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**Technical Articles** 

23 Correlation of Thermal History and Performance in Thermosetting Powder Coatings-K.G. Panjnani,

S.D. Pace, and S.F. Thames This investigation, dealing with critical aspects of powder coatings, will benefit both suppliers and end users.

- 29 Depth Profiling of Acrylic/Melamine Formaldehyde Coatings—G. Haacke, J.S. Brinen, and P.J. Larkin New information on depth dependence of properties of acrylic/melamine formaldehyde coatings is emphasized.
- Interpretation of Corrosion Resistance Properties of Organic Paint Films from Fractal Analysis of 35 Electrochemical Noise Data—M. Moon and B. Skerry

In the paint industry, accelerated laboratory tests are commonly used to assess the corrosion resistance properties of organic coatings. An important new methodology is presented which offers the possibility of interpreting corrosion resistance properties based on electrochemical noise measurements.

Effects of Coating Solvents on the Morphology of Thermoplastic Polyolefins (TPO)-R.A. Ryntz, Q. Xie, 45 and A.C. Ramamurthy

> Focus is on coating of plastics, specifically thermoplastic polyolefins. The wide range of methods presented is applicable to coating other plastic substrates.

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THE JOURNAL OF COATINGS TECHNOLOGY (ISSN 0361-8773) is published monthly by the Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422-2350, Phone (610) 940-0777; Fax: (610) 940-0292. Second class postage paid at Blue Bell, PA and at additional mailing offices. POSTMASTER: Send address changes to JOURNAL OF COATINGS TECHNOLOGY, 492 Norristown Rd., Blue Bell, PA 19422-2350.

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

	U.S. and Canada	Europe (Air Mail)	Other Countries
1 Year	\$30.00	\$60.00	\$45.00
2 Years	\$57.00	\$117.00	\$87.00
3 Years	\$82.00	\$172.00	\$127.00

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

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

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Recently, the FSCT introduced an updated version of its leading industry publication—the JOURNAL OF COATINGS TECHNOLOGY. Some say it's "all in the packaging," but the changes in the JCT reflect a philosophical shift that goes beyond its bolder covers, increased use of color, and fresh style.

Before any changes were made to the Journal, a very select group was consulted—our members! From them we learned that the JCT continues to maintain a competitive edge as the primary source of technical information for the industry. The only peer reviewed journal in North America, the JCT has first rights to papers presented at leading industry events, such as the FSCT Annual Meeting, Waterborne Symposium, Western Coatings Societies Biennial Symposium, and all programs sponsored by Constituent Societies. As part of its change, the JCT will broaden its focus to include more articles on new and emerging technologies, and increased emphasis on areas such as powder coatings, radiation curing, adhesives, and related technologies.

Our members want more. More practical information that will help them deal effectively with challenges posed by increased competition and growing regulatory demands. More problem-solving techniques that can be put to immediate use to improve products and services. The new "Features" department will address this need. Watch for future issues for articles dealing with such topics as manufacturing, statistical design of experiments, quality control, etc.

The JCT is also meeting readers' needs by increasing coverage of the technical conferences and symposia offered by industry, educational institutions, and local Constituent Societies. The "Meetings Update" department is now positioned in the front of the JCT and the "Calendar of Events" presents the most comprehensive listing of the important events for the industry.

We need more. More feedback on the kinds of articles and information you want to see. Responses to the changes we've made so far have been very positive and we appreciate the phone calls, letters, and faxes. Keep them coming!

We're not done yet. Just as the Federation is moving to better address a changing industry, the JCT will continue to evolve, responding to the diverse needs of FSCT members and the industry.

> Patricia D. Viola Editor

ห้องถมุตกรบวิทยาศาสตร์**บริกาว** 

Translations provided by: French—Montreal Society Member Mario Côte of Eastman Chemical Canada Inc.-Montreal; Spanish—Beatriz Alonso Torres, of Instituto Mexicano de Tecnicos en Pinturas y Tintas.

Correlation of Thermal History and Performance in Thermosetting Powder Coatings—K.G. Panjnani, S.D. Pace, and S.F. Thames

JCT, 67, No. 843, 23 (Apr. 1995)

Corrélation de l'Histoire Thermique et de la Performance Dans les Revêtements en Poudre—G. Panjnani, S.D. Pace, et. S.F. Thames

Correlación de Actividad Térmica y Comportamiento en Termodeposición de Recubrimientos en Polvo—K.G. Panjnani, S.D. Pace, y S.F. Thames The heat history of thermosetting powder coatings necessitates careful control of extrusion conditions. It is common industrial practice to minimize pre-reactions by rapidly cooling powder extrudates. To some degree, the quenching process controls the formation of internal stress in the finished product. Similar stress development occurs when powder coated films are cured and cooled rapidly.

This study examines polyester-blocked isocyanate, epoxy-dicyandiamide, polyester-triglycidyl isocyanurate, epoxy-phenolic, and acrylic thermosetting powder coatings via differential scanning calorimetry to quantify annealing and quenching effects on the glass transition temperatures. As a part of this study, panels were coated and cured at 150°C for 30 min. Cooling was effected by either rapid quenching or controlled annealing. Thermal, mechanical, and X-ray diffraction analysis data are presented which corroborate a thermal history dependence on powder coating performance.

