is still being developed. A series of primed hardboard is being tested to compare relative acceleration factors with the previous favorable prefinish data.

*D01.52.15—Film Thickness*—S.B. Schroeder, Acting Chairman for R. Matejka, reported that P. Guevin had withdrawn his D-1 negative ballot on the revised D 2691, "Standard Test Method for Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products." However, his comments were found to be valuable editorial refinements which were incorporated in the method. In the absence of the chairman, planning of the proposed round-robin to develop precision and bias was deferred to the summer meeting.

*D01.52*—The meeting was again poorly attended by wood product producers. This frustrated plans to discuss industry needs for new test methods and limited the discussion to on-going work. It was reported that D 3919, "Standard Test Methods for Quantifying Dirt Collection on Coated Exterior Panels," would go to D01.52 ballot with a February 2 deadline, and it was voted to submit to concurrent D-1/D01.52 ballot both D 358, "Standard Specification for Wood to be Used as Panels in Weathering Tests of Coatings," and D 4144, "Standard Method for Estimating Package Stability of Coatings for Ultraviolet Curing."

### **SUBCOMMITTEE D01.53 COIL COATED METAL**

**R.J. Tucker, Chairman**

*D01.53.01—Pretreatment of Metal Substrates*—H.E.G. Rommal, Chairman, asked D.C. Steele to discuss his progress in writing a rough-draft practice which would include both ASOMA and Porta Spec X-ray fluorescence techniques, capable of measuring concentrations of many elements. After much discussion it was agreed that H.E.G. Rommal and D.C. Steele would prepare a rough-draft before the next meeting, exclusive to the use of the Porta Spec, measuring the element chrome on any substrate.

*D01.53.03—Accelerated Weathering*—D.A. Cocuzzi, Chairman, discussed the general program to evaluate the degree to correlation between a fluorescent UV-condensation cabinet and natural weathering (45° south, south Florida, open rack). Samples are to be collected by coil coaters and submitted with generic descriptions to



D.A. Cocuzzi. Samples will be submitted to two south Florida sites, with two different start times (spring and autumn) to gather real-time data. At some point in the future, accelerated weathering will begin, and Spearman Rank Correlation will be calculated to quantify the degree of correlation.

**D01.53—Coil Coated Metal**—R.J. Tucker, Chairman, discussed D 2092, "Recommended Practices for Preparation of Zinc-Coated Steel Surfaces for Painting," the rewriting of which D01.46 Chairman, K.A. Trimber, sought assistance. D01.53 members will assist when possible. D 3794, "Practice for Testing Coil Coatings," D-1 ballot received two negatives, both of which were resolved. D 3363, "Test Method for Film Hardness by Pencil Test," is due for D01.23 ballot next. D 3003-71, is up for reapproval. D 4447, "Applying Coil Coatings Using the Wire Drawdown Bar," is to be distributed with the minutes and discussed at the next meeting.

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

**G.R. Pilcher, Chairman**

**D01.55.06—Coatings on Plastics**—H.F. Haag, Chairman, reported that D 3002, "Coatings on Plastics," must be extensively revised, and that input from the wood, automotive, building, computer, and possibly other industries, will be needed. A survey will be conducted, and comments solicited through short articles in *JOURNAL OF COATINGS TECHNOLOGY*, *American Paint & Coatings Journal*, and *Standization News*.

**D01.55.08—Transfer Efficiency—Laboratory Conditions**—R.J. Diem, Chairman, reported that negatives cast against the proposed new "Standard Practice for Evaluating and Comparing Transfer Efficiency under General Laboratory Conditions" are in the process of being resolved. The revised document will be resubmitted for D-1 ballot.

**D01.55.09—Transfer Efficiency—Production Conditions**—J.S. Komjathy, Chairman, indicated that the new method, "Practice for the Determination of Transfer Efficiency Under General Production Conditions for Spray Application of Paints," is now ready for a concurrent D-1/D01.55 ballot.

**D01.55.10—VOC of Radiation-Cured Coatings**—Leland H. Carlblom, Chairman, presented a draft method for determination of, "Volatile Content of Radiation Curable Materials," which had recently undergone round-robin testing in the laboratories of member companies of RADTECH International. The amended draft will be submitted

for D01.55 ballot, and will also be sent to D01.21 for comment.

**D01.55.11—Scratch Resistance of Automotive Topcoats**—J.S. Komjathy, Chairman, reported that the start-up meeting addressed the lack of a method to measure the resistance of automotive clearcoats to marling. A working document was presented for discussion by the task group, and comments will also be sought from D01.21.14, which is working on a similar document. Comments will also be sought from the wood industry, which might also have a need for this type of test.

**D01.55**—G.R. Pilcher indicated that five of the 11 methods/practices up for review in 1991 and still not resolved—two are on hold, pending an industry survey, and the remaining three need to be reviewed by appropriate stewards. An article will be placed in several journals, seeking comment for the revision—or the withdrawal—of D 2199, "Method for Measurement of Plasticizer Migration from Vinyl Fabrics to Lacquers."

## **SUBCOMMITTEE D01.56 PRINTING INKS**

**J.M. Fetsko, Chairman**

**D01.56.01—Fineness of Grind**—J. Cichon, Chairman, reported that the five-year update of test method D 1316, "Fineness of Grind of Printing Inks by the NPRI Grindometer," was balloted without negatives or comments. There were no changes other than the addition of keywords.

**D01.56.02—Lightfastness of Prints**—J. Robins, Chairman, reported that the D-1 ballot of revised test method D 3424, "Lightfastness and Weatherability of Printed Matter," received three negative votes, all of which were withdrawn. The method will be included in the April 1992 society ballot. Existing studies will be examined with a view toward developing a precision statement.

**D01.56.11—Nonvolatile Content**—B. Blom, Chairman, reported that test method D 4713, "Nonvolatile Content of Printing Inks," is up for review. It was agreed that the title should include "heatset and liquid."

**D01.56.12—Viscosity of Liquid Inks**—J. Daugherty, Chairman, reported that an in-house study demonstrated that the Shell cup gave much more reproducible results than the Zahn. Test method D 4212, "Viscosity by Dip-Type Cups," will be examined for applicability to liquid ink systems.

**D01.56.14—Setting of Heatset Inks**—D. Ness, new Chairman, reported that the biggest complaint from labs who cooperated in

the previous round-robin on the Sinvatrol was the drying of the test inks on the rollers of the printability tester. He will examine the usefulness of a wedge printing plate.

**D01.56.17—Guide for Printing Inks**—A. Scarlatti, Chairman, reported that D 5010, "Guide for Testing Printing Inks and Related Materials," will be updated to reflect new standards for the GA CAT, coarse particles, and others that may arise.

**D01.56.19—Printing Strength and Opacity**—G. Wouch, new Chairman, agreed to make a literature survey and conduct exploratory studies on a test method, which will involve making densitometer readings on weighed prints on black and white paper strips.

**D01.56.20—Technical Coordination**—B. Blom, Chairman, reported that he is following the activities of a number of other ASTM task forces concerned with VOC of radiation-cured coatings, volatile content of microwaved packaging, Sutherland Rub Tester, recycling, and opacity.

**D01.56.21—Print Abrasion**—G. Vandermeersche, Chairman, reported that the new test method, "Abrasion of Printed Matter by the CAT Comprehensive Abrasion Tester," has been assigned the number D 5181. A negative from the society ballot questioning the use of circles to indicate changes was voted nonpersuasive by 10-0.

**D01.56.22—Drying of Oxidizable Inks**—B. Blom, Chairman, reported that a round-robin was conducted in which five labs used a modified Sutherland Rub Tester to determine the drying time of three oxidizable inks by squalene resistance of freshly prepared prints. Results will be statistically analyzed.

**D01.56.23—Degree of Dispersion (Microscope)**—A. Mercado, new Chairman, reported that he will conduct an in-house study to improve the precision of a proposed new test method for degree of dispersion by microscopy.

**D01.56.24—Grit Determination**—M. Fuchs, Chairman, reported that the D-1 ballot of the proposed new test method, "Coarse Particles in Printing Ink Dispersions," received two negatives, both of which were withdrawn. The method has been assigned the number D 2067 and will be included in the March 1992 society ballot.

**D01.56.27—VOC40 of Non-Heatset Paste Inks**—P. Ford, Chairman, reported that the D-1 ballot of the proposed new test method, "VOC40 of Non-Heatset Paste Inks," received three negative votes and several comments. Negatives from H. Fujimoto objecting to the term "VOC" and from M. Urmezis objecting to statements in the "Significance and Use" section were voted nonpersuasive by 13-0. A late nega-



tive from W. Golton questioning the precision of weight-per-gallon measurements on paste inks and comments from R. Dela Cerna suggesting the addition of D 3792 (water by gas chromatography) require revision and rebalancing.

**D01.56.28—Tinting Strength of Liquid Inks**—D. Ness, Chairman, reported that he found a commercially available tinting base (Pittsburgh Paint 80-160) that may serve as a universal base for aqueous inks. The problem remains on how to develop a sufficiently thick drawdown as required for instrumental measurements. A special cell that will accommodate liquid inks was suggested.

**D01.56.29—Membership**—J. Daugherty, Chairman, welcomed new members G. Wouch, P. Neu, A. Mercado, and R. Dix.

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

**M.D. Gottsegen, Chairman**

**D01.57.02—Lightfastness of Pigments**—T. Vonderbrink, Chairman, entertained a motion, in response to old business from J.T. Luke, to add lightfastness categories IV and V to section 8 of D 4303. Lightfastness category IV will include pigments which show a  $\Delta E$  of 16 but not more than 24 units, and lightfastness category V will include pigments which show a  $\Delta E$  of more than 24 units with no upper limit. Pigments with ratings higher than category II are not sufficiently lightfast to be used in paints that conform to D 4302, D 5607, and D 5098, but the new categories have been suggested for information purposes and to accommodate those pigments that may appear in the new specification for gouache paints. The motion passed, and after some editorial adjustments to section 8 it will be forwarded for D01.57 ballot. Debate on assigning descriptive terms to the categories was tabled until a list of suggestions can be drawn up.

T. Vonderbrink showed data on tests attempting to find a replacement for the fluorescent lamps specified in D 4303. Results from a test using a HPUV apparatus with two lamps (Sho-cool white fluorescent and FS40) showed the best correlation with the presently-specified lamps and also good correlation with the Florida sun tests required by the standard test method. Further studies at different time lengths were suggested since longer exposures might produce different results; it was also suggested to test pigments that are sensitive to light/dark cycles. Testing will continue.

A caveat for section 5 (Significance and Use) of D 4303 suggested by J. Robbins and K. Scott was discussed. The test method allows for relatively large tolerances in placing pigments in lightfastness categories,

but those pigments falling near the borderline between two categories may still present problems; the caveat does not completely resolve the problem. R. Kinmonth moved to accept the caveat, and after a minor editorial change it will be forwarded for D01.57 ballot.

**D01.57.04—Specification for Artists' Paints**—A. Spizzo, Chairman, presented a reformulation of the second ground coat specified in section 7.3.2 of D 5098 after it was reported by B. Gavett that the original formula did not work properly. It was pointed out by B. Gavett that two applications of the formulation for the first ground coat would work just as well as having a second formulation. J.T. Luke moved to remove from the standard any references to a second ground coat, and the motion passed. After A. Spizzo adjusts the wording, the changes will be forwarded for D01.57 ballot.

B. Gavett submitted three pigments and their lightfastness test results for addition to Table I of D 5098. A motion to accept them was passed and they will be forwarded for D01.57 ballot.

B. Gavett discussed the results of lightfastness testing which showed that several pigments darken when exposed to sunlight under glass according to the specifications of D 4303. J.T. Luke also presented a list of pigments that are reported by their manufacturers to darken. J.T. Luke moved that in the future it will be required that new pigments being submitted for inclusion in Table I of D 4302 and D 5098 must be tested in masstone exposures as well as reductions with white as described in the specifications. The motion passed. Since this will result in revisions of D 4302, D 5098, and possibly D 4303, J.T. Luke agreed to research the changes, including gathering information from artist's paint manufacturers. Members of the subcommittee were also asked to submit suggestions as to how the changes and revisions might be made.

B. Gavett observed that the standard specifications require the use of a common name for pigments contained in paints on tube or jar labels, but that there are some common names that are not appropriate for the pigment—for instance, there are several different pigments that have the same common name. This is especially true as the more numerous new organic pigments are adopted. Other manufacturers who are also having this trouble will develop a list of the colorants in Table I of D 4302, D 5067, and D 5098 that the subcommittee can look at for changes.

B. Gavett showed samples and a list of pigments tested for bleeding by method B of D 279. Many of the pigments showed significant bleeding, but R. Gamblin suggested that this is a quality control problem on the part of the manufacturer of the pigment or the paint and that these results can be avoided. B. Gavett agreed to write a

paragraph for section 6 (Quality Assurance) of D 4302 and D 5098 which will reference D 279 and have a method for evaluating the amount of bleeding.

**D01.57.07—Physical Properties**—S. Sheehan, Chairman, reported that R. Gamblin, of Gamblin Artist Colors, has agreed to be the new Chairman of this task group.

**D01.57.10—Consumer Evaluation**—J.T. Luke, Chairman. The task group again voted to pursue the development of a standard practice for the visual evaluation of the lightfastness of art materials by the user. M. Gottsegen moved that the blue contrast mask proposed by J.T. Luke be adopted as the method of determining when the test is complete. Since the last attempt at balloting was withdrawn, previous negative votes were not debated; although J.T. Luke informed the subcommittee that they will be satisfied in a new draft of the practice. A new draft of the method for users will be prepared after some questions about the availability of adjunct materials is settled. A new draft of a version of the practice for art technologists will also be prepared, incorporating changes made to the user's version. Both new drafts will be forwarded for D01.57 ballot.

Representatives of the Pencil Makers Association (PMA) and R. Farfan from the Colored Pencil Society of America were present for discussion about how to develop a lightfastness test method and specification for colored pencils. Options include using D 4303, using the art technologist's practice once it is balloted, or developing a specific method for colored pencils. In all likelihood, D 4303 will be used with adjustments to the method of preparing samples for testing; a rating system will be proposed.

F. Murphy, of Dixon Ticonderoga, will report to the Pencil Makers Association about D01.57's discussions and get a decision on whether or not to go ahead. If the PMA does not want to continue, individual company representatives indicated that they may continue anyway.

**D01.57.11—Gouache Paints**—T. Takigawa, Chairman, reported that there was no new business, but a report on current testing is due in February 1992.

**D01.52.12—Determination of Toxicity**—W. Stopford, Chairman, presented a draft of a new standard test method for determining the bioequivalency of metals in art materials. With some changes and additions, the method should be ready for D01.57 balloting in February 1992.

The citrate buffer method for determining extractable lead and cadmium from glazed surfaces was discussed. An acetic acid-based method is used in 34 countries, and for D01.57 to develop an alternative would seem counterproductive. But after discussion, D01.57 concluded that to have

this method available would provide a helpful option for users, and voted to encourage C21.03 to continue development of such a test method.

A synthetic intestinal juice test method is being using by the Dry Color Manufacturers Association but there is not enough test data to use for the development of an ASTM method. W. Stopford is still looking for laboratories to do interlaboratory testing to confirm the method.

C. Jacobson, of the Consumer Product Safety Commission (CPSC), discussed the guidelines for the Labeling of Hazardous Art Materials Act. Comments from the October 1991 hearings are still being analyzed and a final rule may be issued as early as June 1992. The statutory definition of an art material by Congress is so broad that anything associated with the creation of art could be subject to the rule. The CPSC is leaning towards interpreting the definition as meaning traditional materials that are traditionally marketed to artists, and their main concern seems to be "chemically-compounded" materials. Such articles as brushes may be included, but objects like easels are not. C. Jacobson further reported it is possible that the CPSC will relent on some toxicity levels originally proposed, and that the definition of neurotoxicity will be redone to be a little more realistic.

In M. Rossol's absence, M. Gottsegen read written comments sent by her. M. Rossol's first comment was about products that contain carcinogens at levels which must be reported on Material Safety Data Sheets, but which may be sold to consumers with no warning labels and labeled "non-toxic." The example she used was of white glues which contain vinyl acetate concentrations of 0.6% but which have no warning labels, although a 0.1% concentration must be reported on MSDSs. W. Stopford commented that the Environmental Protection Agency study used a vinyl acetate that was contaminated with a carcinogen, and that there is no data that suggests that vinyl acetate itself is carcinogenic. On the subject of Material Safety Data Sheets themselves, several members of the subcommittee noted that they are sometimes inaccurate or not current. One member noted that MSDSs and right-to-know regulations are apples, and consumer product labels are oranges.

A second comment concerned products containing chemicals whose chronic hazards have not been assessed but which can be labeled "non-toxic." M. Rossol urged that CPSC change their guidelines so that labels on products whose ingredients have never been studied for chronic toxicity say so. M. Rossol also pointed out that the New Jersey Public Employees OSHA Right-to-Know law requires that products for which even 1% of the ingredients is unknown must be labeled "contents unknown" or "contents partially unknown." W. Stopford noted that the Art and Crafts Materials Institute will

not provide certification for products that have unknown ingredients in them. The subcommittee, however, agreed that it might be time to consider dropping the phrase "non-toxic" from labels since it can be so misleading. ACMI commented that the phrase "non-toxic" is demanded by many of the users of ACMI-certified products.

*D01.57*—The minutes of the May 1991 meeting were accepted by unanimous vote of the subcommittee. The subcommittee also unanimously confirmed all votes of the task groups.

M.D. Gottsegen announced that A. Spizzo will be giving up the job of Subcommittee Secretary after four years of service and thanked him for his excellent work. He will continue as Subcommittee Treasurer. B. Gavett has agreed to serve as Secretary.

The next meeting of the subcommittee will be held on May 29, 1992, at the Las Vegas Hilton, in conjunction with the international show of the National Art Materials Trade Association. M.D. Gottsegen will be unable to attend the meeting, and Vice Chairman T. Vonderbrink agreed to run the meeting.

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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, distributed the results of the first round-robin on copies of a "Proposed Test Method for Leveling Efficiency of Paint Brushes." L. Schaeffer, the manufacturer of the "brushout standards" used in the initial testing, informed the task group of the development of these standards and problems associated with their use. It was the decision of the task group to repeat the round-robin using two different substrates, namely primed upon board and CU "hiding" charts. The chairman will supply a paint with known poor leveling. Cooperators will use the three brushes currently under use. The chairman will report on the results of the round-robin at the next meeting.

Draft #4 of the "Proposed Practice for Physical Characterization of Paint Brushes" was reviewed in part by the group. It was the consensus of the task group to submit the method to D01.61 ballot before the next meeting.

*D01.61.02—Paint Rollers*—J.F. Price, Chairman, described the history of the roller method under study and test difficulties en-

countered. A summary included: (1) too few cooperators; (2) roller breakin procedure; (3) weighing procedure; and (4) too high expectations for the method.

After discussion, the following course of action was agreed upon: (1) the chairman will send a more modest version of the test method to all those present and to all past cooperators; (2) comments will be sent back to the task group chairman by March; and, (3) a new round-robin will then be initiated.

*D01.61.05—Bulk Density of Filaments and Bristle*—W.B. Bond, Chairman, led a review of the results of the second round-robin using level filaments supplied by Polymers (J. Moriarty).

This round-robin showed good reproducibility for the method. Agreement between laboratories was best at 50 psig. W. McLaggan and T. O'Brien agreed to complete their tests of the bundles and forward the results to the chairman. Ketema and Du Pont will supply hollow and solid polyester filaments (black and brown, respectively), for the next round-robin to be reported in Minneapolis. Wooster, T.S. Simms, and EZ Paint agreed to experiment with china bristle on the tester and report those results. Plans are to reserve testing of nylon ribbed and quadrilobal until the last half of 1992.

*D01.61.06—Buckling Resistance of Filaments*—W.B. Bond, Chairman, reported that only one order was placed for the Ketema testing device, and none was brought to the meeting. J. Price agreed to bring a tester to the Minneapolis meeting. A first draft of a method was prepared and the group agreed to hold revision of the draft until some experience with the tester was gained. T. O'Brien agreed to bring or send hollow filaments to the Minneapolis meeting for a demonstration of the method and to look into converting the deflection measurement from "% of length remaining at break" to "angle imposed on the ends of the filaments." T.S. Simms is waiting for two orders before building the Ketema device. Wooster (G. Harsch) will decide whether to place an order after the demonstration in Minneapolis.

*D01.61*—F. Burns reported that this subcommittee's recommendation for an expansion in title of D-1 to recognize "applications" had completed the approval process through balloting and Executive Committee actions.

The ballots for procedural revisions to Standards D 5068-90 (on brushes) and D 5069-90 (on rollers) passed subcommittee and D-1 balloting. The subcommittee votes were (6) affirmative, (0) negative, and (3) abstentions for each ballot. D-1 ballots for D 5068-90 were (113) affirmative, (0) negative, and (244) abstentions. D-1 ballots for D 5069-90 were (108) affirmative, (0) negative, and (249) abstentions. These items will now go to society ballot.

# January 1992 Subcommittee Reports of ASTM Committee G-3

The January meeting of G-3 on Durability on Nonmetallic Materials was held on January 20-23, 1992 at the Crown Sterling, Fort Lauderdale, FL. The present membership of G-3 is 86.

## Highlights

A new subcommittee, G03.08, was formed to address new developments in service life prediction of nonmetallic materials. Dr. Jonathan Martin, of National Institute of Standards and Technology, was named Chairman. The first meeting will be on June 30, 1992 in Minneapolis, MN.

A task group was formed in G03.03 to address the problems concerning the classification and specification of UV-fluorescent lamps, as used in G 53.

## Future Meetings

June 28-July 1, 1992: Marriott City Center, Minneapolis, MN.  
January 17-20, 1993: Crown Sterling, Fort Lauderdale, FL.  
June 27-30, 1993: Franklin Wyndham, Philadelphia, PA.  
January 23-26, 1994: Crown Sterling, Fort Lauderdale, FL.

### SUBCOMMITTEE G03.01 EDITORIAL AND DEFINITIONS

#### M.J. Crewdson, Chairman

There was one negative from R.A. Kinmonth on the latest G-3 ballot on E 41, "Standard Terminology Relating to Conditioning," which was withdrawn after discussion at the meeting. This revised standard will now proceed to society ballot. A companion standard on the handling and conditioning of nonmetallic materials will be presented at the next meeting.

The latest draft of the general G-3 terminology standard was distributed for discussion. Several changes were made and the revised draft will now proceed to ballot at the next opportunity.

### SUBCOMMITTEE G03.02 NATURAL ENVIRONMENTAL TESTING

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

Data has been presented at this and previous meetings concerning the transmittance of glass used in G 24, "Conducting Exposures to Daylight Filtered Through Glass." At this meeting, a G3.02 ballot which required laboratories to measure and report glass transmittance was discussed. Since a negative was not resolved, the discussion will continue at the next meeting.

Data was also presented concerning the interlab variability of G 90, "Performing Accelerated Outdoor Weathering of Non-metallic Materials Using Concentrated Natural Sunlight." The G3.02 ballot of G 90 four negatives. After discussion, it was decided to submit a corrected draft to a second G3.02 ballot.

A G3.02 ballot on a new standard for the transfer of calibrations from reference to

field radiometers received two negative votes. Because of the similarity of this standard to E 824, "Transfer of Calibration from Reference to Field Pyranometers," E 44 will be contacted to make them aware of this new standard. One of the negative votes was not resolved, and the discussion will continue at the next meeting.

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

#### R.M. Fischer, Chairman

A special meeting was held to discuss specifications for fluorescent lamps used in G 53, "Standard Practice for Operating Light and Water-Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Non-Metallic Materials." Information on the spectral power distributions of several commercial UV-B lamps was presented by P. Brennan, W. Ketola, and K. Scott. Currently, there are several UV-B lamps that have maximum irradiance at 313 nanometers and a 1% transmission cut-on near 280 nm. This is the only lamp information that is required in G 53's report form. The maximum irradiance can be considerably different for these lamps depending on the manufacturer. Test results can be significantly affected by these differences. J. Evans presented data that confirmed that confusion relative to lamp irradiance levels and radiometer calibration procedures is causing problems in other related standards (e.g., SAE J2020). W. Ketola and R. Fischer distributed a proposal for classifying fluorescent lamp types by their spectral power distributions. Several types may eventually be listed in G 53. A task group was formed (G03.03.06—*Fluorescent Lamp Specifications*—R. Fischer, Chairman) to address the problems concerning the classification/specification of fluorescent lamps.

G. Frohnsdorff presented background information on the development of E 632, "Standard Practice to Aid Prediction of the Service Life of Building Components and Materials." He proposed the broadening of the scope of E 632 to encompass a general methodology for the service life prediction of any material. Those in attendance were in general agreement that a G-3 subcommittee should be formed to address new developments in service life prediction.

G03.03 ballot results for the new "Standard Practice for Conducting Cyclic Immersion/Atmospheric Exposure Tests for Nonmetallic Materials" were (15) affirmative, (5) negative, and (16) abstentions. A negative by P. Brennan to discontinue work on this practice was withdrawn. Negatives by P. Brennan and J. Robbins, III that suggested splitting this practice into two standards concerning equipment with xenon arc sources and fluorescent lamp sources were found persuasive. Two new task groups were formed to facilitate this new direction: K. Trimber, Chairman, G03.03.05—*Fluorescent Cyclic Immersion*, and J. Robbins, III, Chairman, G03.03.06—*Xenon Cyclic Immersion*.

G03.03 ballot results for the addition of a Lamp Application Index to G 53-91, "Standard Practice for Operating Light and Water Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Non-Metallic Materials," were (23) affirmative, (2) negative, and (12) abstentions. Negatives by K. Scott and W. Ketola were found persuasive. The revised Lamp Application Index will be submitted for concurrent G-3/G03.03 ballot.

G03.03 ballot on improved water quality (specimen spray) for G 23-90, "Standard Practice for Operating Light Exposure Apparatus (Carbon Arc Type) With and Without Water for Exposure on Non-Metallic Materials," passed with no negatives. Editorial changes by W. Ketola were deemed technical in nature. This revision will be submitted for concurrent G-3/G03.03 ballot.

G03.03 ballot results for improved water quality (specimen spray) for G 26-90, "Standard Practice for Operating Light Exposure Apparatus (Xenon Arc Type) With and Without Water for Exposure of Non-Metallic Materials," were (24) affirmative, (1) negative, and (11) abstentions. The negative by K. Scott was found persuasive and the revised standard will also be submitted for concurrent G-3/G03.03 ballot.

Two proposed amendments to G-3 bylaws (Device Inclusion in G-3 Standards and Creation of New Standards) that originated in G03.03 were passed on to G03.90 Executive Committee. Both amendments were approved by G03.90 for G-3 ballot.

*G03.03.01—Round-Robin Studies*—R. Fischer, Chairman, reported on the progress with G 26, "Standard Practice for Operating Light Exposure Apparatus (Xenon Arc Type) With and Without Water for Exposure of Non-Metallic Materials." The round-robin protocol has been completed by four laboratories. Considerable variability (similar to levels observed in other Standard Practices—G 53 and G 23) was observed for the gloss loss rate of identical samples tested in the participating labs. Two additional labs will have completed the round-robin by the upcoming June meeting of G-3, and a final report will be issued at that time.

*G03.03.03—Education*—D. Grossman, Chairman. R. Fischer distributed a draft, "Guide for Weathering and Related Durability Testing." Recommendations for modifying this draft were requested by March 1, 1992. Suggestions will be incorporated and the guide will be sent out for G03.03 ballot.

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

**W.D. Ketola, Chairman**

In a discussion led by M. Crewdson, G03.01 Chairman, definitions for reference material, reference specimen, file specimen, masked area, weathering reference mate-

rial, and control were agreed upon. These will be part of a simultaneous G-3/G03.01 main committee ballot on the new G-3 terminology standard.

W. Ketola led a discussion of the procedures to be used by the G-3/VAMASTWA-12 round-robin on reference materials. The round-robin will be divided into three parts: (1) exposures of polysulfone films in fluorescent UV/condensation devices according to Practice G 53; (2) exposures of polystyrene chips in water cooled xenon arc devices according to SAE J1960; and (3) exposures of polyethylene film in filtered open flame carbon arc devices according to Practice G 23. Preliminary exposures using all three material/device combinations have been completed. Round-robin participants include researchers in the United States, Japan, Germany, New Zealand, Canada, and Great Britain. Initial measurements on specimens to be used in the round-robin are in progress and specimens are scheduled to be sent to participants in March.

N. Searle presented results from experiments to determine the activation spectrum for polysulfone exposed to UVB-313 fluorescent lamps and polystyrene exposed to a quartz/borosilicate filtered water cooled xenon arc. The activation spectra were obtained by determining property change in specimens exposed behind a series of sharp cut filters. For polysulfone, the fastest increase in 330 nm absorbance was produced by radiation in the 310-330 nm band. For polystyrene, radiation in the 310-345 nm band produced the fastest increase in yellowing. In exposures of polysulfone to monochromatic radiation conducted by P. Trubiroha, 310 nm radiation produced the most change in 330 nm absorbance.

### **SUBCOMMITTEE G03.07 LIAISON AND HARMONIZATION**

**R.A. Kinmonth, Chairman**

The organizational meeting of G03.07 was held on January 21, 1992 at the Crown Sterling Suites Hotel, Fort Lauderdale, FL.

A total of 13 members and 1 guest attended.

The need for the new subcommittee was outlined by R.A. Kinmonth. After extensive discussion, the following was adopted unanimously as the scope:

—To interact with other committees responsible for preparing standard test methods, practices, and specifications which make reference to the application of standards under the jurisdiction of G-3, to provide critical review of such standards when requested, to offer assistance to task groups and others in the preparation of standards requiring test methods and practices relevant to the durability of nonmetallic materials.

—To direct and coordinate formal liaison activities between G-3 and other ASTM Committees.

—To communicate among the subcommittees of G-3 such information as may become available that may be used to improve standards under its jurisdiction to better serve the needs of these testing the durability of non-metallic materials.

—To provide assistance in the harmonization of standards under the jurisdiction of G-3 with durability standards of other committees to assure the increased acceptance of such voluntary consensus standards in national and international trade.

After adoption of the scope, the following title was adopted unanimously with one abstention: Liaison and Harmonization.

It was agreed to begin operations with two standing working groups: .01—ASTM Committees; and .02—Other Standards Generating Organizations.

Future meetings will be scheduled during the meeting times for G-3.

# Society Meetings

## BALTIMORE ..... JAN.

### "Precipitated Calcium Carbonate"

George Green, of Pfizer, Inc., presented a talk on "PRECIPITATED CALCIUM CARBONATE FOR USE IN COATINGS."

Dr. Green said that the major use of precipitated calcium carbonate is in paper. He indicated that the paper industry is changing over to the nonacid process which uses calcium carbonate for sizing. Also, certain crystal structures of precipitated calcium carbonate extend the pulp to give a better yield of paper.

The speaker pointed out the various crystal shapes formed, including: pencil shaped, barrel shaped, football shaped, spherical shaped, cubic shaped, etc. Dr. Green stated that cubic crystals are used in food, and the "jacks" shaped crystal is used as a titanium dioxide extender in coatings.

It was explained that crystal should be used as a replacement for calcined clays. According to Dr. Green, it costs less and gives a bluer white as opposed to a yellowish white, and has a higher dry brightness, more pigment extension, better gloss retention, and lower effective resin demand.

Dr. Green said that a crystal is stable in a sandmill, but can be broken in a ball mill. It possesses a high speed dispersion of five to six grind. Also, on a pound for pound replacement of calcined clay, scrub resistance improves, but dry hiding is reduced, so the vehicle content has to be reduced to match dry hide.

According to the speaker, it is important to follow a proper order of addition. Dr. Green said not to add filler fines until the precipitated calcium carbonate and titanium dioxide are fully dispersed. Other formulation concerns when replacing calcined clay are: dry hide, sheen/burnishing, tints, stain resistance, scrub resistance, touchup, cost, and mudcracking. He stated that touchup and mudcracking are the two biggest problems.

In conclusion, the speaker stated that future research using precipitated calcium carbonates is focusing on semi-gloss formulations, synergistic effects with other extenders, and new crystal types like platy precipitated calcium carbonate for specific applications.

*Q. Is acid rain a problem? Does stearate treatment help?*

A. Yes, acid rain is a problem, but the vehicle helps protect it. This is the reason why automotive coatings don't use it. Also, blistering can form. In regards to stearate

treatment, it doesn't help enough to develop acid resistance.

*Q. What is the order of addition?*

A. It doesn't matter whether precipitated calcium carbonate or titanium dioxide is added first, however, no other pigment should be added before them.

JOHN KURNAS, *Secretary*

## CDIC ..... MAR.

### "Hazardous Materials Regulations"

A moment of silence was observed in memory of Society member E.E. "Woody" Pease, of Paint America Company, who died recently.

The Nominations Committee has nominated Bill M. Hollified, of Perry & Derrick Company, Inc., for Society Representative, and Alan L. Machek, of Dow Corning Corporation, for Treasurer.

Federation Honorary Member Lewis P. Larson presented Timothy A. Carsey, of Akzo Coatings, Inc., with the Lewis P. Larson Scholarship. Mr. Carsey stated that he intends to use the scholarship money to attend a coatings course at North Dakota State University in June.

The technical presentation was delivered by Jeffrey J. Wells, of Hazmat Environmental Group, Inc. Mr. Wells' talk focused on "HM-181: DOT PERFORMANCE PACKAGING (DOT'S NEW HAZARDOUS MATERIALS REGULATIONS)."

A description of the Department of Transportation (DOT) Final Rule 49 CFR Part 106 et al, Performance-Oriented Packaging Standards, published December 20, 1991, FR Volume 56, No. 245, 66124 was presented.

Mr. Wells stated that the new legislation is an amendment which makes revision to a final rule published by the Research and Special Programs Administration (RSPA) in the *Federal Register* under Dockets HM-181, HM-181A, HM-181B, HM-181C, HM-181D, and HM-204 (55FR 54402, December 21, 1990) with respect to hazard communication, classification, and packaging requirements. He indicated that changes were based on the United Nations (UN) Recommendations on the Transport of Dangerous Goods. Mr. Wells said that the U.S. would not be able to transport goods to foreign countries and foreign countries would not be able to transport goods to the U.S. if our packaging laws did not conform to UN standards.

According to the speaker, the new laws simplify and reduce the volume of Hazardous Materials Regulations (HMR), enhance safety through better classification and packaging, promote flexibility and technological innovation in packaging, reduce the need for exemptions from the HRM, and facilitate international commerce.