L'histoire de chaleur de revêtements en poudre thermoset nécessite un contrôle soigneux des conditions d'extrusions. Il est pratique industrielle courante de minimiser les pré-réactions en refroidissant rapidement les extrudats en poudre. Le procédé "quenching" de formation contrôle, jusq'à un certain degré, la formation de stress interne dans le produit fini. Un développement similaire de stress survient lorsque des films de revêtements en poudre sont durcis et refroidis rapidement.

Cette étude examine les revêtements en poudre polyester-bloqué isocyanate, époxiedicyandiamide, polyester-triglycidyl isocyanurate, époxie-phénolique et acrylique thermosets à l'aide de la calorimétrie à balayage différentiel pour quantifier les effets "quenching" et "annealing" sur les températures de transition vitreuses. Faisant partie de cette étude, des panneaux furent recouverts et durcis à 150°C pour 30 minutes. Le refroidissement fut effectué par un "quenching" rapide ou par un procédé contrôlé "annealing." Des données d'analyses thermiques, mécaniques, et par diffraction aux rayons X sont présentées qui corrobore une histoire thermique de dépendance sur la performance d'un revêtement en poudre.

El proceso de calentamiento en deposición térmica de recubrimientos necesita un control cuidadoso de las condiciones de extrusión. Comúnmente en la industria se practica minimizar las pre-reacciones enfriando rápidamente los extrudados en polvo. El proceso de enfriamiento controla en algún grado, la formación de esfuerzo internos en el producto terminal. Cuando las películas de recubrimientos en polvo se curan y enfrían rápidamente, se incurre en un efecto similar.

Este estudio examina la termodeposición de recubrimientos en polvo como el isocianto bloqueado con poliéster, la epoxi-dicianidiamida, el isocianurato de triglicidi-poliéster y el fenoli-epóxico via un calorimetro diferencial para cuantificar las efectos de enfriamiento y templado en los temperaturas de transición cristalina. Como parte de este estudio, se recubrieron y curaron panels at 150°C por 30 min. Se observó el efecto de enfriamiento al hacerlo rápidamente o al controlar el templado. Se presentan datos de análisis térmicos, mecánicos y de defracción de rayos X, que corroboran una dependencia térmica en el desarrollo de recubrimientos en polvo.

#### Depth Profiling of Acrylic/ Melamine Formaldehyde Coatings—G. Haacke, J.S. Brinen, and P.J. Larkin

JCT, 67, No. 843, 29 (Apr. 1995)

The depth dependences of the glass transition temperature, the crosslink density, and the triazine/styrene and nitragen/carbon ratios have been investigated in acrylic/melamine coatings. Depth profiles were determined by using a microtome technique which produced slices large enough (5 x 5 cm) to employ as analytical tools dynamic mechanical analysis, differential scanning calorimetry, and elemental analysis. The triazine/styrene ratios were derived from infrared absorption spectra. Depending on the cure conditions, gradients perpendicular to the coating surface were observed for the glass transition temperature, the crosslink density, and the triazine/styrene infrared absorption ratios of the crosslink density, and the triazine/styrene infrared absorbance ratio. For 30 min cure time, these gradients existed in coatings cure below 160°C. The nitrogen/carbon ratios of the gradyed constant in samples in which the other parameters investigated changed with depth. The gradients of the glass transition temperature and crosslink density are believed to originate from a shift of the chemical reaction equilibrium of the crosslink ing reaction. Trapped isobutanol, a reaction by-product, is assumed to be responsible for this shift.

Profilage d'Intensité de Revêtements Acrylique/Mélamine Formaldéhyde—G. Haake, J.S. Brinen, et P.J. Larkin Les dépendances d'intensité de la température de transition vitreuse, de la densité de réticulation, et des rapports triazine/styréne et azote/carbone ont été étudié dans les revêtements acrylique/mélamine. Des profiles d'intensité furent déterminé en utilisant une technique employant un microtome qui produisit des tranches assez épaisses (5 x 5 cm) pour



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employer comme outils analytiques, l'analyse dynamique mécanique, la calorimétrie à balayage différentiel et l'analyse élémentaire. Les rapports triazine/styrène furent dérivés de spectres d'absorption infrarouge. Dépendant des conditions de durcissement, des gradients perpendiculaires à la surface du revêtement furent observés pour la température de transition vitreuse, la densité de réticulation et le rapport d'absorbance infrarouge triazine/styrène. Pour un temps de durcissement de 30 minutes, ces gradients existèrent dans des revêtements durcis sous 160°C. Les rapports azote/carbone des résines réticulées, déterminés à partir d'analyse élémentaire et de spectroscopie photoélectron rayons X, demeurèrent constant dans les échantillons auxquels les autres paramètres étudiés changèrent avec l'intensité. L'on croit que les gradients de la température de transition vitreuse et la densité de réticulation. De l'isobutanol changement de l'équilibre de réaction, est présumé être responsable pour ce changement.