Mr. Wells said that hazardous materials, under the new system, are broken down into nine basic classes as opposed to the 22 different classes under the old system. The new classes are as follows: Class 1—explosives; Class 2—gases; Class 3—flammable liquids; Class 4—flammable solids; Class 5—oxidizers; Class 6—poisons; Class 7—radioactives; Class 8—corrosives; and Class 9—miscellaneous.

The speaker said the revised transition period requirements in §171.14 were published in a separate rulemaking issue on September 18, 1991 (56 FR 47158) to give people subject to the HRM more time to comply with the revised final rule.

According to Mr. Wells, the requirement for using new shipping descriptions and labels goes into effect on October 1, 1993. Placarding requirements go into effect October 1, 1994, because placards are expensive and it will take a long time to change over to the new system. The final part of the rule does not go into effect until October 1, 1996.

*Q. What do you use if the flash point of the material is 110°F, that is, nonregulated?*

A. With the old system, a flammable material was anything with a flash point less than 100°F. A combustible liquid was anything between 100° and 200°F. So, under the old system, your liquid was a combustible liquid and regulated under some circumstances. Under the new system, a flammable liquid is anything with a flash point less than 141°F unless you ship domestically.

The meeting's educational speaker was Lewis P. Larson. His presentation was "A TRIP TO ALASKA." Mr. Larson showed slides of his June 1991 trip to Alaska and British Columbia, Canada.

PAUL R. GUEVIN, JR., *Secretary*

## CLEVELAND ..... FEB.

### "Hansen Solubility Parameter"

A moment of silence was observed in memory of Joseph Shiffler who died recently.





**CDIC SPEAKER—Jeffrey J. Wells speaks at the Society March meeting on "New Hazardous Materials Regulations"**

Wesley L. Archer, of The Dow Chemical Company, spoke on "UTILIZING THE HANSEN SOLUBILITY PARAMETER THEORY IN THE REFORMULATION OF SOLVENT-BASED COATINGS."

The solubility parameter theory whereby each solvent and resin is characterized by a nonpolar (delta D), polar (delta P), and hydrogen bonding (delta H) solubility parameter was discussed. Mr. Archer said that the solubility parameter value for many materials can be obtained from literature. Also, the solubility parameters can be calculated from physical properties or estimated from chemical structure or by matching solubility performance.

It was explained that some solvents may have one type of behavior, while other solvents may have a balance of all three parameters. Mr. Archer used hexane and acetone as examples to these behaviors.

The speaker pointed out that the solubility parameters of a solvent blend can be determined by averaging the solubility parameters of each individual solvent on a volume fraction basis. He said that these types of calculations can be performed on a spreadsheet computer program.

In conclusion, Mr. Archer stated that reformulation of solvent-based coatings can be simplified by using Hansen solubility parameters. He said this is useful when trying to eliminate aromatic solvents from a formulation. Also, by determining the solubility parameters for individual solvents, the solubility of a resin in any blend of these solvents can be easily determined with a minimal amount of work.

*Q. What criteria do you use to determine resin solubility?*

A. We use a simple yes/no answer; it is or is it not soluble. After preliminary testing, the degree of solubility is considered.

*Q. Are these methods valid for water-reducible resins?*

A. Yes, there is some correlation between the hydrogen bonding solubility parameter and coupling ability. Currently, we are trying to relate this to water-miscibility, however, more work is required.

FREIDUN ANWARI, *Secretary*

## LOUISVILLE.....FEB.

### "Fluorochemical Additives"

A 25-Year Pin was presented to George Bless, of D.A. Campbell Company, Inc., in recognition of his service in the paint and coatings industry and membership in the Federation of Societies for Coatings Technology. Mr. Bless also is a member of the CDIC Society.

"FLUORO-CHEMICAL ADDITIVES FOR PAINT AND COATINGS" was the topic of a talk presented by Michael E. Killian, of the 3M Company. Mr. Killian is a member of the Northwestern Society.

An overview of the basic function of surfactants in organic coatings and the mechanisms that they affect in systems was discussed. It was noted that many of the surface defects in coating films occur from resin or pigment flow in the paint film after application. Mr. Killian said this movement is caused primarily from surface tension forces and its main opposition, viscosity. A brief explanation of the causes associated with these coatings defects was presented.

The speaker talked about the effect of surfactants used at low levels to control surface tension values in industrial applications.

In conclusion, Mr. Killian gave some basic hints for the practical use of the types of products discussed when certain surface defects are experienced by the formulator.

MIKE MOILANEN, *Secretary*

## NEW ENGLAND.....FEB.

### "Higher Solids Coatings"

Richard Shain, of King Industries, Inc., spoke on "FORMULATING HIGHER SOLIDS COATINGS WITH NOVEL, LOW MOLECULAR WEIGHT POLYESTERS AND POLYURETHANES."

The talk began with a discussion of the requirements for high-solids resins, including: low molecular weight resins, narrow molecular weight distributions, solubility and compatibility, and crosslinking, featuring faster cure and acid catalysts.

Mr. Shain reviewed the polyester diol factors: low molecular weight, narrow molecular weight distribution, saturated cycloaliphatics, low acid member, and low water content. Also, the benefits, wide com-

patibility with resins and rapid solvent compatibility, were explained.

The speaker said two-component, polyurethane clear coats possess the following properties: low VOC, hard film, good solvent resistance, and hardness. In comparison with other resins, polyurethanes exhibit good modification and flexibility. Also, a polyurethane's gloss improves with time.

Mr. Shain explained that polyurethane diols feature low molecular weight, narrow molecular weight distribution, an aliphatic urethane composition, high viscosity, and contain no isocyanates.

Their benefits include the lower viscosity, QUV exposure (good gloss retention), water solubility without amines, polar organic solubility, and improved checking.

JOANNE MONIQUE, *Secretary*

## NEW YORK.....JAN.

### "Coating Problems and Solutions"

Technical Committee Chairman Larry Waelde, of Troy Chemical Corporation, acknowledged that the group is on schedule to produce two technical papers this year.

New York Society member Thomas A. Daquila, of Daniel Products Company, presented "PIGMENTED COATING PROBLEMS AND SOLUTIONS ASSOCIATED WITH PARTICLE SIZE."

Mr. Daquila said particle size has a great influence on the properties of a coating. He emphasized that problems which are thought to be due to poor resin or pigment selection can be frequently corrected or improved greatly by altering or reducing the particle size of the pigment(s) used.

The complex science of paint making with the various grinding media, horizontal mills, and changing raw materials was highlighted. According to the speaker, there is a tendency today to treat any problem chemically or with an additive addition. He stated that many of the problems can be eliminated or reduced.

According to Mr. Daquila, particle size affects both the appearance and performance of a coating. Its effect on appearance is seen in terms of hue, tinting strength, hiding power, and gloss. It affects performance in terms of rheology, stability, and durability.

The speaker presented illustrations of these phenomena.

*Q. Does better dispersion always give better tinting strength?*

A. Yes, in most cases. However, in some pigments, the improvement is small. Yellow iron oxide for instance shows very little improvement in tinting strength when the grind is improved from a 3.0 to a 6.0 Hegman. Thus, the difference in tinting strength with improved dispersion varies from pigment to pigment.

*Q. Can you explain why additional grinding (sic) of Toluidine Red would cause a haze to form in the film even though the fineness of grind continued to improve?*

A. The equipment may be inadequate, or the excessive dispersion time could cause bleeding. Fineness of grind only tells about the largest particles present.

ARMAND J. STOLTE, *Secretary*

## NORTHWESTERN ..... FEB.

### "High Solids Alkyds"

The initial segment of the program was presented by Clive C. Coady, of Amoco Chemical Company. The Chicago Society member discussed "EXPERIMENTAL HIGH SOLIDS ALKYDS."

The speaker discussed the following three formulations: an acrylated alkyd for an industrial maintenance coating, a high solids bake alkyd based on soya fatty acid with Cymel® 303 and catalyst, and a high solids architectural alkyd. Mr. Coady explained the properties of the formulations.

In conclusion, it was noted that the choice of an aromatic diacid, such as isophthalic acid, is important to improve dry time, after tack, and the nonyellowing characteristics of alkyd coatings. Mr. Coady stated that another important consideration in the performance of a high solids alkyd coating is the selection of a drier package.

"Prime Time Live," a videotape on lead paint abatement, served as the second part of the meeting's program.

The film contained information on studies being conducted regarding the level of lead in soil along interstate highways in California. According to one researcher, it was discovered that the highest levels of lead were found in soil near major roads. It was discovered that the pattern of soil contamination followed the pattern of traffic levels in any given area.

Also addressed in the videotape were the following issues: lead emissions from automobiles, leaded gas, and the Environmental Protection Agency's Superfund.

The third and final segment program was presented by Martha O'Hara and Lynda Stanton who focused on "INTERIOR DESIGN."

Included in the talk were novel decorating ideas, and tips for decorating with the right color, fabric, and textures.

SARAH OEBSER, *Secretary*

## NORTHWESTERN ..... MAR.

### Annual Symposium

The Society's 21st Annual Symposium was conducted on March 3, in Bloomington,

MN. The theme of the program explored "Clean Air and Coatings—Focus for the 90s."

The Symposium's first speaker was Society member William J. Stewart, of Valspar Corporation. His talk was on "A GUIDE TO THE NEW FEDERAL REGULATIONS, OR HOW TO KEEP YOUR CEO OUT OF JAIL."

Discussed were the Clean Water Act, the Resource Conservation and Recovery Act (RCRA), the Toxicity Characteristic Leaching Procedure (TCLP), and National Ambient Air Quality Standards (NAAQS).

Mr. Stewart focused his presentation on the history of the Clean Air Act from 1970 to 1990. The following areas of the Clean Air Act were addressed: Title I—nonattainment provisions; Title III—air toxics; Title V—permitting; and Title VII—enforcement.

In conclusion, the speaker discussed some ideas which may help the paint and coatings industry in its ongoing struggle with federal regulations. Mr. Stewart made the following recommendations: get to know local state legislators and let them know how regulations affect the industry; be active in the National Paint and Coatings Association; stay up to date on regulations and be proactive about compliance, avoid waiting until regulation deadlines; do not violate regulations; and attempt to change existing regulations (if you do not agree with them).

*Q. Are stormwater permits required if you are storing empty drums outside?*

A. If the drums are clean, no permit is required.

*Q. Regarding the disposal of industrial waste, is there a different definition for industrial landfill versus public landfill?*

A. If your waste is classified as hazardous, it cannot be landfilled.

The next speaker was Minnesota State Senator Steve Novac who discussed "THE CLEAN AIR ACT AND ITS EFFECT ON MINNESOTA, AND OTHER ENVIRONMENTAL LEGISLATION."

The Senator began his presentation by stating that prior to 1991, there had not been any state legislation specifically regulating the coatings industry. However, in 1991,

two bills were passed in Minnesota affecting the industry: a bill prohibiting the addition of heavy metals to coatings, and a bill placing a limitation on certain toxics in packaging components. Currently, a new bill, which deals with lead contamination, is being considered. This bill would modify requirements for lead education, assessment, screening, and abatement. According to Senator Novac, the bill expands blood testing requirements and places time limits on addressing identified lead exposures; establishes a program to license lead abatement contractors; transfers authority for lead abatement programs from the Pollution Control Agency to the Department of Health; and it places a fee on petroleum tanks and a wholesale tax on paint to support the lead abatement program.

Senator Novac indicated that the bill will have to be passed in both the Minnesota House and Senate Environmental Committees in order to move on to the entire legislature. Failure of the bill to pass in the Environmental Committees would result in it going through debate in the Tax Committee, since new taxes are proposed in the bill.

The speaker stated that the legislature currently is debating Select Committee on Packaging and the Environment (SCOPE) legislation. The SCOPE bill would reduce the amount of packaging that can be thrown away. The House committee has passed the bill, which will be heard in the current session in the Senate committee.

*Q. Can you talk about amendments to the Clean Air Act and their impact on Minnesota?*

A. Since we are in an area of attainment, the impact may not be all that great. States are allowed to strengthen the federal law in their own states.

*Q. What control does the legislature have over the Minnesota Pollution Control Agency?*

A. Substantial. Funding allocations for all state agencies are made in the legislature.

Society member Joe Ziegewald, Consultant, spoke on the "BEST OPTIONS IN AND PRACTICAL APPLICATION OF COMPLIANCE COATINGS."

Mr. Ziegewald discussed the relation-



PAST-PRESIDENTS—Eleven former Presidents of the Louisville Society attended the January meeting, including (from left): Nick Lanning, John C. Ballard, Joseph A. Bauer, Larry F. Pitchford, James A. Hoec, Raymond L. Mudd, Herb Wilson, Paul M. Nilles, J. Kirk Menefee, Fred E. Newhouse, and Louis F. Holzknecht

## Constituent Society Meetings and Secretaries

**BALTIMORE** (Third Thursday—Willow Grove Restaurant, Linthicum, MD). JOHN KURNAS, Mineral Pigments Corp., 12116 Conway Rd., Beltsville, MD 20705.

**BIRMINGHAM** (First Thursday—Strathallan Hotel, Birmingham, England), D.C. MORRIS, PPG Industries (UK) Ltd., P.O. Box 359, Birmingham, B16 0AD, England.

**CDIC** (Second Monday—Location alternates between Columbus, Cincinnati, Dayton, and Indianapolis). PAUL R. GUEVIN, JR., P.R. Guevin Associates, P.O. Box 811, Westerville, OH 43081-0811.

**CHICAGO** (First Monday—Sharko's Restaurant, Villa Park, IL). GREGORY E. McWRIGHT, ANGUS Chemical Co., 2911 Sanders, Northbrook, IL 60062.

**CLEVELAND** (Third Tuesday—Brown Derby, Independence, OH in Sept., Oct., Nov., Dec., Feb., March; Jan. and Apr. meetings, Landerhaven, Mayfield Heights, OH). FREIDUN ANWARI, Coatings Research Group, Inc., 2340 Hamilton Ave., Cleveland, OH 44114.

**DALLAS** (Thursday following second Wednesday—The Harvey Hotel, Dallas, TX). ROBERT GIBNEY, Kerr-McGee Chemical Corp., P.O. Box 565026, Dallas, TX 75212.

**DETROIT** (Second Tuesday—meeting sites vary). RON ANDRUS, BASF Corp., 5935 Milford Ave., Detroit, MI 48210.

**GOLDEN GATE** (Monday before third Wednesday—alternates between Francesco's in Oakland, CA, and Holiday Inn in S. San Francisco). DONALD NOLTE, John K. Bice Co., 280 Missouri St., San Francisco, CA 94107.

**HOUSTON** (Second Wednesday—Hobby Airport Hilton, Houston, TX). RICHARD W. RYAN, Exxon Chemical Co., P.O. Box 5200, Baytown, TX 77520.

**KANSAS CITY** (Second Thursday—Cascone's Restaurant, Kansas City, MO). YVONNE D'ARCY, Cook Paint & Varnish Co., P.O. Box 419389, Kansas City, MO 64141.

**LOS ANGELES** (Second Wednesday—Steven's Steakhouse, Commerce, CA). PHILIP C. BREMENSTUHL, Ashland Chemical, Inc., 999 Town & Country Rd., Orange, CA 92668.

**LOUISVILLE** (Third Wednesday—Executive West Motor Hotel, Louisville, KY). MIKE R. MOILANEN, United Catalysts, Inc., P.O. Box 32370, Louisville, KY 40232.

**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 Biftheque, Ville St. Laurent, Quebec). JACQUES BROUILLETTE, BAPCO, 1470 Nobel St., Boucherville, Que., J4B 5H3, Canada.

**NEW ENGLAND** (Third Thursday—Sheraton Lexington Hotel, Lexington, MA, alternate meetings twice a year in Sturbridge, MA and Providence, RI). JOANNE E. MONIQUE, Ashland Chemical Inc., 400 Main St., Tewksbury, MA 01876.

**NEW YORK** (Second Tuesday—Landmark II, East Rutherford, NJ). ARMAND J. STOLTE, RHEOX Inc., P.O. Box 70, Hightstown, NJ 08520.

**NORTHWESTERN** (First Tuesday after first Monday—Jax Cafe, Minneapolis, MN). SARAH OEBSER, H.B. Fuller Co., 3530 Lexington Ave., N., St. Paul, MN 55126.

**PACIFIC NORTHWEST** (PORTLAND SECTION—Third Tuesday; PUGET SOUND SECTION—Third Wednesday; VANCOUVER SECTION—Third Thursday). FLORA WONG, Saguro Ltd., 106-150 E. Fifth St., N. Vancouver, B.C., V7N 1L5, Canada.

**PHILADELPHIA** (Second Thursday—Williamson's Restaurant, GSB Bldg., Bala Cynwyd, PA). BRIAN O'CONNOR, McWhorter Resins, Inc., 7600 State Rd., Philadelphia, PA 19136.

**PIEDMONT** (Third Wednesday—Ramada Inn Airport, Greensboro, NC). DENNIS GILLESPIE, Lomas Minerals and Chemicals, P.O. Box 605, Indian Trails, NC 28079.

**PITTSBURGH** (Second Monday—Montemurro's Restaurant, Sharpsburg, PA). TIMOTHY ZEFFIRO, J.M. Gillen Co., P.O. Box 588, Bridgeville, PA 15017.

**ROCKY MOUNTAIN** (Monday following first Wednesday—Zangs Brewery, Denver, CO). LOUIS HARTNELL, Mountain-West Resources, Inc., P.O. Box 16784, Denver, CO 80216.

**ST. LOUIS** (Third Tuesday—Salad Bowl Restaurant, St. Louis, MO). MICHAEL SCHNURMAN, Kop-Coat, Inc., 328 Henley Ind. Ct., St. Louis, MO 63144-1599.

**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). MARY G. FINNIGAN, McCullough & Benton, Inc., 2900 G Carolina Center, Charlotte, NC 28208.

**TORONTO** (Second Monday—Cambridge Motor Hotel, Toronto). DAVID JACK, Technical Coatings Co Ltd., 1164 Walkers Ln., Burlington, Ont., L7M 1V2, Canada.

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

ship between technical and marketing personnel in the paint and coatings industry. He encouraged users to examine relationships with their vendors. In addition, the speaker recommended using performance criteria when writing up paint specifications. Also, turn key systems were proposed.

Several types of coating systems were examined as application options, including autodeposition, electrodeposition, powder coatings, and high-solids technology.

The Symposium's final speaker was Daniel N. King, of Exxon Chemical Company. The Houston Society member gave a presentation on "COMPLYING WITH EMISSIONS REGULATIONS—A SUPPLIER'S VIEW."

Mr. King spoke about the three initiatives that regulate and monitor the sources of emissions: the Environmental Protection Agency's voluntary emissions reduction program, known as the 33/50 Project; Title I of the Clean Air Act, which addresses the reduction of VOC; and the Hazardous Air Pollutants (HAPS) emissions list.

According to the speaker, the 33/50 Project calls for a collective 50% reduction in the emissions of 17 chemicals by the end of 1995. The chemicals were placed on the list on a release volume, emission reduction potential, and toxicity basis. Compliance with the program's requirements are voluntary.

However, he stated that Title I of the Clean Air Act will have the greatest impact in nonattainment areas. VOC will be regulated by emissions source categories. According to Mr. King, the impact of Title I has been to spur the growth of alternative technologies. Also, it has increased the use of engineering controls.

The speaker said that that the HAPS list contains 190 chemicals from the SARA 313 and is based on the release volume, emissions reduction potential, or toxicity. He explained that EPA will establish control requirements and deadlines by prioritizing source categories. Also, there are no regional impacts for ozone nonattainment.

SARAH E. OEBSER, *Secretary*

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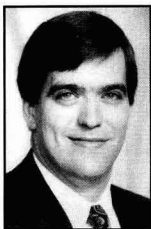
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# People

**Mark S. Koskiniemi** has been named Product Development Manager/Coatings and Plastics for Buckman Laboratories International, Inc., Memphis, TN. In this capacity, Mr. Koskiniemi will be responsible for the worldwide development of products for the coatings and plastics industries. He previously served as Laboratory Manager of the Coatings and Plastics Technical Service Laboratory in Memphis.



M.S. Koskiniemi



C.E. Carncross

Also, **Charles E. Carncross** has been appointed Manager of Buckman Laboratories' Coatings and Plastics Division. Under this title, Mr. Carncross will be responsible for the domestic sales and marketing of the company's products. He is based in Chicago, IL, and is a member of the Chicago Society.

In addition, **Michael McLaurin** was named Group Leader/Coatings and Plastics. Mr. McLaurin will spearhead the marketing efforts to international coatings and plastics companies. He has been with Buckman Labs since 1984.



M. McLaurin

**Richard Q. La Fond**, Vice President of the Coatings and Resins Group of PPG Industries, Inc., Pittsburgh, PA, has resigned from the company to pursue other interests. Group Vice President **Richard Rompala** stated that the architectural finishes unit would report directly to him pending the appointment of Mr. La Fond's successor.

In other PPG news, the Coatings and Resins Group has named **Carl A. Seneker** Senior Development Chemist and Project Coordinator for industrial coatings product development.

**Bohlin Instruments, Inc.**, Cranbury, NJ, has announced the addition of **Randy Byrne** to its staff as Sales and Marketing Manager for its North American headquarters. Mr. Byrne brings to this position many years of experience in the rheological field, and has worked in a variety of technical sales/sales management and marketing positions.

**Unocal Corporation**, Schaumburg, IL, has established a new business unit responsible for marketing bulk quantities of hydrocarbon aromatics and aliphatics. The following people have been appointed to head the unit: **William F. Murphy**—Vice President; **John C. Maher**—General Manager/Marketing and Sales; **Jim W. Crase**—Manager/Supply; **J.R. Phillips**—Manager/Transportation; **Robert L. Hinrichs**—Technical Manager; **Lisa Mosgo**—Manager/Administration; and **Jim Bollinger**—Manager/National Accounts, Hydrocarbon.

Mr. Hinrichs is a member of the Chicago Society.

**Frank Coccia** has been promoted to the position of National Sales Manager for the Electron Optics Division of Carl Zeiss, Inc., Thornwood, NY. Mr. Coccia's first position with the company was as a Service Engineer, followed by Product Specialist, District Sales Manager, and most recently, Regional Sales Manager/Eastern U.S. and Canada. He has been with the company for 18 years.

**Air Products and Chemicals, Inc.**, Allentown, PA, has announced several changes within its senior management personnel. The changes, which became effective on May 1, coincide with **H.A. Wagner** becoming Air Products' Chairman and Chief Executive Officer. He succeeds **Dexter F. Baker** who will continue as a member of the company's Board of Directors and Chairman of the Board's Executive Committee. Other appointments include:

**J. Robert Lovette** has been named Executive Vice President/Gases and Equipment. For the past four years, Dr. Lovette had served as Group Vice President/Chemicals.

The position of Group Vice President/Chemicals has been filled by **Robert E. Gadomski**. Mr. Gadomski, a 22-year veteran of Air Products, has also served in

**Don Walker** has accepted the position of Eastern Division Sales Manager for **Datacolor International**, Lawrenceville, NJ. His territory includes the Southeast and Mid-Atlantic regions, New England states, and Canada. Mr. Walker previously served as Regional Sales Manager and has been with the company for more than 10 years.



D. Walker

**Exxon Chemical Company**, Houston, TX, has announced that **Tim J. Hearn** has joined the company as Worldwide Vice President/Intermediates Department, Performance Products Group. Mr. Hearn's most recent position was Vice President, Retail/Commercial of Imperial Oil in Canada.

**Hüls America Inc.**, Piscataway, NJ, has named **Thomas E. Maggio** Executive Vice President of the company's Colorants and Additives Division. In this capacity, Dr. Maggio will direct the division's business operations, which include six manufacturing facilities in North America. He formerly served as Executive Vice President of the firm's Chemicals Division.

several manufacturing and general management positions in chemicals.

**John P. Jones** has been appointed Group Vice President/Process Systems. A 20-year veteran of the company, Mr. Jones previously served as Vice President and General Manager/Environmental and Energy Systems.

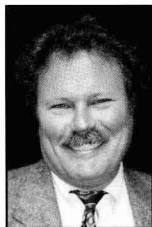
**Wayne A. Hinman** has accepted the position of Vice President and General Manager/Environmental and Energy Systems, succeeding Mr. Jones. Mr. Hinman has been with Air Products for 18 years, most recently serving as Vice President/Corporate Planning.

The position of Vice President/Corporate Planning has been accepted by **Thomas R. Hall**. Prior to this appointment, Mr. Hall held various controllership and general management positions in the Chemicals Group.

**Ray Millard** has accepted the promotion to Senior Vice President for Development/Operations of Avacor, Inc., Vonore, TN. Mr. Millard, previously Vice President of Corporate Development, has over 30 years of experience in the plastics compounding industries in the U.S. and Europe. He was formerly employed with National Chemical Industries in the Middle East and what is now Norsk Hydro in the United Kingdom.

**Mark E. Ulrich** has joined the Macbeth Division of Kollmorgen Instruments Corporation, Newburgh, NY, as Director of Marketing for its Color Data Products Group. In this position, Mr. Ulrich will direct marketing efforts for the company's spectrophotometers, color quality control and color matching/formulation systems, and production line color control systems. He joins Macbeth from Fujitsu Imaging Systems of America where he was Manager of new product development.

**Bob Hicks** has joined the staff of S.P. Morell & Company, Tarrytown, NY, as a company representative to the New England region. Prior to joining the firm, Mr. Hicks' previous experience included sales of latex polymers for Rohm and Haas Company, and distribution sales for Fish Chemical.



**B. Hicks**

**Donald B. Kurfees**, former owner and current Vice President and Chief Executive Officer of Kurfees Coatings, Inc., Louisville, KY, has announced his retirement from the company. Mr. Kurfees joined the company in June 1955, succeeding his father, **J. Frank Kurfees** as President and CEO. He later inherited the firm, which was founded by **J.F. Kurfees, Sr.**, his grandfather. Mr. Kurfees is a Past-President of the National Paint and Coatings Association.

The Valspar Corporation, Minneapolis, MN, has appointed **Karl Sheffer** Vice President/Manufacturing Division. Mr. Sheffer formerly served as Manager of Manufacturing Service/Domestic for Amoco Chemical Company, where he also worked for the firm's International Manufacturing Services Division.

## Obituary

**Ralph Kardashian**, Commercial Development Manager, ICI Resins US, Wilmington, MA, died March 19, 1992. He was 51 years old.

Mr. Kardashian had been with ICI for two-and-one-half years. Prior to that, he was

with Energy Sciences, Inc. for 10 years, most recently as Marketing Manager.

He was a member of the American Chemical Society, Radtech International, TAPPI, and SAMPE.

## Letter to the Editor

### The Un-Noticed Passing of a Giant

Adolph C. Elm, 89, formerly of Palmerton, PA, died on July 4, 1990. Born in Broeckkabel, Germany in 1900, he was the son of the late Heinrich and Emily Elm. He was the husband of Mertle M. (Derr) Elm, who died in 1983.

"Mike," as he was known to his friends, died un-noticed by the paint and coatings industry he served for over 46 years. He attended school in Germany and received his Ph.D. in Organic Chemistry from the University of Marburg.

Dr. Elm came to the United States in January 1924. He was first employed by E.I. du Pont de Nemours & Company, McCloskey Varnish Company, and American Asphalt Paint Company. In 1929, he started a career with the New Jersey Zinc Company (now Zinc Corporation of America) which spanned 41 years. At the time of his retirement he was Supervisor of Research.

During his 41 years spent with New Jersey Zinc, Dr. Elm distinguished himself in many ways. He was a leading authority in the field of zinc dust paints, and had done considerable research with drying oils, paints, and pigments. The following is a list of his accomplishments:

- 1) Published over 50 papers related to the paint and coatings industry.
- 2) Owned many patents related to pigments.
- 3) Presented the Federation of Societies for Coatings Technology's Joseph J. Mattiello Memorial Lecture in 1953.
- 4) Was the recipient of the Philadelphia Society for Coatings Technology's Liberty Bell Award in 1965.
- 5) Received the Federation's George Baugh Heckel Award in 1968.
- 6) Was an Honorary Member of the Federation.

Included in his many memberships are those with the Philadelphia Society for Coatings Technology, American Chemical Society, National Association of Corrosion Engineers, Engineers Club of the Lehigh Valley, and the National Paint, Varnish, and Lacquer Association.

Dr. Elm is survived by a son, Gerhard W., of Pittsburgh, PA and four grandchildren.

—Submitted by  
Wayne Kraus, Aqualon Company  
Philadelphia Society Member



### International Colour Materials Conference Slated for November 4-6, in Osaka, Japan

The 1992 International Conference on Colour Materials, sponsored by the Japan Society of Colour Material, is scheduled for November 4-6, at the Osaka Sun Palace in Expo Park, in Osaka, Japan.

The theme of the conference is "Recent Advances in Colour Material Science and Technology." Sessions on materials (pigments, fillers, dyes, resins, oils, etc.), paints and coatings (water-based paints, powder coatings, etc.), printing inks, and related products will be covered during the conference.

At this time, the scheduled presentations include three Plenary Lectures, the Motegi Memorial Lecture, and 14 technical presentations.

The three Plenary Lectures include:

"Environmentally Friendly Coatings: Past, Present, Future"—A.T. Misev, of DSM Resins BV, The Netherlands;

"The Use of Diblock Copolymer as Pigment Dispersant in Nonaqueous Media"—D.T. Wu, of DuPont Company; and

"Organic-Inorganic Polymer Hybrids"—T. Saegusa, of Kansai Research Institute.

The Motegi Memorial Lecture will be presented by Professor Y. Aboshi, of Kansai University, on "The Colour-Tones of Mural Paintings in Burial Mounds."

The following technical papers are scheduled to be presented in English:

"Formation and Characterization of Carbonaceous Beads from the Sol-Gel Method"—K. Esumi, of Science University of Tokyo;

"Measurement of Viscoelasticity of Coatings During Film Formation in Relation to Its Workability"—T. Fujitani, of Kansai Paint Company, Ltd.;

"Formation of Uniform Lanthanum Basic Carbonate Particles in the Presence of Ethylenediamine"—S. Hamada, Y. Kudo, and H. Matsuda, of Science University of Tokyo;

"Dynamics of Ink Transfer"—T. Hayashi and T. Amari, of Chiba University;

"Plasma Coating onto Pigments"—T. Ihara, S. Ito, and A. Kiboku, of Kinki University;

"Plasma Surface Modification of Pigments for Better Dispersibility"—Y. Iriyama and S. Ikeda, of Nippon Paint Company Ltd.;

"Interaction between Resin and Pigment in Concentrated Resin Solution"—M. Ishihara and T. Kanai, of Dai Nippon Paint Company, Ltd.;

"A Novel Crosslinking System Applicable to Lower Temperature Baking and Ambient Temperature Curing Surface Coatings"—O. Isozaki, of Kansai Paint;

"Nonlinear Viscoelastic Properties of Suspensions"—H. Kanai and T. Amari, of Chiba University;

"Pigment Dispersion in Aqueous Paint Systems"—T. Kobayashi and S. Ikeda, of Nippon Paint;

"Ultra Hydrophobic Surface Coating—Its Characteristics and Applications"—H. Kogure, of Kansai Paint;

"Plasma Treatment of Polymer Powders"—K. Minamihashi, S. Ito, T. Ihara, and J. Shiokawa, of Kinki University;

"Degradation of Organic Coatings under Weathering Test"—M. Nagai, O. Ogawa, and H. Tanabe, of Dai Nippon Paint; and

"New Technology in Powder Coatings"—K. Tsutsui, T. Ueno, A. Uenaka, and Y. Toyoda, of Nippon Paint.

For more details on the '92 International Conference on Colour Materials, including registration information, contact: S. Tochiara, c/o Japan Society of Colour Material, Kitamura Bldg. 5F, 9-12, 2-chome, Iwamoto-cho, Chiyoda-ku, Tokyo 101, Japan.

### Louisville Society for Coatings Technology Hosts Symposium on Water Base Coatings

The Louisville Society for Coatings Technology sponsored a "Water Base Coatings Symposium" on April 15, at the Executive West Hotel, in Louisville, KY.

Following the Introduction by Symposium Chairman Lloyd Browning, of Kelley Technical Coatings, Inc., and the Welcoming Address by Louisville Society President Kris Grauer, of Ashland Chemical Company, a total of nine technical presentations, focusing on water-based coatings, were presented. The topics and speakers were as follows:

"Acrylic-Styrene Latex in Anticorrosive Paints"—Alan Smith, of BASF Corporation;

"Solvent Selection for Waterborne Industrial Coatings"—Ronald K. Litton, of Eastman Chemical Products;

"New Generation High Gloss Corrosion Resistant Emulsions"—Mike Wildman, of McWhorter Corporation;

"Elastomeric Wall Coatings"—John R. Craddock, of Rohm and Haas Company;

"Stabilization of Anticorrosive Pigments in Water Base Paint through Use of Amines"—Albert Gesenhues, of Goodyear Corporation;

"Enhancing Freeze-Thaw Stability in Latex Paints through Proper Choice of Coalescent, Amine, and Antifreeze"—Albert Gesenhues;

"New Waterborne Systems for the Water-Repellent Market"—Lori A. Stark and

Dipak B. Narula, of Dow Corning Corporation;

"Water Base Epoxies"—Gary Green, of Pacific Coast Epoxy; and

"Waterborne Polyurethanes—Technology and Applications"—Bernard Taub, of Reichhold Chemicals, Inc.

For more information on the "Water Base Coatings Symposium," contact Lloyd Browning, Symposium Chairman, Kelley Technical Coatings Inc., 1445 S. 15th St., Louisville, KY 40210.

### University of Minnesota to Offer Course on Coating Process

The Center for Interfacial Engineering and the Department of Chemical Engineering and Materials Science of the University of Minnesota, Minneapolis, MN, are offering a course in "Coating Process Fundamentals" this spring.

The class also is being offered as a short course on the following dates: May 18-20, in Antwerp, Belgium; and June 24-26, in Minneapolis.

For more information, contact: J.D. O'Brien, Dept. of Chemical Engineering and Materials Science, University of Minnesota, 421 Washington Ave., S.E., Minneapolis, MN 55455; Telephone: (612) 625-9075.

# OCCA SURCON/SURFEX '92 Attracts 2,500 To Harrogate Joint Events in March

For the first time, the Oil & Colour Chemists' Association (OCCA) combined its alternate biennial conference (SURCON) and exposition (SURFEX). Held March 16-18 in Harrogate, England, the combined events were attended by 2,500 industry personnel from England, Europe, North America, and Japan.