Perfilamiento Profundo de Recubrimientos de Formaldehído Acrílico/Melamina—G. Haacke, J.S. Brinen, y P.J. Larkin Se ha investigado la dependencia de la profundidad con la temperatura de transición, la densidad de entrecuzamiento y las relaciones de triazina/estireno y nitrógeno/carbón en recubrimientos de acrílico/melamina. Los perfiles de profundidad se determinaron usando un técnica de microtomia que produce películas lo suficientemente grandes [5 x 5 cm] para utilizarlos como herramientas analíticas en análisis mecánicos dinámicos, calorimetría diferencial y análisis elemental. Los razones de triazina/estireno se derivaron de espectros de absorción infrarroja. Dependiendo de las condiciones de curado, se observaron gradientes perpendiculares a la superficie del recubrimiento en los radios de absorción infrarrojos de la temperatura de transición cristalina, la densidad de entrecruzamiento y la razón triazina/ estireno. Para tiempo de curado de 30 min, estos gradientes existieron en recubrimientos curados por debajo de los 160°C. Las razones nitrógeno/carbono de las resinas entrecruzadas, determinadas a partir de análisis elemental y espectroscopia fotoelectrónica de rayos-X, permanecieron constantes en muestras en las que los otros parámetros investigados cambiaron con la profundidad. Los gradientes de temperatura de transición cristalina y densidad de entrecruzamiento se creé que se originan por una desviación del equilibrio de la reacción química entrecuzamiento. La reacción colateral que produce isobutanol, aparece como la responsable de este desvio.

Interpretation of Corrosion Resistance Properties of Organic Paint Films From Fractal Analysis of Electrochemical Noise Data— M. Moon and B. Skerry

JCT, 67, No. 843, 35 (Apr. 1995)

Interprétation de Propriétés de Résistance à la Corrosion de Films de Peinture Organique à l'Aide d'Analyses Fractales de Données de Bruit Électrochimique—M. Moon et B. Skerry Time records of electrochemical noise (ECN), i.e., spontaneous voltage and current fluctuations, were collected while coated metal panels were immersed in an electrolyte solution. After 2,000 hr, the panels were removed and the blistering and corrosion rated visually. In order to evaluate coating performance during the course of the experiment, the time records were analyzed. While visual and statistical analyses (especially the "derived noise resistance") were valuable for comparing the performance of the coatings, they did not provide information regarding mechanisms of corrosion, inhibition, and coating degradation. The frequency structure of the ECN time records was, therefore, analyzed with fast Fourier transforms. However, the results obtained for these relatively small data sets (64 observations per time record) were too limited to allow a meaningful comparison to be made. Because the voltage and current data resembled time records that are known to be fractals, an established technique from fractal geometry was used to evaluate the Hurst exponent (H). The results ranged from  $2H \approx 0$  (coatings where little or no corrosion was observed) to  $2H \approx 2$  (coatings where extensive corrosion activity was evident). The Hurst exponent can be used as an index of the level of corrosion resistance provided by a coating. In addition, H can be interpreted in terms of the color and persistence characteristics of the time records. This interpretation forms the basis of a description of the electrochemical processes which give rise to electrochemical noise

Des registres de temps de bruit électrochimique (ECN), c'est-à-dire voltage spontané et fluctuations de courant furent collectionnés pendant que des panneaux de métal revêtus furent immergés dans une solution électrolytique. Après 2,000 heures, les panneaux furent retirés et la corrosion de même que les cloques évaluées visuellement. De façon à évaluer la performance d'un revêtement pendant l'expérience, les registres de temps furent analysés. Tandis que des analyses visuelles et statistiques (spécialement la "résistance au bruit dérivé") furent valables pour comparer la performance des revêtements, ils n'ont pas procuré de l'information au sujet des mécanismes de corrosion, inhibition, et dégradation de revêtement. La structure de fréquence des registres de temps ECN fut par conséquent analysée à l'aide de transformations de Fourier. Cependant, les résultats obtenus pour ces ensembles relativement petits de données (64 observations par registre de temps) furent trop limités pour permettre à une comparaison significative d'être faite. Parce que les données de voltage et de courant ressemblent à des registres de temps connus sous le nom de fractals, une technique établie de géométrie fractal fut utilisée pour évaluer l'exposant d'Hurst (H). Les résultats ont variés de 2H ≈ 0 (revêtements où peu ou aucune corrosion ne fut observée) jusqu'à 2H = 2 (revêtements où une activité de corrosion élaborée fut évidente). L'exposant d'Hurst peut être utilisé comme un index du niveau de résistance à la corrosion fournit par un revêtement. De même, H peut être interprété comme les caractéristiques de "couleur" et "persistance" des registres de temps. Cette interprétation forme la base d'une description des procédés électrochimiques donnant lieu au bruit électrochimique.