Beginning on March 16, the two-day SURCON, cosponsored by the Paint Research Association, featured industry presentations on the theme, "Creative Advances in Coatings and Inks Technology: The Impact of the Environment." Speakers and topics included were:

"An Overview of Industrial Waterborne Coatings"—Dr. John Nicholson, of LGC;

"Whither Nonaqueous Coatings?"—Steven Rees, of Cray Valley Ltd.;

"The Coloration of Advanced Coatings"—Adrian Abel, of Hoechst UK;

"Dispersions and Resins for Waterborne Inks and Coatings"—"Chip Dispersion"—Stanley Jones, of Foscolor; and "Converting with Water"—Paul Bass, of IGP;

"Regulations and Practice in Waste Water Disposal"—Bill Ferguson, of E&A West;

"Intrinsically Intumescent Polymers: Environmental Safety Without Environmental Hazards"—David Aslin, of Prometheus Developments;

"Meeting the Compliance Agenda: Are You Ready?"—Dr. Helma Jotischky, of PRA;

"Barriers to Using New Technology—Radiation Curing"—Dr. Richard Holman, of PRA;

"Zinc Borate in Waterborne Coatings"—Dr. Kieran Quill, of Borax Consolidated;

"Harnessing Product Innovation to Environmental Care in Waterborne Paints"—Dr. Jeremy Lane, of Vinamul;

"Additives and Innovations"—Lionel Morpeth, of BYK-Chemie;

"How Green is (My) Ink"—Gerry Burdall, of Usher Walker; and

"Advantages of Catalytic IR Curing for Effective Emission Control"—John Sullivan, of Teknikat, and Grahame Kitchin, of Juna Products.

The exposition, SURFEX '92, was held from March 17-18, and featured the exhibits of 116 supplier companies at the Harrogate Exhibition Centre.

Representatives of several international coatings technical organizations were also in attendance and delivered welcoming remarks on behalf of their associations. Speaking were President Fred J. Morpeth, of OCCA, President Michael Symes, of SLF (Scandinavia), President Max Raaff, of FATIPEC (Europe), and Executive Vice President Robert F. Ziegler, of FSCT.

Highlighting the meeting was the signing of a cooperative agreement between OCCA and SLF to cosponsor a conference and exposition in Copenhagen, Denmark, in 1993. The agreement, formalizing relations between the two organizations, also made possible joint membership for qualifying persons.



FSCT Corrosion Committee Chairman Jay Austin greets visitors at the OCCA SURFEX '92 Exhibition

The Oil & Colour Chemists' Association is headquartered at Priory House, 967 Harrow Rd., Wembley, Middlesex (London), England HA0 2SF. Inquiries should be directed to General Secretary Christopher Pacey-Day.

## Southwestern Paint Convention Features New Format; 222 Attend Events at Houston Resort, March 29-31

In response to a member survey, the Southwestern Paint Convention presented a new look at its 1992 event, held March 29-31, at the South Shore Harbour Resort & Conference Center, in League City (Houston), TX.

Following its 1990 meeting, the Houston Society conducted a survey of its membership to determine the needs of attendees. 1992 General Chairman Dennis Crozier, of Crozier-Nelson Sales, Inc., and his committee effected changes in the format to include simultaneous program sessions on a variety of topics and a suppliers table top exhibit.

Featured in the program were the following sessions:

### INTRODUCTION TO COATINGS

"Introduction to Surface and Colloids Coatings"—a one-half day seminar presented by Professor Richard J. Ruch, of Kent State University.

### FORMULATIONS

"Polymer Emulsions—What the Paint Formulator Should Know"—Stephen C. Cooper, of Unocal Chemicals;

"Functional Mineral, Synthetic, and Metallic Pigments in Compliance Coatings"—Edward F. McCarthy, of Cyprus Industrial Minerals;

"Waterborne Silicone-Based Coatings for High Temperature Service"—Jay W. Adams, of Tego-Chemie Service U.S.A.;

"Epoxy Waterborne and High Solids Curing Agent Technology for Compliant Coatings"—Z.K. Risinger, of Pacific Anchor Chemical; and

"New Catalysts for High Solids Coatings"—Robert G. Middlemiss, of Mooney Chemicals.

### OPERATION & ENVIRONMENT

"Emergency Planning and Community Right-to-Know Act of 1986"—Fendol M. Chiles, of U.S. E.P.A.;

"Hazardous Waste Disposal and Site Contamination"—Marty Sanderlin, of Texas Water Commission;

"Clean Air Act"—David Howell, of T.A.C.B.;

"HM181, Hazardous Material Shipping"—Bob McDilda, of D.O.T.; and

"Wrap-Up and Overview"—John Tenini, Environmental Consultant.

A General Session featuring a panel discussion of executives representing both manufacturers and suppliers was moderated by James Ignatow, of Rectorseal Corpora-



OCCA President Fred J. Morpeth (left) and SLF President Michael Symes sign cooperative agreement in Harrogate

(Continued on next page.)

## “Responding to the Challenges of the 90s” Focus of Washington Paint Group’s 1992 Symposium

The Washington Paint Technical Group (WPTG) held its 32nd Annual Symposium, “Responding to the Challenges of the 90s,” on April 7-8, at the Ramada Inn, in Tysons Corner, VA.

This year’s Keynote Address was delivered by Marcel Gaschke, of CIBA-GEIGY Corporation, whose topic was “Industry Responding to the Challenges of the 90s.”

Other symposium presentations included:

“Air Monitoring During Blasting Operations”—Gwen Russel, of Tracor Advanced Technology Inc.;

“Coatings: Status of Regulations”—Madeline Harding, of The Sherwin-Williams Company;

“Laser Cleaning of Coatings”—Bill Moeny, of Tetra, Inc.;

“New and Promising Methods of Surface Preparation”—Jim Fuller, of NAVSEA 07;

“Environmental Issues Affecting Abrasive Selection”—Jim Hansink, of Barton Mines, Inc.;

“Recyclable Abrasives”—Bill Hitzrot, of Chesapeake Specialty Products;

“Abrasive Blasting with Foam Encapsulated Abrasive”—Jon Cavallo, of Sponge-Jet, Inc.;

“U.S. Navy Efforts Towards VOC Compliance”—Jim Rudroff, of SEA 5141;

“Virginia DOT Approach to VOC Compliance”—Tom Neal, of Virginia DOT;

“The Impact of VOC Regulations on Bridge Fabricators”—Arthur Miles, of Atlas Machine and Iron, Inc.;

“Advances in Application Methods”—Bob Thompson, of Graco, Inc.;

“Advances in Resin Technology”—David Watson, of Rohm and Haas Company;

“VOC Compliant Urethane Bridge Coating Systems”—Carl Angeloff, of Miles, Inc.;

“Containment Technology”—Gerry McNamara, of IPEC Advanced Systems;

“Lead Stabilization”—Lloyd Smith, of Corrosion Control Consultants and Labs, Inc.; and

“Future of Zinc Coatings”—John Malone, of American Galvanizers Association.

For more information on the symposium, write WPTG, P.O. Box 7686, McLean, VA 22106.

## Record-Breaking Attendance Highlights 19th Annual Waterborne Symposium

A total of 428 people attended this year’s 19th Annual Waterborne, Higher-Solids, and Powder Coatings Symposium, held on February 26-28, at the Hyatt Regency Hotel, in New Orleans, LA. The Symposium is co-sponsored by the Southern Society for Coatings Technology and the Polymer Science Program at the University of Southern Mississippi (USM), Hattiesburg, MS.

The symposium, which focused on the chemistry, formulation, and new developments in waterborne, higher-solids, and powder coatings, featured a total of 40 technical presentations, including the Plenary Lecture which was delivered by Robert Y. Lochhead, of USM, on “Role of Surface Active Agents in Formulating Waterborne Coatings.”

This winner of this year’s Elias Singer Best Paper Award, sponsored by Troy Chemical Corporation, was the presentation

“The Dominant Role of Organic Peroxide Initiators in Acrylic HSC Resin Synthesis and Properties,” by Peter A. Callais, Vasanth R. Kamath, and Michael G. Moskal, of Atochem North America. Mr. Callais presented the award-winning paper to the record-breaking crowd of attendees.

In addition, the two short courses held immediately prior to the Symposium on February 24-25, realized an attendance of 81 people. The courses, “Modern Coatings Technology” and “Water-Soluble Polymers,” were conducted by USM faculty members.

For more details on the Waterborne Symposium or the short courses, or to order a copy of the Symposium’s Proceedings, write USM, Dept. of Polymer Science, P.O. Box 10076, Southern Station, Hattiesburg, MS 39406-0076.

## Manufacturer and Supplier Panel Discussion Highlights Southwestern Paint Convention

(Continued from previous page.)

tion, on the final morning. The speakers included the following: Gerry Skinner, U.S. Sales Manager, of Tioxide, Inc.; Jim Awalt, President, of Monarch Paint Company; John Ballard, National Coatings Manager, of Burgess Pigment Company; Gary Cianfichi, Market Manager, of SCM Chemicals, Inc.; Tom Taylor, Vice President/General Manager—Marine Sales, of International Paint; and Paul Johnston, Vice President/General Manager, of Union Carbide Corporation.

The meeting concluded with an address by Federation of Societies for Coatings Technology President William F. Holmes, of National Pigments & Chemicals, Inc., and Executive Vice President Robert F. Ziegler, who presented an update on Federation affairs and programs, and noted that the FSCT 1993 Spring Week meetings also will be held at the South Shore Harbour Resort & Conference Center.

### CALL FOR PAPERS

Symposium  
on  
“Plain and Simple:  
Compliance”

Sponsored by  
Piedmont Society for  
Coatings Technology  
Technical Committee

March 1993  
High Point, NC

The Technical Committee of the Piedmont Society for Coatings Technology is inviting authors to submit papers to be presented at their symposium on “Plain and Simple: Compliance,” scheduled for March 1993, in High Point, NC.

All papers on compliance and related areas are invited. A one-page abstract, single spaced with one-inch margins on white bond paper is required. All abstracts are due no later than September 15, 1992. Authors of selected abstracts will be notified by November 1, 1992.

Also, a supplier exhibition is scheduled to run concurrently with the technical program.

For more information, or to submit abstracts, contact: Robert C. Matejka, Akzo Coatings Inc., 1431 Progress St., P.O. Box 2124, High Point, NC 27261; Telephone: (919) 841-5111.

## Du Pont Announces Environmental Seminar Schedule; Safety Training Observation Programs Expanded

Du Pont Safety and Environmental Resources, Wilmington, DE, has announced its 1992 seminar schedule.

The company is sponsoring the seminar, "Managing Environmental Issues: Techniques that Work for the Environmental Professional," at various locations in the U.S.

The two-and-one-half-day course is designed to help environmental professionals manage the growing network of environmental information, requirements, and issues.

The seminar is scheduled for the following dates and locations: May 12-14, Houston, TX; June 9-11, Atlantic City, NJ; September 29-October 1, Nashville, TN; and December 8-10, New Orleans, LA.

Du Pont also is offering the two-and-one-half-day seminar, "Fundamentals of RCRA."

The Resource Conservation and Recovery Act class will provide environmental managers and specialists, plant managers, and process engineers with a working knowledge of the regulations, and is de-

signed to help participants determine the effectiveness of existing compliance plans.

The seminar will be offered: July 14-16, Dallas, TX; October 6-8, Williamsburg, VA; and December 8-10, Wilmington.

In other news, Du Pont has announced a new program designed to teach employees the principles of ergonomics and how to eliminate ergonomic risk factors.

The "Safety Training Observation Program (STOP)" for Ergonomics is based on the principles that all injuries and occupational injuries can be prevented and that safety is everyone's responsibility.

A new international-Spanish translation of the STOP program has been produced. The Spanish edition of the program is designed to aid organizations in training their Spanish-speaking personnel in safety.

For additional information on the seminars or the STOP programs, contact: Linda Manelski, Du Pont Safety and Environmental Resources, Montgomery Bldg. 277A, P.O. Box 80800, Wilmington, DE 19880-0800.

## Air Quality Regulations Course Slated for UC Berkeley, June 9-12, in San Francisco, CA

The course, "Getting into Compliance with Air Quality Regulations for Paints, Coatings, and Printing Facilities," will be sponsored by Continuing Education in Engineering, University Extension, University of California, Berkeley, on June 9-12, 1992, in San Francisco, CA.

The three-and-one-half-day class will present information about the most important consequences of the Clean Air Act and state and local regulations for paints, coatings, and printing facilities. Also, special sessions on the new Clean Air Act of 1990 and air toxics are scheduled for presentation.

Instructors for the course are: Ron Joseph, of Ron Joseph Associates; Ajjay Wilson, of California's South Coast Air Quality Management District; John Howell, Jr., of Galson Corporation; and Robert E. Kalweit, of Swanson Environmental.

For more information, contact: Alice Boatwright, UC Berkeley Extension, 2223 Fulton St., Berkeley, CA 94720.

## 35th Annual Cleveland Society Conference on Advances in Coatings Technology

*"Advances in Environmentally Compliant  
Coatings Technology"*

Thursday, May 28, 1992

NASA, Lewis Research Center, Cleveland, OH

Meet the Speaker Social Hour, Banquet, and After Dinner Speaker to Follow at:  
The Sheraton Airport Hotel, Cleveland, OH

Member of



Since 1922

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Company Name: \_\_\_\_\_

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Telephone Number: \_\_\_\_\_

If FSCT Member: \_\_\_\_\_ Society

### Send registration to:

James A. Miller  
3057 Kent Road  
Cuyahoga Falls, OH 44224  
Phone: (216) 352-0218

FSCT Member Conference Registration .....	( )	\$ 90.00
Member Registration with Banquet .....	( )	115.00
Retired Member—Conference Only .....	( )	45.00
Full Time Student—Conference Only .....	( )	45.00
Retired/Student—Conference & Banquet ...	( )	70.00
Non-FSCT Members, Add .....	( )	10.00
Registration after May 10, Add .....	( )	10.00
Speaker Social and Banquet Only .....	( )	30.00

### Banquet Dinner Choice:

Prime Rib \_\_\_\_\_ Chicken Piccata \_\_\_\_\_ Vegetarian \_\_\_\_\_

### Accommodations:

Sheraton Airport Hotel Cleveland  
5300 Riverside Drive  
Cleveland, OH 44135  
Phone: (216) 267-1500 or (800) 362-2244

Please make your reservation directly with the Sheraton Hotel. Use I.D. #GA8223 for CSCT Conference Rate of \$70.00 for Standard Room.

# Book Review

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## **AN INFRARED SPECTROSCOPY ATLAS FOR THE COATINGS INDUSTRY. Volumes I and II. Fourth Edition**

Edited by  
Darlene R. Brezinski

Published by  
Federation of Societies for Coatings  
Technology  
492 Norristown Rd.  
Blue Bell, PA 19422 (1991)  
1024 Pages, List Price: \$200.00  
Member Price: \$150.00

Reviewed by  
Thomas J. Miranda  
Consolidated Research, Inc.  
Mt. Prospect, IL

One of the most valued publications of the Federation of Societies for Coatings Technology is its *Infrared Spectroscopy Atlas for the Coatings Industry*. This Fourth Edition is a welcome update to this popular analytical tool. Authored by leading experts in the field, this two volume set is greatly updated and expanded over the previous publication produced in 1980. Fully indexed, the new edition contains 2,500 spectra of materials commonly used in the coatings industry. These spectra were obtained using high resolution Fourier Transform infrared spectrophotometers. Spectral curves were computer enhanced by Nicolet Instrument Corp., Madison, WI, to remove the interfering effects of water vapor and carbon dioxide resulting in a pure spectral curve of the represented chemicals listed.

Volume I consists of eight chapters which cover the theory, instrumentation, accessories for infrared instrumentation, sample preparation for infrared analysis, qualitative analysis, quantitative analysis, and bibliography; the most complete for this type of text and a catalog of 1224 spectra.

Volume II contains the remaining spectra which are printed in a three to a page format. Spectral curves are clearly drawn and well identified. The index lists spectra by number, chemical type, supplier, and trade name. An alphabetical list is also provided.

The first chapter discussed in detail an overview of the theory of Infrared Spectroscopy, tables of assigned frequencies, and nomenclature. Chapter two covers instrumentation and compares Dispersive and Fourier Transfer instruments, while chapter three describes accessories for IR instrumentation such as Attenuated Total Reflec-

tance (ATR), Multiple Internal Reflectance (MIR), and microsampling devices. Sample preparation, a most important aspect of IR analysis, is covered in chapter four which includes thermal methods such as pyrolysis and surface contamination. Chapter five describes the use of IR in making qualitative analysis of materials by function group content in the spectra.

Chapter six presents a discussion of the quantitative aspects of IR using absorbance curves. Chapter seven includes a list of 1427 references which were gleaned from a review of some 13,000 references. These are categorized by section ranging from Theory and Instrumentation to polymer and coatings, with emphasis based on these materials. Chapter eight lists spectra which is continued into the second volume of the Atlas.

The spectra of chemicals include: polymers, monomers, solvents, amines, inorganic

pigments and extenders, organic pigments, additives, and miscellaneous materials such as stopcock grease, mineral oils, and others.

Another valuable feature of this new publication is the availability of these spectra on a computerized database which can be purchased from Nicolet at an additional cost. This two-volume set is case bound and packaged in an attractive, sturdy slip case.

What is really valuable is the excellent work done by the Infrared Spectroscopy Advisory and Working Committees who undertook this monumental task and completed the work in a timely manner. These volumes are an essential addition to any laboratory using this important analytical tool and will again prove its value as a major contribution of the volunteer efforts of dedicated members of the coatings industry.

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## **ADVANCED SURFACE COATINGS: A Handbook of Surface Engineering**

Edited by  
D.S. Rickerby and  
A. Matthews

Published by  
Blackie and Son Ltd.  
c/o Routledge, Chapman & Hall  
29 W. 35th St.  
New York, NY 10001 (1991)  
368 Pages, \$149.95

Reviewed by  
Michael Graham  
Northwestern University  
Evanston, IL

This book is a cohesive and concise presentation of the phenomenology associated with creating and characterizing coatings for engineered surfaces. The central idea is that the tools exist for designing and engineering surfaces to meet application needs. The book is organized in a thoughtful and focused way to provide engineers and scientists of varied backgrounds with a clear understanding of the advanced techniques and respective places in the vast array of surface engineering technologies. The style and structure of the individual chapters, though written by different authors, is controlled so that there is a continuity that contributes to the readability of the book. The authors are all respected contributors in their

fields and provide authoritative reviews of the state-of-the-art in those fields.

The authors emphasize the basic understanding of the phenomena in the respective technologies, while using selected data as illustrative material. Attention is paid to existing and potential applications where appropriate. The book is perhaps strongest in its treatment of plasma based processes, so it is fitting that Chapter 2 is a review of the generation of plasmas, and some developments in the use and control of plasmas in certain processes. Succeeding chapters cover ion implantation, ion-assisted coatings, evaporation, sputtering, physical vapor deposition, chemical vapor deposition, thermal spraying, and laser treatments. The chapters summarize the materials developments associated with the technologies and include case studies and applications information to assist the reader in assessing the various technologies, their limitations, and their capabilities.

In addition to the process oriented chapters, there are two chapters on the characterization and evaluation of coatings. Chapter 11 reviews the analytical techniques available and their appropriate use in determining chemical and structural information, while Chapter 12 deals with the physical methods for determining mechanical properties of treated surfaces. In the final chapter, the book takes a forward look into the anticipated developments in the processing technology, new materials and their utilization for creating "designer surfaces."



## POLYMER GELS (Fundamentals & Biomedical Applications)

Edited by  
Danilo DeRossi, Kanji Kajiwara,  
Yoshihito Osada, and  
Aizo Yamauchi

Published by  
Plenum Publishing Corp.  
233 Spring St.  
New York, NY 10013  
345 Pages, \$85.00

Reviewed by  
Joseph V. Koleske  
Consolidated Research  
Charleston, WV

This book is a well-edited compilation of papers that were presented at the Symposium on Polymer Gels sponsored by The Society of Polymer Science, the Science Technology Agency, and MITI, all of Japan, at Tsukuba Science City, on September 18-19, 1989. Overall, the papers in this book deal with progress in understanding the properties of polymer gels and how these products are applied to biomedical, environmental, and robotic fields. The papers are grouped into five sections: Fundamentals, Biologicals, Separation, Sensors and Actuators, and Drug Delivery Systems.

The papers primarily deal with the swelling behavior of artificial and natural hydrogels and chemomechanical systems. Energy is converted or transferred in these systems by means of a chemical or physical stimuli. Research into polymeric networks that are capable of actuation by, of processing, and of sensing such stimuli have inspired development of new controllable membrane processes, delivery systems, and actuators. Artificial chemomechanical systems are useful for imitating and investigating fundamental processes of the biological world, but offer potential for future commercial applications. From such knowledge will come novel robotics and functional replacement limbs or other devices.

At first, it may seem that this book has little applicability to coatings. Yet, coatings are not impervious films. They are porous membranes whose porosity depends on the particular polymers used, degree of crosslinking, the degree of swelling, etc., that takes place. Factors such as these allow passage of materials to the substrate they are protecting and corrosion can occur. One can envision that an understanding of the dynamics of coatings relative to polymer gels could lead to improved coatings. Coatings that could sense changes in their environment and inhibit, for example, salts from passing through the coating. Coatings that sense changes and expand or contract and perhaps actuate another mechanism, might protect a substrate during changes in tem-

perature, humidity, or other hostile change. Drug delivery systems are akin to delivery of a coating additive within a coating in a controlled or measured manner. In the environmental sense, there is information that deals with removal of water from biological slurries such as activated sludges and other

organic waste slurries in waste-water treatment plants.

Overall, this book does not answer current problems of the coatings industry. However, it could be the stimulus that provides ideas for novel technology that would have utility in the future.

## SILANE COUPLING AGENTS Second Edition

Authored by  
Edwin P. Plueddemann

Published by  
Plenum Press  
233 Spring St.  
New York, NY 10013  
253 Pages, \$55.00

Reviewed by  
Gerald L. Witucki and  
Peter G. Pape  
Dow Corning Corp.  
Midland, MI

Formulators in the paint, ink, adhesives, water repellents, reinforced plastics and other related industries, require materials that resist peeling and penetration in spite of prolonged exposure to heat, cold, moisture, and chemical attack. The focus on quality, necessary to compete in the global marketplace, requires top performing, cost effective raw materials. Research into these needs resulted in the development of alkoxy functional silanes for use as adhesion promoters, crosslinkers, and hydrophobing agents.

Written by the "father" of silane coupling agent science, *Silane Coupling Agents* is the most comprehensive text dedicated solely to silane coupling agents. This book includes the author's philosophy of science as it relates to adhesion and the role of silane coupling agents. Updated to include scientific results since the first printing in 1982, this second edition contains additional information in specific areas such as: metal oxide bonding, equilibrium constants, UV cure coatings, and ionomer bonding.

The first five chapters of this book are research oriented with detailed explanations of chemical theories, mechanisms, and basic phenomena associated with silane coupling agents. Copious references are included for each chapter.

The remaining three chapters deal with the application and performance of silane coupling agents in numerous industrial markets. Having worked closely with numerous companies in solving adhesion and coupling problems, the author's experience makes this text an invaluable tool for understanding the practical applications of silane coupling agents. Detailed experimentation and simplified test methods allow readers to apply the technology to their own specific application.

The "Windy City" will play host for the  
Federation's 1992 Annual Meeting  
and Paint Industries' Show in October  
at McCormick Place North.

So if you don't want to be blown away by the  
competition, make your plans **NOW** to attend  
the biggest paint  
industry event of the year!!!



McCormick Place North  
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For more details, contact:  
Federation of Societies for  
Coatings Technology,  
492 Norristown Road, Blue Bell,  
Pennsylvania 19422  
Phone: 215/940-0777  
FAX: 215/940-0292

# Elections

## BALTIMORE

### Associate

*Behan, David J.*—Dunleary, Inc., Towson, MD.  
*Rand, Jeffrey L.*—D.H. Litter, York, PA.  
*Roche, Patrick J.*—Penn Bottle & Supply, Baltimore, MD.

## CDIC

### Active

*Centofante, Dennis A.*—Lilly Industrial Coatings, Indianapolis, IN.

### Associate

*Ellis, Kevin A.*—Michelman Inc., Cincinnati, OH.  
*Inman, Steve*—B.F. Goodrich, Cincinnati.  
*Paul, Edward G.*—Queen City Barrel, Cincinnati.  
*Shawver, Leslie S.*—W.R. Grace & Co., Cincinnati.  
*Towell, David B.*—Michelman Inc., Cincinnati.

## CHICAGO

### Active

*Boyd, Douglas D.*—Ace Hardware Corp., Paint Div., Matteson, IL.  
*Bullis, Jonathon P.*—Rust-Oleum Corp., Pleasant Prairie, WI.  
*Byrd, Robert W.*—NSCC/Thiem Div., Oak Creek, WI.  
*Caplan, Beth A.*—Valspar Corp., Wheeling, IL.  
*De Brocke, Susan L.*—Pre Finish Metals Inc., Elk Grove Village, IL.  
*Grazys, Gint*—Daubert Chemical Co., Chicago, IL.  
*Hallberg, Steve P.*—Ace Hardware Corp., Paint Div., Matteson.  
*Lapin, Stephen C.*—DSM Desotech, Elgin, IL.  
*Mann, Richard E.*—Elpaco Coatings, Elkhart, IN.  
*Pfeiffer, Susan M.*—Valspar Corp., Wheeling.  
*Runge, Meredith A.*—Tru-Test Mfg., Cary, IL.  
*Schroeder, William F.*—Valspar Corp., Wheeling.  
*Van Valkenburg, Terry*—Premier Coatings Inc., Elk Grove Village.  
*Zable, Robert E.*—Pre-Finish Metals Inc., Elk Grove Village.  
*Zucker, Mark S.*—Valspar Corp., Wheeling.

### Associate

*Jaegersberg, Rolf K.*—CPA International Consulting Services, Big Bend, WI.  
*Krinsky, Peter R.*—SCM Chemicals, Olympia Fields, IL.

## LOS ANGELES

### Active

*Burkes, Louis E.*—Textured Coatings, Los Angeles, CA.  
*Cao, Long T.*—BAF Industries, Inc., Santa Ana, CA.  
*Grant, Jim C.*—San Diego Power Wash, Lemon Grove, CA.  
*Hall, James D.*—Sinclair Paint Co., Los Angeles.

*Thompson, Carl V.*—Continental Coatings Inc., Fontana, CA.

### Associate

*Kaczmarek, Mitchell A.*—Norman International, Los Angeles, CA.

## LOUISVILLE

### Active

*Talbott, Gordon W.*—PPG Industries, Inc., Louisville, KY.

### Associate

*Gratzle, Jennifer L.*—Cargill Inc., Carpentersville, IL.  
*Reisinger, Mark A.*—Avganic Industries, Carmel, IN.

## MONTREAL

### Associate

*Cavalancia, Bruno A.*—QIT-Fer Et Titane Inc., Montreal, Que.  
*Giroux, Richard*—Les Systemes Adex, Quebec, Que.  
*Montagano, Michael J.*—St. Lawrence Chemical, Baie D'Urfé, Que.

## NEW YORK

### Active

*Abe, Amy*—L&F Products, Montvale, NJ.  
*Gilman, William S.*—Leberco Testing Inc., Roselle Park, NJ.  
*Ingoglia, Augie A.*—RBH Dispersions, Bound Brook, NJ.  
*Lim, Nonato Y.*—Transpo Industries, Inc., Edison, NJ.  
*Marinulich, Gerald E.*—Lonza Inc., Annandale, NJ.  
*Tubbs, Forrest W.*—Allied-Signal Inc., Morristown, NJ.  
*Walsh, John P.*—Thibaut & Walker Co., Inc., Newark, NJ.

### Associate

*Curry, Delrika A.*—Aqualon, Wilmington, DE.  
*Oliver, Richard B.*—RBH Dispersions, Inc., Bound Brook, NJ.  
*Orr, Edward W.*—Byk-Chemie USA, Wallingford, CT.  
*Silver, Ben*—General Color Co., Newark, NJ.  
*Taylor, Samuel O.*—DuPont Chemicals, South Orange, NJ.

### Retired

*Leister, Edwin S.*—Shorthills, NJ.  
*Wexler, Marvin*—Ridgefield, NJ.

## NORTHWESTERN

### Active

*Linert, Jeffrey G.*—3M, St. Paul, MN.

*Radosovich, Todd M.*—Knight Color & Chemical, Montrose, MN.

*Van Iseghem, Lawrence C.*—Van Technologies, Inc., Duluth, MN.

### Associate

*Benson, Dan W.*—Chemcentral/Minnesota, Lakeville, MN.  
*Velkovich, Dan*—Chemcentral/Minnesota, Lakeville.

## PHILADELPHIA

### Active

*Ba, Sujuan*—Arco Chemical Co., Newtown Square, PA.  
*Bossert, Emily Carol*—Elf Atochem North America, Somerville, NJ.  
*Charnoski, Jan M.*—Silberline Mfg. Co., Tamaqua, PA.  
*Grant, John K.*—IPA Systems, Inc., Philadelphia, PA.  
*Holton, James S.*—Elf Atochem North America, King of Prussia, PA.  
*Miller, Michael W.*—Whitford Corp., West Chester, PA.  
*Patel, Ashok C.*—Devoe Coatings Co., Pennsauken, NJ.  
*Senior, Donald R.*—Congoleum Corp., Trenton, NJ.  
*Shalov, Allen A.*—Congoleum Corp., Trenton.

### Associate

*Druzbacky, James S.*—American Cyanamid Co., Titusville, NJ.  
*Firestone, Daniel F.*—S.E. Firestone Associates, Philadelphia, PA.  
*Hawk, John A.*—IBC Systems, Reading, PA.  
*Thomas, Del*—Alcoa, Raymond, NH.

## SITUATIONS WANTED

Twenty plus years experience, R&D, manufacturing operations, technical and general management. Ph.D. coatings chemistry (NDSU). Seeks challenging, responsible opportunity; prefers western location. F.D. Williamson, 7441 Muirwood Ct., Pleasanton, CA 94588, (510) 846-5052.

\* \* \*

I am an Industrial Chemist with 20 years of experience which includes coil coating, technical service, and management and have a solid background in formulation with alkyd, epoxy, vinyl, and urethane. I am a Canadian citizen with a U.S. Green Card and seek an opportunity to move to the United States. Ajmal Mahmood, 1706 Woodside Lane, Pickering, Ontario L1V 4W3 Canada.

## Viscosity Sensor

The announcement of a new in-process viscosity sensor for measuring viscosity in processes where cleanliness is important has been made through literature. Sensor features include: FM approval for hazardous fluids; all stainless steel construction; internal RTD temperature probe; and temperature compensated viscosity. For additional information on the Model SPC311 sensor, write Cambridge Applied Systems, Inc., 57 Smith Place, Cambridge, MA 02138.

## Cellulose Fillers

A technical data sheet on cellulose and cotton fiber functional fillers has been printed. Applications include: theroset and thermoplastic resins, building products, pet food, asphalt mixtures, rubber goods, gaskets, sealants, brake linings, latex paints, and welding electrodes. The fillers are available in more than 20 grades with cellulose contents ranging from 70 to 90%. Write to Celite Information Center, 1601 Park Avenue W., Denver, CO 80216, for more details on Fibra-Cel® fillers.

## Surface Profiler

The introduction of a high resolution noncontact surface profiler has been made through a bulletin. The profiler offers a 512 x 512 pixel detector with 15 µm pixels to provide spatial sampling to 0.15 µm at 100x. The instrument can be used for measuring surfaces with high frequency detail such as thin film head pole tips, magnetic and optical media, and precision machined surfaces. For further in-depth details on the Model 5800 Profiler, contact Zygo Corp., Laurel Brook Rd., P.O. Box 448, Middlefield, CT 06455-0448.

## Column Module

A high performance column module designed to enhance HPLC systems has been introduced through literature. The module is suited for isolation and purification of naturally occurring and synthetic, hydrophilic and hydrophobic peptides, lipids, phospholipids, etc., of biological and pharmaceutical interest. Further information on the High Performance Centrifugal Partition Chromatography (HPCPC™) Module can be obtained by writing Sanki Laboratories, Inc., Information Div., 520 Fellowship Rd., Ste. D-406, Mt. Laurel, NJ 08054-3410.

## Spectroscopy Software

Spectroscopy software designed to operate in scanning, photometric, time-course, and quantitative modes is highlighted in literature. The system features pull-down menus and keyboard or mouse operation. For more information on the PC-1201 software system, contact Shimadzu Scientific Instruments, Inc., 7102 Riverwood Dr., Columbia, MD 21046.

## Polyurethane Rubber System

The development of a fast-setting, solvent-free, two-component liquid polyurethane rubber system has been announced through a product bulletin. The product can reportedly be used to repair a wide-range of rubber products including conveyor belts and mechanically molded goods. For more details, write Alan Peach, Urylon Plastics Inc., 385 Elmira Rd., Guelph, Ont., N1K 1H3 Canada.

## Color Management System

The development of a computerized color management system for semi-transparent wood stains has been announced. The system is designed to allow stain manufacturers to consistently create identical colors. Contact Brian R. Kates, Marketing Manager, Colorgen Industrial Group, 28 Green St., Newbury, MA 01951, for further details on the StainMatch system.

## Electrochemical Accessories

A 40-page catalog detailing an extensive line of accessories for electrochemical measurement instruments and systems has been issued. The publication features accessories for rotators, polarographic instruments, corrosion measurement systems, flat cells, and micro cells. Write EG&G Princeton Applied Research, Electrochemical Instruments Div., P.O. Box 2565, Princeton, NJ 08543 for more details.

## Polyurethane Dispersion

A high-solids aqueous polyurethane dispersion is the topic of a technical data sheet. The product is specially suited for plastic, rubber, and leather materials. Contact Witco Corp., Polyurethane Specialties Group, 2701 Lake St., Melrose Park, IL 60160-3041 for more details on Witcobond® W-404 dispersion.

## Safety Reference Guide

A 44-page laboratory safety reference guide, which includes a review of spill and exposure classifications for 22 hazardous or potentially hazardous chemical groups, has been printed. Also included in the guide is an incompatible chemicals chart which identifies chemicals that can cause a hazardous condition if they react together. For information on how to obtain a copy of the "1992 EM Science Product Catalog and Laboratory Safety Guide," write EM Science, P.O. Box 70, 480 Democrat Rd., Gibbstown, NJ 08027.