Interpretación de Propiedades de Resistencia a la Corrosión en Películas de Pintura Orgánica a Partir de Datos de Análisis Fractal de Ruido Electroquímico— M. Moon y B. Skerry

Se registraron datos de ruido electroquímico (ECN), es decir, voltaje espantáneo y fluctuaciones mientras paneles de metales recubiertos se sumergían en una solución electrolitica. Después de 2,000 horas, los paneles fueron removidos, y se midieron visualmente las protuberancias y la corrosión. Para evaluar el comportamiento del recubrimiento en el curso del experimento, se analizaron los registros de tiempo. Aunque los análisis visuales estadísticos (especialmente el de "resistencia derivada al ruido") sirvieron para comparar el comportamiento de los recubrimientos, no pudieron proveer información acerca de los mecanismos de corrosión, inhibición y degradación. La estructura más frecuente de los registros de tiempo de ECN se analizó con transformadas de Fourier. Sin embargo, los resultados obtenidos para este relativamente pequeño grupo de datos (64 observaciones por registro de tiempo) fueron muy limitados para permitir hacer una comparación significativa. Debido a que el voltaje y los datos reflejaron registros de tiempo que se conocen como fractales, se usó una técnica establecida para geometría fractal al evaluar el exponente de Hurst (H). Los resultados variaron de 2H  $\approx$  0 (no se observó corrosión) basta 2H  $\approx$  2 (era evidente la actividad corrosiva). El exponente de Hurst puede usarse como índice del nivel de resistencia a la corrosión ofrecida por un recubrimiento. Además, H puede interpretarse en términos de las características de 'color" y "persistencia" de los registros de tiempo. Esta interpretación forma la base de la descripción de los procesos que aumentan el ruido electroquímico.

Effects of Coating Solvents on the Morphology of Thermoplastic Polyolefins (TPO)— R.A. Ryntz, Q. Xie, and A.C. Ramamurthy

JCT, 67, No. 843, 45 (Apr. 1995)

Effets de Solvants de Revêtements sur la Morphologie de Polyoléfines Thermoplastiques (OTP)—R.A Ryntz, Q. Xio, et A.C. Ramamurthy

Effectos de los Solventes en la Morfológia de Poliolefinas Termoplásticas (TPO)—R.A. Ryntz, Q. Xie, y A.C. Ramamurthy Adhesion to thermoplastic olefin (TPO) substrates is strongly influenced by the type and amount of solvent contained within paint applied. Morphological changes in the TPO substrate are accomplished in the presence of solvent from the coating and vary depending on paint bake times and temperatures. Amorphous droplets at the TPO surface, which appear to form on heating of the substrate, are shown to be the cause for paint dewetting. These morphological changes at and near the surface of TPO affect not only the paint adhesion to the substrate but also the cohesive integrity of the painted plastic composite. This paper attempts to delineate the influence of paint and paint processes on the adhesion/cohesion and mechanical properties of coated TPO parts. Initial adhesion to TPO appears to be a function of polypropylene crystallinity at the surface only as it relates to the ability of solvents to permeate through and into the rubberrich layer beneath it. The amount of swelling of the rubber-rich layer can result in a lowered cohesive integrity of the top TPO layer. One must carefully select the correct solvent balance contained within the paints applied to TPO. It is essential to attain swelling within the substrate to afford initial paint adhesion, but the solvent must evaporate and the rubber shrink (deswell) to attain cohesive integrity of the substrate once painted.

L'adhésion à des substrats oléfines thermoplastiques (OTP) est fortement influencé par la sorte et la quantité de solvant contenue dans la peinture appliquée. Des changements morphologiques dans de substrat OTP sont accomplis en présence du solvant du revêtement et varient en fonction des temps et température de cuisson de la peinture. Des gouttelettes amorphes à la surface OTP, qui semblent se former lors du chauffage du substrat, sont montrés être la cause du "démouillage" de la peinture. Ces changements morphologiques à et près de la surface OTP affectent non seulement l'adhésion de la peinture au substrat mais aussi l'intégrité cohésive du plastique composite peinturé. Cette étude tente de délinéer l'influence de la peinture et des procédés de peinture sur l'adhésion/cohésion et propriétés mécaniques d'éléments OTP revêtus. L'adhésion initiale à l'OTP apparaît être une fonction de la cristallinité du polypropylène à la surface seulement comme la relation à l'habilité des solvants d'être perméable à travers et à l'intérieur des couches riches en caoutchouc sous elle. Le degré de gonflement de la couche riche en caoutchou peut résulter en une intégrité cohésive diminuée de la couche OTP de la surface. On doit attentivement sélectionner le mélange de solvants adéquat contenu à l'intérieur des peintures appliquées à l'OTP. Il est essentiel d'obtenir le gonflement à même le substrat pour obtenir une adhésion initiale de peinture, mais le solvant doit s'évaporer et le caoutchouc rétrécir (dégonfler) pour obtenir l'intégrité cohésive du substrat une fois peinturé.

La adhesión a sustratos de olefinas termoplásticas (TPO) es fuertemente influenciada por el tipo y la cantidad de solvente contenido cuando se aplica una pintura. Se desarrollan cambios morfológicos en el sustrato TPO en presencia del solvente del recubrimiento y varían dependiendo de las veces que se hornee la pintura y las temperatura. Las gotas amorfas en la superficie de la TPO, que aparecen cuando el sustrato se calienta, parecen ser la causa del desgaste de la pintura. Estos cambios morfológicos en y cerca de la superficie de la TPO, afectan no solo adhesión de la pintura del sustrato, sino además la integración cohesiva del compuesto plástico pintado. En este artículo se delinea la influencia de la pintura y procesos de pintado en la adhesión/cohesión y propiedades mecánicas de partes de TPO recubiertas. La adhesión inicial a la TPO parece ser fución de la cristalinidad del polipropileno solamente en la superficie, y que se relaciona a la habilidad de los solventes para permecibilizar a través y hacia la capa rica en componentes elásticos. La cantidad de deformación de la capa elástica puede conllevar a una disminución de la integridad cohesiva de la capa superior de la TPO. Uno debe seleccionar cuidadosamente al balance correcto de solventes en las pinturas que se aplican a la TPO. Es esencial alcanzar la dilatación exacta del sustrato para asegurar la adhesión inicial de la pintura, pero el solvente debe evaporarse y el elástico encogerse (antideformación) para alcanzar la integridad cohesiva del sustrato una vez pintado.

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## New FSCT Seminar Focuses Attention on Polymer Chemistry for Formulators

A newly created seminar, sponsored by the Professional Development Committee of the Federation of Societies for Coatings Technology, will be offered on "Polymer Chemistry for the Coatings Formulator." To be held Tuesday-Wednesday, June 20-21, at the Chicago Marriott—Schaumburg, Schaumburg, IL, the seminar is designed to provide up-todate information on all aspects of polymer chemistry. Engineers, chemists, technicians, and technical service individuals involved in the coatings, inks, and adhesives arenas will benefit from this program.

The seminar speakers will offer information on basic polymer types utilized in the coatings industry, describing basic polymer makeup, reasons for selecting particular chemistries, and ways to achieve desired properties through resin selection. The attendee will also receive a broad overview of various testing methods used to determine film properties, including thermal and mechanical test methods and hardness testing. In addition, three "Problem Solving" sessions where attendees can pose questions to the speakers in a forum-type setting are scheduled.

Topics to be covered include:

• "Condensation"—Fritz Walker, Air Products & Chemicals, Allentown, PA

"Addition/Polymerization"—Dave
Nordstrom, DuPont Automotive, Troy, MI

#### Peso's Fall Causes Cancellation of FSCT Spring Seminar

The Federation regrets to announce the cancellation of its Spring Seminar, "Coatings in the Americas: Regulatory and Technological Issues," which was to be held on May 17-18, 1995, in Cancun, Mexico.

In making the announcement, Robert F. Ziegler, FSCT Executive Vice President, noted that the cancellation was the result of the recent devaluation of the peso. "It is extremely regrettable that due to the current economic situation, we have found it necessary to cancel this very worthwhile program," he said. "We look forward to continuing our good relationship with our Latin American members at the earliest opportunity," he added.

The Federation will hold its Board of Directors Meeting and Incoming Society Officers Meeting on May 20 and 21, respectively, at the Fiesta Americana Coral Beach Hotel, in Cancun. • "Polyester/Alkyd"—Ray Mooney, McWhorter, Minneapolis, MN

• "Phenolics"—Kevin McGuinn, Georgia-Pacific, Naperville, IL

• "Diluents"—Marina Hoffman, AN-GUS Chemical, Buffalo Grove, IL

• "Vinyls"—Rocky Prior, Union Carbide, Cary, NC

 "Acrylics"—Patricia M. Lesko, Rohm and Haas Co., Spring House, PA

• "Urethanes"—Anthony Pajerski, Zeneca Resins, Wilmington, MA

• "Epoxy"—David Helfand, Ciba Geigy, Ardsley, NY

• "Melamines"—Nick Albrecht, Cytec, Inc., Stamford, CT

• "Isocyanates"—Ron Wojcik, Olin Chemicals, Cheshire, CT

• "Characterization Overview"—Cliff Schoff, PPG Industries, Inc., Allison Park, PA

• "Characterization Specifics"—Basil Gregorovich, DuPont, Philadelphia, PA

Registration fees are \$295 for FSCT members and \$395 for nonmembers. Included with the registration are luncheons, refreshment breaks, and reference materials.

Attendance is limited and registrations will be accommodated on a first-come, firstserved basis.

Requests for seminar hotel accommodations at the Chicago Marriott—Schaumburg must be made on the seminar registration form, available from the Federation. The guest room rate is \$112 for single or double occupancy. To guarantee room availability and rates, reservations must be made by May 29, 1995.

The hotel offers complimentary transportation to and from O'Hare Airport.

To register or to receive additional information, contact Michael Bell, Director of Educational Services, Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422-2350; (610) 940-0777.

## FSCT Holds Successful Clean Air Act Seminar

Sixty-five attendees learned of the most recent developments regarding compliance with the Clean Air Act at the FSCT seminar, "Formulating for the New Clean Air Act," held on March 21-22, 1995.

Sponsored by the Professional Development Committee of the FSCT, the two-day seminar emphasized the status of new and future regulations and described what these regulations mean. In addition, discussions focused on such areas as solvent selection, formulation, VOC content issues, and reactive diluents. Techniques and secrets of formulation, abatement technology, additives, and UV/EB curing solutions were among the topics also covered in the presentations. The successful program will be repeated

on November 6-7, in Denver, CO.

For more information, contact FSCT, 492 Norristown Rd., Blue Bell, PA 19422-2350.