## Surfactants

A line of surfactants for wood coatings producers is the topic of literature. They are designed to be used in spray-applied coatings, waterborne acrylics, and acrylic-urethanes typically used in wood applications. Contact Air Products and Chemicals, Inc., Performance Chemicals Div., 7201 Hamilton Blvd., Allentown, PA 18195-1501, for further information on Surfynol® surfactants.

## Chemical Finishing Systems

A reference guide providing detailed information on a complete line of production finishing systems for wood, metal, and plastic is available. The 72-page publication provides information on related topics such as safety, environmental regulations, application techniques, specification-writing, surface preparation, and trouble-shooting. All inquiries for additional information should be identified as "Chemical Coatings Finishing Systems Guide (SWS-3670)," and sent to The Sherwin-Williams Co., c/o HKM Direct, 5501 Cass Ave., Cleveland, OH 44102.

## Shipping and Storage Container

A new 60-gallon shipping and storage container, developed to provide an environmentally safe, secure, returnable small drum, has been introduced through literature. The container features heavy-duty double-wall construction for full secondary containment and forklift entry tubes for sure handling. Additional information on the new 60-gallon Jumbo™ Drum is available from Clawson Tank Co., P.O. Box 350, 4545 Clawson Tank Dr., Clarkston, MI 48346-0350.

## Acrylic Resin

A high solids, low viscosity acrylic polyol for two-package, ambient cure acrylic urethane systems is the subject of a data sheet. The resin has been designed for coatings requiring durability, solvent resistance, and anti-abrasion properties which would be typically found in industrial flooring or decking topcoats. Write George Roy, Rhône-Poulenc Inc., Specialty Resins Group, 9808 Bluegrass Pkwy., Louisville, KY 40299, for more in-depth information on CMD 9028 resin.

## Accelerated Weathering Tester

A brochure introducing an accelerated weathering tester has been printed. The publication provides detailed information on features, as well as technical specifications and data. For a copy of the brochure on the UVCON accelerated weathering device, write Atlas Electric Devices Co., 4114 N. Ravenswood Ave., Chicago, IL 60613.

## Gas Chromatography Column

The introduction of a column for gas chromatography has been made through a product release. The column is designed to be used at elevated temperatures that are above the typical capillary column operating temperature of 300-350°C. For further details on the new DB™-5ht column, contact J&W Scientific, 91 Blue Ravine Rd., Folsom, CA 95630.

## Acrylic Emulsions

A new line of high performance specialty acrylic emulsions designed for maintenance, industrial, and specialty architectural applications is the topic of a data sheet. The emulsions are engineered to provide corrosion resistance, humidity resistance, and high "alkyd-like" gloss characteristics. For further details on Carboset® CR 760 series emulsion polymers, write The BFGoodrich Co., Specialty Polymers and Chemicals Div., Industrial Coatings Products Group, 9911 Brecksville Rd., Cleveland, OH 44141-3247.

## Chromatography Systems

Gas chromatography products and systems designed to increase a paint and coatings laboratory's analysis options is the subject of a brochure. As outlined in the bulletin, the range of products extends from simple valve installations in gas chromatographs, to automated, engineered chromatography systems. For a free copy of the brochure, "Engineered Chromatography Products and Systems," contact The Perkin-Elmer Corp., 761 Main Ave., Norwalk, CT 06859-0012.

## Polyvinyl Alcohol

A data sheet describing the properties of standard and specialty grades of a polyvinyl alcohol has been published. Descriptions of each grade and their properties are listed in a full-page chart. For a copy of the publication, "Airvol® Polyvinyl Alcohol Typical Properties," contact Air Products and Chemicals, Inc., Polymer Chemicals Div., 7201 Hamilton Blvd., Allentown, PA 18195-1501.

## Hydroxyl Functional Polyols

A new line of low viscosity, 100% solids hydroxyl functional polyols is being highlighted in a product sheet. Fifteen different alkoxyated products based on bisphenol A, trimethylolpropane, glycerol, neopentyl glycol, and phenol are available. More details on Photonol® PHO-7000 products can be obtained by contacting Henkel Corp., 300 Brookside Ave., Ambler, PA 19002.

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## Epoxy Curing Agent

Product literature has been released on a new low viscosity curing agent. The agent is designed to exhibit resistance to water, salts, and acids; fast cures at low temperatures (<40°F) with long working times; moisture resistance during cure; and adhesion to marginally prepared surfaces. Contact Dierdre Igo, Cardolite Corp., 500 Doremus Ave., Newark, NJ 07105, for further information on CARDOLITE NC-541LV.

## Amino Crosslinking Agents

A four-color brochure featuring two high solids amino crosslinking agents is now obtainable. Property comparisons and typical characteristics charts, as well as the important characteristics of the resins, are detailed throughout the brochure. For a copy of the publication detailing CYMEL® and BEETLE® high solids amino crosslinking agents, contact American Cyanamid Co., One Cyanamid Plaza, Wayne, NJ 07470.

## Filtration Products

Ultrafiltration membrane components and their applications are the topic of a brochure. Color photos and charts aid in describing the characteristics and benefits of the membrane components. For a copy of the brochure, "Advanced Membrane-Based Products for Filtration," contact Joan Slep, Hoechst Separations Technologies, Separation Products Div., Hoechst Celanese Corp., 13800 S. Lakes Dr., Charlotte, NC 28273.

## ISO 9000 Standards

An ISO 9000 Standards training video, which comes in two modules that can be used together, is now available. Training, employee orientation, group discussions, a management overview, etc. are subjects covered in the video. For further information on "ISO 9000: The First Step to the Future" video, write Du Pont Quality Management & Technology, Louviers, 33W46, P.O. Box 6090, Newark, DE 19714-6090.

## Diode Array Detector

Technical literature has been released on a new diode array detector. The instrument reportedly provides the ability to collect up to 2,000 spectra per run and store up to 25 spectra in the instrument's spectral library, allowing extended analysis of paints and coatings. Write The Perkin-Elmer Corp., 761 Main Ave., Norwalk, CT 06859-0012, for further details on the LC-235C Diode Array Detector.

## Clearcoat Finish

A data sheet highlighting an automotive clearcoat finish has been released. The product reportedly offers consumers "environmental-etch" resistance of two-component (2K) systems without compromising scratch and mar resistance. For further details on "Generation IV" clearcoat, write Du Pont Co., Automotive Products, 950 Stephenson Hwy., Troy, MI 48007-7013.

## Quick Air-Drying Enamel

A data sheet introducing a low VOC compliant, high-gloss, quick air-drying enamel has been released. Available in a full range of colors, the enamel can be applied by conventional, airless, HVLP, air-assisted airless, and electrostatic spray equipment. For more details on Quick Dry 350 Enamel (SWS-3660), write Sherwin-Williams, Chemical Coating Sales Group, c/o HKM Direct, 5501 Cass Ave., Cleveland, OH 44102.

## Cold Grinding Systems

Carbon dioxide cold grinding systems, designed to grind a wide range of materials to desired particle size, is the focus of a brochure. Products in the chemical, food, and pharmaceutical processing industries that are typically ground by this type of system include: plexiglas, PVC resin, epoxy, nylon, vinyl, nitrile rubber, spices, coffee beans, cocoa powder, and mustard seed. The brochure (Form #6942) is available from Liquid Carbonic, Advertising Dept., 135 S. LaSalle St., Chicago, IL 60603.

## UV/EB

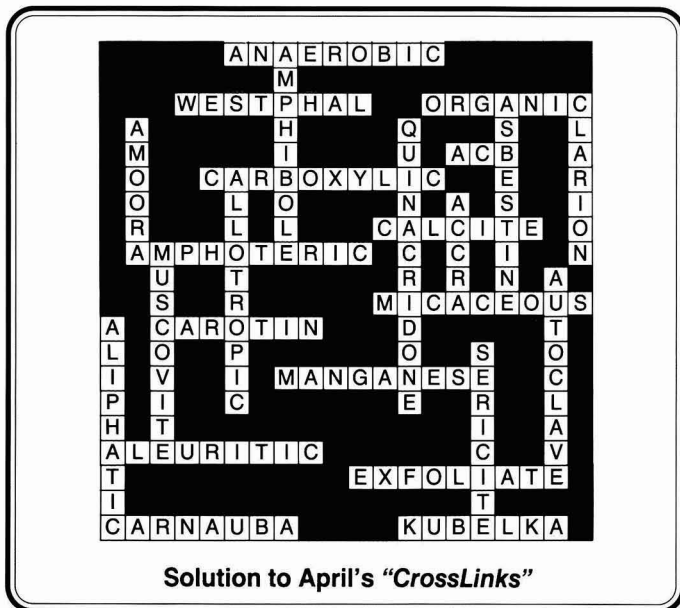
A four-page publication explaining the basics of ultraviolet (UV) and electron beam (EB) curing and drying is now available. The brochure highlights the chemistry of UV/EB technology to its environmental friendliness in everyday language. Contact Chris Dionne, RadTech International, 60 Revere Dr., Ste. 500, Northbrook, IL 60062, for information on obtaining copies of the brochure, "UV/EB: The Green Technology."

## Additive

A surface slip and anti-mar additive for radiation-cured coatings is being highlighted in literature. The new additive reportedly offers a solvent-free alternative for improving the flow rate and leveling of ultraviolet and electron beam-cured coatings based on a wide variety of resins. Write Union Carbide OrganoSilicon Products, Systems and Services, Dept. H2375, 39 Old Ridgebury Rd., Danbury, CT 06817-0001, for more in-depth details on SILWET® Surfactant RC-73.

## Gas Chromatography

A series of six application notes on industrial chemical analysis by gas chromatography (GC) has been introduced. Each four-page application includes information, chromatograms, and analytical results on a different industrial chemical application. To receive all six copies of the industrial chemicals GC Application Notes, write J&W Scientific, 91 Blue Ravine Rd., Folsom, CA 95630.





# Coming Events

## FEDERATION MEETINGS

For information on FSCT meetings, contact Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422 (215) 940-0777, FAX: (215) 940-0292.

### 1992

(May 17-20)—Federation "Spring Week." Board of Directors Meeting on the 17th; Incoming Society Officers Meeting on the 18th; Spring Seminar on the 19th and 20th. Sheraton Boston Hotel and Towers, Boston, MA.

(June 8-10)—"How to Implement Total Quality Management and Secure ISO 9000 Series Certification." Sponsored by Professional Development Committee. Adam's Mark Hotel, Philadelphia, PA.

(June 11-12)—"Gauge/Measurement Process Assessment and Improvement." Sponsored by Professional Development Committee. Adam's Mark Hotel, Philadelphia, PA.

(Sept. 14-15)—"Statistical Process Control and Its Application in the Coatings Industry" (SPC Level I). Sponsored by Professional Development Committee. Atlanta, GA.

(Sept. 16-18)—"Practical Application of Intermediate Statistics in a Total Quality Management System" (SPC Level II). Sponsored by Professional Development Committee. Atlanta, GA.

(Oct. 21-23)—70th Annual Meeting and 57th Paint Industries' Show. McCormick Place, Chicago, IL.

### 1993

(Oct. 27-29)—71st Annual Meeting and 58th Paint Industries' Show. World Congress Center, Atlanta, GA.

### 1994

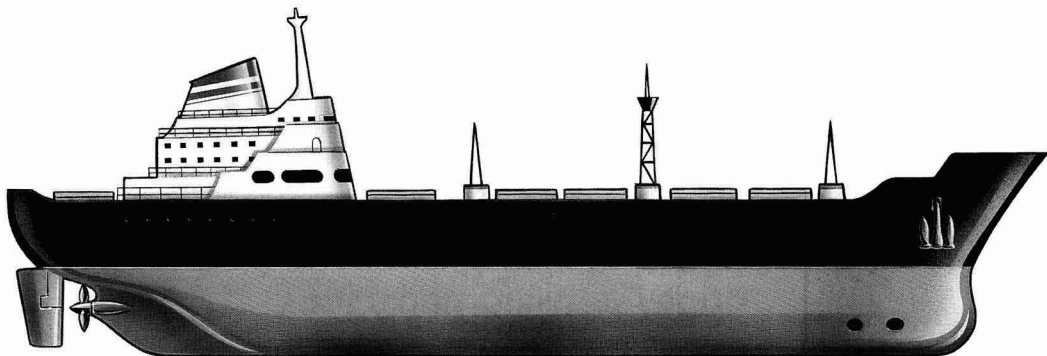
(Oct. 12-14)—72nd Annual Meeting and 59th Paint Industries' Show. New Orleans Convention Center, New Orleans, LA.

## SPECIAL SOCIETY MEETINGS

### 1992

(May 28)—Cleveland Society. "Advances in Environmentally Compliant Coatings Technology." 35th Annual Technical Conference on Advances in Coatings Technology. NASA Lewis Research Center, Cleveland, OH. (Richard Mikol, Tremco, Inc., 10701 Shaker Blvd., Cleveland, OH 44104).

(June 15)—Golden Gate Society. "Ideas for the Future." Manufacturing Committee Conference. Holiday Inn, S. San Francisco, CA. (Louie F. Sanguinetti, Jasco Chemical Corp., P.O. Drawer J, Mountain View, CA 94042).



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1993

(Mar. 23-25)—Golden Gate, Los Angeles, Pacific Northwest, and Rocky Mountain Societies. Western Coatings Societies' 21st Biennial Symposium and Show, Disneyland Hotel and Convention Center, Anaheim, CA. (Geneva H. Wells, H.M. Royal of California, Inc., 6880 8th St., Buena Park, CA 90620).

OTHER ORGANIZATIONS

1992

(May 18-20)—"Coating Process Fundamentals." Short course sponsored by Center for Interfacial Engineering and University of Minnesota. Antwerp, Belgium. (J.D. O'Brien, Dept. of Chemical Engineering and Materials Science, University of Minnesota, 421 Washington Ave. S.E., Minneapolis, MN 55455).

(May 18-22)—International Automation Conference and Training Course. Cosponsored by Instrument Society of America (ISA) and International and Scientific Computing and Automation. Montpellier, France. (ISA, 67 Alexander Dr., P.O. Box 12277, Research Triangle Park, NC 27709).

(May 19-21)—PaintCon '92. Conference and exhibition sponsored by *Industrial Finishing Magazine*. O'Hare Expo Center, Chicago (Rosemont), IL. (PaintCon '92, 2400 E. Devon Ave., Des Plaines, IL 60018).

(May 19-21)—"Additives for Coatings." Course sponsored by Center for Professional Advancement, East Brunswick, NJ. (Center for Professional Advancement, P.O. Box 1052, East Brunswick, NJ 08816).

(June 1-4)—Fifth International Symposium on Polymer Analysis and Characterization. Inuyama Hotel, Inuyama City, Japan. (Howard Barth, Du Pont Co., Experimental Station, P.O. Box 80228, Wilmington, DE 19880-0228).

(June 1-5)—"Adhesion Principles and Practice for Coatings and Polymer Scientists." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education-Chemistry, KSU, P.O. Box 5109, Kent, OH 44242-0001).

(June 2-6)—"Advances in Emulsion Polymerization and Latex Technology." Short course sponsored by Emulsion Polymers Institute, Lehigh University. Bethlehem, PA. (Mohamed S. El-Aasser, Emulsion Polymers Institute, Lehigh University, 111 Research Dr., Bethlehem, PA 18105).

(June 8-12)—"Environmentally Compliant Coatings." Short course sponsored by North Dakota State University (NDSU), Fargo, ND. (Jeanette Shaw-Lynch or Deborah M. Shasky, Dept. of Polymers and Coatings, NDSU, Fargo, ND 58105).

(June 8-12)—"Water Based Polymers." Course sponsored by Center for Professional Advancement. Chicago, IL. (Center for Professional Advancement, P.O. Box 1052, East Brunswick, NJ 08816).

(June 9-12)—"Getting into Compliance with Air Quality Regulations for Paints, Coatings, and Printing Facilities." Course sponsored by University of California-Berkeley. The Cathedral Hill Hotel, San Francisco, CA. (Continuing Education in Engineering, University Extension, University of California, 2223 Fulton St., Berkeley, CA 94720).

(June 10-11)—"Plastics in Construction" Seminar. Sponsored by the Canadian Plastics Institute. Toronto Radisson Hotel, Toronto, Ont., Canada. (Karen Wolfe, Canadian Plastics Institute, 1262 Don Mills Rd., Ste. 48, Toronto, Ont., Canada M3B 2W7).

(June 14-18)—XXIth FATIPEC Congress. RAI International Congress Centre, Amsterdam, The Netherlands. (General Secretary Francis Borel, 34 Chemin du Halage, La Bonneville, Mery sur Oise, France).

(June 15-17)—Euroformula '92. International Trade Fair. RAI International Exhibition and Congress Centre. Amsterdam, The Netherlands. (RAI, Europaplein, 1078 GZ, Amsterdam, The Netherlands).

(June 15-26)—"Coatings Science." Short course sponsored by North Dakota State University (NDSU), Fargo, ND. (Jeanette Shaw-

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And Secure ISO 9000 Series Certification"**

**JUNE 8-10, 1992**

and

**"Gauge/Masurement Process  
Assessment and Improvement"**

**JUNE 11-12, 1992**

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492 Norristown Road, Blue Bell, PA 19422  
(215) 940-0777

Lynch or Deborah M. Shasky, Dept. of Polymers and Coatings, NDSU, Fargo, ND 58105).

(June 16-18)—"Radiation Curing: Ultraviolet Light and Electron Beam Technology." IL. Sponsored by Center for Professional Advancement. Chicago, IL. (Center for Professional Advancement, P.O. Box H, East Brunswick, NJ 08816).

(June 21-24)—ISCC 61st Annual Meeting and AIC 25th Anniversary. International symposium on "Computer Color Formulation." Princeton University, Princeton, NJ. (Danny Rich, ISCC Secretary, ACS Datacolor, 5 Princess Rd., Lawrenceville, NJ 08648).

(June 24-26)—"Coatings Process Fundamentals." Short course sponsored by Center for Interfacial Engineering and University of Minnesota. Minneapolis, MN. (J.D. O'Brien, Dept. of Chemical Engineering and Materials Science, University of Minnesota, 421 Washington Ave. S.E., Minneapolis, MN 55455).

(July 13-17)—International Symposium on Surface Phenomena and Latexes in Water-Based Coatings and Printing Technology. Sponsored by Fine Particle Society. Riviera Hotel, Las Vegas, NV. (M.S. El-Aasser, Emulsion Polymers Institute, 111 Research Dr., Iacocca Hall, Lehigh University, Bethlehem, PA 18015).

(July 22-24)—"Basic Coatings for Sales and Marketing Personnel." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. Airport Marriott Hotel, St. Louis, MO. (Norma Fleming, Sr. Coordinator, UMR, Continuing Education, 119 M.E. Annex, Rolla, MO 65401-0249).

(Aug. 3-7)—"Coatings Science for Coatings Technicians." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Deborah A. Theisen, USM, Dept. of Polymer Science, Southern Station, P.O. Box 10076, Hattiesburg, MS 39406-0076).

(Aug. 10-14)—"Coatings Science for Coatings Chemists." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Deborah A. Theisen, USM, Dept. of Polymer Science, Southern Station, P.O. Box 10076, Hattiesburg, MS 39406-0076).

(Aug. 16-21)—"Polymer Chemistry: Principles and Practice." Seminar sponsored by The American Chemical Society (ACS), Washington, D.C. Marriott Inn, Blacksburg, VA. (ACS, Dept. of Continuing Education, Meeting Code VPI9203, 1155 Sixteenth St., N.W., Washington, D.C. 20036).

(Aug. 17-21)—"Formulating Coatings." Short course sponsored by The University of Southern Mississippi (USM), Hattiesburg, MS. (Deborah Theisen, PSC Coatings Short Course, USM, Southern Station Box 10076, Hattiesburg, MS 39406-0076).

(Sept. 14-18)—65th Introductory Short Course, "The Basic Composition of Coatings." Sponsored by University of Missouri-Rolla (UMR), Rolla, MO. Rolla Campus. (Norma Fleming, Sr. Coordinator, UMR, Continuing Education, 119 M.E. Annex, Rolla, MO 65401-0249).

(Sept. 28-Oct. 2)—25th Introductory Short Course, "Paint Formulation." Sponsored by University of Missouri-Rolla (UMR), Rolla, MO. Rolla Campus. (Norma Fleming, Sr. Coordinator, UMR, Continuing Education, 119 M.E. Annex, Rolla, MO 65401-0249).

(Oct. 5-8)—"Introduction to Coatings Technology." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education-Chemistry, KSU, P.O. Box 5109, Kent, OH 44242-0001).

(Oct. 6-8)—Powder Coating '92. Sponsored by the Powder Coating Institute (PCI). Cincinnati Convention Center, Cincinnati, OH. (PCI Headquarters, 1800 Diagonal Rd., Suite 370, Alexandria, VA).

(Oct. 14-16)—"Accelerated and Natural Weathering Techniques for Coatings and Polymers." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education-Chemistry, KSU, P.O. Box 5109, Kent, OH 44242-0001).

(Oct. 19-21)—105th Annual Meeting of the National Paint and Coatings Association (NPCA). Palmer House, Chicago, IL. (NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005).

(Oct. 25-30)—Fourth Corrosion and Protection Iberoamerican Congress and First Panamerican Congress on Corrosion and Protection. Mar del Plata, Argentina. (CIDEPINT, 52 entre 121 y 122, 1900 La Plata, Argentina, South America).

(Nov. 4-6)—'92 International Conference on Colour Materials. Sponsored by the Japan Society of Colour Material. Osaka Sun Palace, Expo Park Senri, Osaka, Japan. (S. Tochihara, Chairman of Executive Committee of the '92 ICCM, c/o Japan Society of Colour

Material, Kitamura Bldg. 5F, 9-12, 2-chome, Iwamoto-cho, Chiyoda-ku, Tokyo 101, Japan).

(Nov. 8-12)—Annual Conference 32. Sponsored by the Australasian Corrosion Association Inc., Hobart, Tasmania. (Conference Secretariat, Australasian Corrosion Centre, P.O. Box 250, Clayton, Victoria 3168, Australia).

(Nov. 15-20)—SSPC '92 National Conference and Exhibition. Sponsored by Steel Structures Painting Council (SSPC), Pittsburgh, PA. Kansas City Convention Center, Kansas City, MO. (Rose Mary Sargent, Manager of Conferences and Exhibits, SSPC, 4400 Fifth Ave., Pittsburgh, PA 15213-2683).

(Nov. 16-20)—"Fundamentals of Chromatographic Analysis." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education-Chemistry, KSU, P.O. Box 5109, Kent, OH 44242-0001).

(Dec. 6-11)—"Polymer Chemistry: Principles and Practice." Seminar sponsored by The American Chemical Society (ACS), Washington, D.C. Marriott Inn, Blacksburg, VA. (ACS, Dept. of Continuing Education, Meeting Code VPI9203, 1155 Sixteenth St., N.W., Washington, D.C. 20036).

## 1993

(Jan. 19-20)—"Accelerated and Outdoor Durability Testing." Symposium sponsored by ASTM Committee G-3 and Subcommittee D01.27. Ft. Lauderdale, FL. (Warren D. Ketola, 3M Co., Bldg. 553-A, 3M Center, St. Paul, MN 55144, or Douglas Grossman, The Q-Panel Co., 26200 First St., Cleveland, OH 44145).

(Feb. 21-26)—16th Annual Meeting of The Adhesion Society, Inc. Williamsburg Lodge, Colonial Williamsburg, VA. (Louis H. Sharpe, 28 Red Maple Rd., Hilton Head Island, SC 29928, or F.J. Boerio, Dept. of Materials Science and Engineering [ML 12], University of Cincinnati, Cincinnati, OH 45221).

(Apr. 18-23)—"Durability of Coatings" Symposium sponsored by American Chemical Society, Division of Polymeric Materials: Science Engineering, Denver, CO. (Jonathan W. Martin, NIST, Bldg. 226, Rm. B348, Gaithersburg, MD 20879; David Bauer, Ford Motor Co., SRL-E3198, P.O. Box 2053, Dearborn, MI 48121; F. Louis Floyd, Glidden Research Ctr., 16651 Sprague Rd., Strongsville, OH 44136).

(Apr. 20-22)—Surface Treatment '93. "Computer Methods and Experimental Measurements for Surface Treatment Effects." International Conference sponsored by Wessex Institute of Technology. Novotel, Southampton, United Kingdom. (Sue Owen, Conference Secretariat, Wessex Institute of Technology, Ashurst, Southampton, Hants, United Kingdom So4 2AA).

(May 2-6)—RadTech Europe '93. Third Annual RadTech conference. Sponsored by RadTech Europe. Italian vessel T/S Eugenio Costa. (RadTech Europe, Business Office, Péroles 24, CH-1700 Fribourg, Switzerland).

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## 'Humbug' from Hillman

I was very pleased to receive an encouraging note from David Heath, of ICI Paints, of Birmingham, England. Enclosed were two pages of comments from *Executive Travel* written by Perrot Phillips, which should supply, in bits and pieces, some wry humor for this and columns to come. We'll begin with:

• Resplendent in all his canonicals, the Bishop of Lincoln, the Rt. Rev. Robert Hardy stopped at a Little Chef on the way to an engagement. "Ooh," said the waitress, "what a smart purple shirt. Are you gay?" "Er, no, actually," the cleric replied, "I'm a Bishop." Delighted to have such an important customer, the waitress produced a paper napkin and asked for his autograph. The Bishop completed it with the sign of a cross. The waitress squealed in delight, "Kisses, too!"

• Why United Airlines should be singled out for poor in-flight meals beats me (Mr. Phillips). But *The Guardian* reported that a stewardess on a flight from New York to Chicago announced the dinner service, then added: "We shall be holding a competition for the passenger who can guess what he ate."

A few days later, the *Daily Telegraph* went one better. The steward informed passengers there was a lunchtime choice of beef stroganoff or chicken. "In the event that we are unable to serve you your preference," he went on, "United has ensured that both taste the same."

• Just as he saw his suitcase vanish into the X-ray machine at Tangle Airport, a *Times* journalist suddenly remembered he had packed two antique pistols inside. Terrified of the reaction, he dashed to the operator and blurted out his mistake. "Don't worry, sir," came the reply, "our machines will never pick them up."

Owen Carpenter writes that his wife, Helen, found the following in her hometown *Adirondack Echo*, of Old Forge, NY. It is a reprint from a 1924 *Benson* (Neb.) *Times*.

My father says the paper he reads ain't put up right.  
He finds a lot of fault, too, perusin' it all night.  
He says there ain't a single thing in it worth to read,  
And that it doesn't print the kind of stuff the people need.  
He tosses it aside and says it's strictly on the bum.  
But you ought to hear him holler when the paper doesn't come.

He reads about the weddin's and he snorts like all get out.  
He reads the social doin's with a most derisive shout.  
He says they make the papers for the women folks alone.  
He'll read about the parties and he'll fume and fret and groan.  
He says, of information, it doesn't have a crumb,  
But you ought to hear him holler when the paper doesn't come.

He is the first one to grab it and reads it plumb clean through.  
He doesn't miss an item, or want ad—that is true.  
He says they don't know what we want, the darn newspaper guys.

"I'm going to take a day sometime and go and put 'em wise;  
Sometimes it seems as though they must be deaf, blind and dumb."

But you ought to hear him holler when the paper doesn't come.

Many thanks to Ivan "Quack" Quackenbush for finding this excerpt from the "Court Jester" by Peter V. Macdonald in the *Toronto Star*—a story to remember for your next customer lunch.

On a bitterly cold winter's day in northern British Columbia, an RCMP constable on patrol came across a motorcyclist, who was swathed in protective clothing and helmet, stalled by the roadside.

"What's the matter?" asked the policeman.

"Carburetor's frozen," came the terse reply.

"Pee on it. That'll thaw it out."

"Can't."

"Okay, I will."

The constable lubricated the carburetor as promised. The bike started; and off went the rider, waving.

A few days later, the detachment received a note of thanks from the father of the cyclist. It began:

"On behalf of my daughter, who recently was stranded . . ."

Humbug feels that you've had enough time to recover from our bold decision to publish some of Bob Ahlf's wise (?) words of comfort for the forlorn. Here's one more try, courtesy of Tom Miranda, at helping you to give up hope.

—Choose one: (1) Old and Rich — (2) Old and Poor.

—This isn't really happening. You'll wake up in a minute, sweating.

—More banks are robbed from the other side of the teller's cage.

—Hard work and education will get you more studying and more hard work.

—Congress doesn't give you any money you didn't give them.

—Elvis is alive and drunk in a bar in downtown East St. Louis.

—Sometimes when the brain doesn't have a problem, it invents one.

—It is possible to be so ignorant that you don't know you're ignorant.

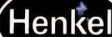

Sid Lauren confesses: At the age of 8, I had a confrontation with my mother on the issue of cooked oatmeal. I said I wouldn't eat it because I hated it. She said I ought to eat it because it was good for me. My response was, "If it's so good for people, why don't you eat it?" . . . She answered simply, "Because I don't like it!"

Recently, he bowed to the anti-cholesterol propaganda and started eating oatmeal. He goes on to say, "I discovered a conspicuous violation of the Law of the Conservation of Matter. After carefully measuring out the package recipe for a single serving, I discovered the following reaction, expressed volumetrically: 1/3 cup Oats + 2/3 cup Water = 2 cups Mush. Now, I have to persuade myself that I like it!"

Don't complain Sid, in these days of recession, you're getting more for your money. So—like it, like it!!

—Herb Hillman  
Humbug's Nest  
P.O. Box 135  
Whitingham, VT 05361



The Henkel logo is located in the top right corner, consisting of the word "Henkel" in a white, sans-serif font inside a white oval.A rectangular white sample showing significant surface mildew, appearing as numerous small, dark, irregular spots and patches.

Problem: Mildew

A rectangular white sample showing a clean, smooth surface, representing the result of applying the solution.

Solution:  
Nopccocide® Mildewcides

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One additive protects the paint on the right against mildew, and still helps you stay on the right side of the environment. It's Henkel's Nopccocide®. It prevents fungal growth for many years under the most adverse conditions of heat and humidity. And doesn't contain mercury.

Nopccocide®, based on the active ingredient chlorothalonil, is just one of the paint additives offered by Henkel that are ecologically compatible. We offer the most extensive line of additives that help the paint and coatings industry manufacture better coatings of all kinds.

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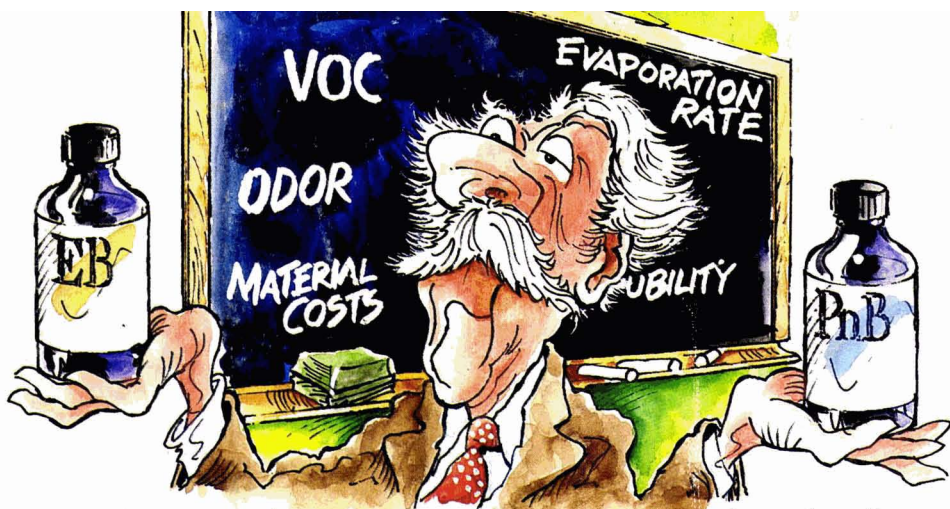
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## Considering Alternatives to E-Series Glycol Ethers? DOWANOL PnB and DPnB Reduce Handling Concerns and Deliver Comparable Solvent Performance.

There are no such things as drop-in replacements for familiar E-series glycol ethers. However, DOWANOL\* PnB and DPnB

P-series glycol ethers can offer surprisingly similar performance in both industrial and architectural formulations.

### DOWANOL PnB: Fast Hardness Development Like EB in Industrial Formulations

When you want fast evaporation, DOWANOL PnB (propylene glycol n-butyl ether) performs almost identically to EB glycol ether. The efficiencies of PnB and EB in lowering minimum film formation temperatures (MFFT's) are in the same range. And as the example in Figure 1 shows, their hardness development rates in typical industrial latexes are very similar.

Also, blends of DOWANOL PnB with other P-series glycol ethers and/or C<sub>4</sub> alcohols yield organic phase coupling behavior comparable to EB.

### DOWANOL DPnB: High Efficiency, Easier VOC Compliance, and Lower Odor

DOWANOL DPnB (dipropylene glycol n-butyl ether) is a good alternative to DB glycol ether. While their evaporation rates are similar, DOWANOL DPnB can offer better MFFT-lowering efficiency

than DB (Figure 2). And since DOWANOL DPnB has much lower water solubility, it's less likely to cause compatibility

problems with associative thickeners.

In addition, DOWANOL DPnB often provides greater coalescing efficiency than popular ester-alcohols in acrylic-based architectural latex formulations. So for about the same formulated cost, you can reduce both VOC's and odor levels.

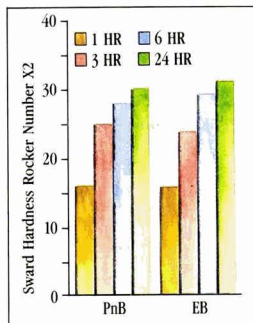


Figure 1 – Hardness Development Rates in Typical Acrylic-Based Latex

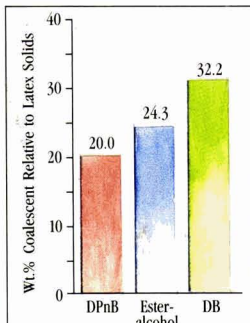


Figure 2 – Coalescent Levels Required for 50°F MFFT in Typical Acrylic-Based Latex

### Answers to Reformulating Questions

With our extensive reformulating database, we can help make your switch to P-series alternatives easier. If you need to fine-tune solvent performance to address specific temperature and humidity considerations, we can offer unmatched assistance.

### Call Today For Literature and Samples

It's easy to learn more about DOWANOL PnB or DPnB and how they may be ideal alternatives in your current formulations. Our 20-page brochure provides comprehensive information, and we'll be glad to provide samples for your evaluation.

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