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## NPCA v. City of Chicago Decision Reversed; Proposition 65 Challenged in California Court

statement released by the National Paint and Coatings Association (NPCA), Washington, D.C., revealed that Judge Aspen's 1993 decision in NPCA v. City of Chicago was reversed by the U.S. Court of Appeals for the Seventh Circuit; therefore, determining the Chicago city ordinance banning the retail sale of spray paint constitutional.

The ordinance, as passed by city council and signed by the Mayor of Chicago, provided the following: "It shall be unlawful for any person holding a retail business license to sell paint in spray cans to any person or to sell any marker containing a fluid which is not water-soluble and has a point, brush, applicator, or other writing surface of threeeights of an inch or greater to any person." The law, however, does not include a definition of the phrase "paint in spray cans." Consequently, any product typically considered to be spray paint is a banned product. This law applies to "persons holding a retail business license." Therefore, if a business entity in the City of Chicago does not hold a "retail business license," then the law does not apply.

The Seventh Circuit also rejected NPCA's request to prohibit the Chicago city government from enforcing the law while an appeal to the U.S. Supreme Court is initiated. Therefore, for those sellers that hold a retail license, it is currently illegal to sell spray paint within the city limits.

Judge Aspen's earlier decision voided the Chicago city ordinance. He found the ordinance to be in violation of the United States Constitution under the Commerce Clause and the principles of Substantive Due Process. In addition, Judge Aspen held that the ordinance exceeded the city's police powers granted under the Illinois Constitution. All of these findings were predicated on the overwhelming factual evidence which clearly demonstrated that the product ban ordinance would not reduce the tagging activity of graffiti vandals. He viewed the ordinance as irrational.

On the other hand, the Seventh Circuit opinion indicates that Judge Aspen was wrong to even conduct an evidentiary hearing in this case. In contrast to Judge Aspen's decision (which was primarily based upon the Commerce Clause analyses), the Seventh Circuit focused on the rational basis test—meaning that if there is any conceivable basis which might support the ordinance, it is acceptable. The Seventh Circuit disregarded all of Judge Aspen's findings, citing that reducing the availability of spray paint, raising its price, and/or increasing travel time and costs to procure spray paint could conceivably reduce graffiti vandalism. According to this Seventh Circuit panel, a legislature could rationally conclude that even the most committed taggers could change their behavior in the face of fines and other criminal punishments.

The Substantive Due Process claim was dismissed because, in this court's view, no fundamental right was involved. Moreover, the court's analysis under the Commerce Clause failed to recognize that where a local law only incidentally affects interstate commerce, there must still be some balancing act of the law's benefits and burdens. It was this balancing test that led Judge Aspen to conclude that the retail sales ban was irrational and unconstitutional.

NPCA filed an application for a stay of enforcement with the Supreme Court, requesting that the Supreme Court issue an order preventing the City of Chicago from enforcing the law during the final appeal.

If the request is granted, then enforcement of the law will be enjoined and retailers will be permitted to sell spray paint until the issue is finally determined by the Supreme Court. If the request is denied, the city may continue to enforce the activities.

#### 

In other legal news, an *amicus curiae* brief filed by NPCA, with the California Superior Court, urged the Court to find the state's Proposition 65 as applied to paint products containing toluene is preempted by the Federal Hazardous Substance Act (FHSA). Proposition 65 requires "point-ofsale" warnings for products containing "hazardous substances."

The brief was submitted by NPCA in support of a motion for summary judgment filed by Cotter and Co.

NPCA told the court that the practices of those pursuing enforcement of Proposition 65 have left no "actual alternative" to "affixing the Proposition 65 warning" to a container. Thus, "labeling on the product provides the only certain means of ensuring compliance." However, requiring a statemandated warning on the product label which is "in addition to" or "different from" that required by the FHSA places the Proposition in direct conflict with preemptive labeling requirements imposed under the FHSA, according to NPCA's brief.

In support of the position, NPCA cited a 1992 U.S. Supreme Court decision, Cipollone v. Liggett Group (Cipollone), which provides "the definitive standard regarding federal preemption of state regulations that address the same subject as federal law." It is clear, NPCA's brief points out, that the FHSA and Proposition 65 both address the same subject: warning labels on products containing hazardous substances. Applying the preemption standard set forth in Cipollone, Proposition 65's de facto labeling mandate is in direct conflict with the labeling required by the FHSA.

According to NPCA, the Cipollone decision supersedes an earlier, 1991 federal appeals court decision which upheld the validity of Proposition 65. The earlier case, Chemical Specialty Manufacturers Association v. Allenby, was, in NPCA's opinion, "wrongly decided" before enforcement had fully begun and did not address an industry structure that imposes a de facto labeling requirement, like that of the paint and coatings industry.

The brief concludes that under the express preemption provisions of the FHSA and the principles enunciated in *Cipollone*, the enforcement of Proposition 65 warning requirements for sale of paint and coatings products "should be deemed preempted."

#### Reichhold Purchases Coatings Manufacturer Resinas de Mexico from Celanese Mexicana

The Resinas de Mexico division of Celanese Mexicana S.A. has been purchased by Reichhold Chemicals Inc., Research Triangle Park, NC. The operations of Resinas de Mexico, a manufacturer of coating resins and unsaturated polyesters, will be merged with Reichhold's Mexican subsidiary, Reichhold Quimica de Mexico.

Under the terms of the agreement, Reichhold Quimica de Mexico will operate the Resinas de Mexico plant in Toluca, Mexico, a 13-acre site with manufacturing capacity of more than 50 million pounds. A total of 90 Resinas employees will join Reichhold. Financial arrangements of the purchase were not disclosed. The combined product lines of the two companies include alkyd, acrylic, polyester, amino, and emulsion resins. The acquisition consolidates the service of two leading suppliers to the coatings, construction, plastics, and adhesives markets in Mexico.

Reichhold currently operates a similar production facility in Vallejo.

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#### Minerals Technologies Expands Adams, MA, Facility; May Acquire Genstar's Calcium Carbonate Business

Specialty Minerals Inc. a wholly owned subsidiary of Minerals Technologies Inc., New York, NY, will invest \$18 million to expand its Adams, MA, facility. The company will increase production capacity for precipitated calcium carbonate (PCC) used in the plastics, sealants, and food industries.

The expansion, which is slated for completion by the third quarter of 1995, will allow Specialty Minerals to produce

## Arco Completes Transfer of TDI from Rhone-Poulenc, Inc.

Arco Chemical Co., Newtown Square, PA, has completed the transfer of the Rhone-Poulenc toluene di-isocyanate (TDI) activities to the company.

The arrangement calls for Arco Chemical to take over the marketing of TDI from Rhone-Poulenc, which will continue to manufacture the product at its sites at Pontde-Claix (Isère) and Lille, France. The supply of TDI from Rhone-Poulenc to Arco Chemical will be secured through long-term agreements between the two companies. PCC products of varying crystal shape and size. These PCC products are used as functional fillers, calcium sources, and neutralizing agents.

In other news, Minerals Technologies signed a letter of intent with Redland PLC of the United Kingdom for the acquisition of the fine-ground calcium carbonate operations of Genstar Stone Products Co., Hunt Valley, MD.

Genstar, a wholly owned subsidiary of Redland PLC, will continue to own and operate its limestone aggregate business. The acquisition is expected to be completed during the second quarter of 1995.

#### Macbeth and Akzo to Develop Spectrophotometer

Macbeth, a Division of Kollmorgen Instruments Corp., New Windsor, NY, and Akzo Nobel have signed a multimilion dollar contract designating Macbeth as Akzo's sole source for multi-angle spectrophotometers.

Macbeth will also serve as a preferred supplier to Akzo Nobel worldwide, for all other color and appearance instrumentation.

This multi-angle spectrophotometer applies spectroscopy and computer power to automate the color matching process, which until recently relied on the human eye for judgement. This new process reportedly results in the reduction of labor and less wasted paint than previous methods.

#### INX and Canadian Fine Color Form Ink Company

INX International Ink Co., Elk Grove Village, IL, and Canadian Fine Color Co., Ltd., Toronto, Ont., have formed the CFC/INX Ltd., in Canada.

Jointly owned by CFC and INX, this new alliance will serve the Canadian market and be supported by a North American network of over 1,600 employees in 60 facilities, providing in excess of 125 million pounds of ink annually.

INX will continue to service the metal decorating business in Canada. All other ink business in Canada will be serviced by CFC/INX Ltd.



## With Genstar Calcium Carbonate Added, It's Almost Fun To Watch.

Mostly because your paint would have an efficient pigment extender giving it high brightness, good color and tinting qualities, and low angle sheen. Our Camel-Wite, Camel-Carb and Camel-Tex carbonates work for gloss, semigloss, flats, primers and sealers. They promote rapid dispersion, give you pH control, and are available in average particle sizes from sub-microns to 12 microns. Call us at 1-410-527-4221 for more information. You'll look at wet paint in a whole new way.



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T his 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, Washing-



ton, D.C. 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.

Architectural and Industrial Coatings Developments—Since the first of the year, several states which were considering going forward with AIM coatings VOC regulations have decided to suspend rulemaking activity for the time being in anticipation of the issuance of a proposed rule by U.S. EPA in May. The states that have made this determination are: Connecticut, Illinois, Indiana, New York, Tennessee, and Wisconsin.

**EPA Informs NPCA Representa**tives There Are No Plans to Revise Pre-1990 Surface Coating CTGs Until 1999—In a February 27 meeting with NPCA staff and member companies held to discuss planned agency activities in 1995, Bruce Jordan, the Director of U.S. Environmental Protection Agency's (U.S. EPA) Clean Air Emissions Standards Branch, announced that presently the agency has no plans to consider revising federal control technique guidelines (CTGs) for limiting VOC emissions from surface coating processes that were issued prior to 1990. Federal CTGs are the template for state regulations, and states rarely go beyond the standards recommended by the CTGs. The Clean Air Act Amendments of 1990 require EPA to consider revisions for pre-1990 CTGs beginning in November 1993. The manufacturing operations benefiting from the policy are the following: surface coatings of miscellaneous metal parts, flat wood paneling, large appliances, coils, cans, paper, fabrics, metal furniture, OEM automotive vehicles, insulation for magnetic wire, and graphic artsrotogravure and flexography. A drawback to the policy is that it does not prevent states from revising standards in these areas prior to 1999. Though unlikely to be widespread,

some states may act before 1999 and at least one state, Illinois, already has proposed revisions to some of these VOC standards, e.g., surface coatings of miscellaneous metal parts. Additionally, certain federal Clean Air Act requirements imposed on the states for establishing limits on hazardous air pollutants on a case-by-case basis when facilities are built or modified may also require reformulations of these coatings before 1999.

EPA Requests Comments on CAA Risk Management Program— EPA is requesting additional comments on its proposed risk management program regulations mandated under the accidental release provisions of the Clean Air Act (CAA). The rule, proposed on October 20, 1993, seeks to reduce the number and severity of chemical accidents. Based on information presented at public hearings and in comments on the proposed rule, EPA would like additional comments on the following options and issues:

(1) Approaches for increasing compliance flexibility and decreasing cost while still ensuring preparedness by tiering the regulatory requirements to take into consideration differences between various types, classes, and kind of sources, devices, and systems;

(2) The hazard assessment approaches (including worst-case scenarios):

(3) Accident information reporting;

(4) public participation in risk management program and plan oversight;

(5) Inherently safer approaches for sources' design and operations; and

(6) The implementation of a CAA section 12(r) regulations, including methods of integrating these requirements into the Title V permitting requirements and the codification of approved state section 112 (r) requirements.

Comments are due May 12. Written comments should be submitted in duplicate to U.S. EPA, Attn: Docket (A-91-73), Room 1500, 401 M Street, S.W., Washington, D.C. 20460. For further information, contact Dr. Lyse De. Helsing, U.S. EPA, at (202) 260-6128.

CPSC Container Safety Update— The Consumer Product Safety Commission (CPSC) has recently discontinued its rulemaking process to prevent infant drownings associated with five-gallon plastic buckets. In addition, CPSC has proposed changes to the child-resistent packaging testing requirements under the Poison Prevention Packaging Act of 1970.

The CPSC voted unanimously on February 8, 1995, to terminate the rulemaking process on five-gallon plastic buckets, initiated on July 22, 1994, citing the substantial cost of the regulations to the commission as well as to industry. The commission responded favorably to industry's information and education campaign undertaken by the five major bucket manufacturers, who have committed \$500,000 over the past five years to support the effort. The commission was also informed that over 80% of five-gallon buckets were labeled.

The commission expressed its intent to continue working with industry and the American Society for Testing and Materials (ASTM) to develop an acceptable performance standard for industrial containers. ASTM continues to consider labeling and performance standard options for five-gallon buckets. The ASTM subcommittee on five-gallon buckets has yet to present a design solution with uniform application.

In addition, the CPSC published a reconsideration of a final rule on February 21, 1995 (60 FR 9654) requesting additional written comments pertaining to the childresistent packaging testing requirements. The commission's original proposal sought to change the current subject test panel aged 18-45 to represent adults aged 60-75 citing concerns for older citizens in opening "child-resistent" closure. Child-resistent closures are an issue for the paint and coatings industry primarily for products packaged in Fstyle (oblong) containers.

RCRA Organic Air Emissions Standards for Tanks and Containers—EPA recently promulgated a

Final rule which will require paint manufacturers classified as large quantity hazardous waste generators (> 220 lbs annually) to reduce volatile organic compound (VOC) emissions from any hazardous waste tanks and containers on site as well as expand recordkeeping, reporting, and monitoring requirements.

The final rule, which goes into effect on June 5, 1995, is expected to reduce VOC emissions from hazardous waste tanks and storage containers by more than 95% below the current levels. EPA estimates that about 2,300 existing RCRA facilities that have permits for treatment, storage, and disposal will be required to comply with the rule, with some 20,000 hazardous waste generators also affected.

The EPA published this final rule in the December 6, 1994, Federal Register (59 FR 62896). While the rule primarily seeks to restrict air toxic emissions from hazardous waste treatment, storage, and disposal facility (TSDF) owners and operators, the rule also requires hazardous waste generators accumulating waste on-site in RCRA "permit-exempt" tanks and containers to include air emission control requirements. Under these standards, airtight covers and vapor control devices must be placed on tanks and containers that store hazardous waste. In addition, the regulation specifies: (1) a 95% efficiency rate for air emission control equipment; (2) requirements to control losses from hazardous waste transfer and other miscellaneous units; (3) inspection and monitoring requirements; (4) recordkeeping and reporting requirements; and (5) waste determinations. Finally, the rule establishes a new EPA reference test method (Method 25E) to determine

the volatile organic concentration of a waste.

This rule will have a significant affect on paint manufacturers which operate RCRA-permitted facilities and/or generate and store hazardous waste that have a volatile organic concentration of  $\geq$  100 parts per million by weight (ppmw). Affected paint manufacturers and other generators will be required to modify hazardous waste storage tank covers, provide emission controls on hazardous waste tanks and distillation units and have portable leak detection and continuous monitoring equipment for the control devices.

The rule is effective June 5, 1995. All hazardous waste placed in the affected tanks, surface impoundments, containers, and other affected units on and after this date must be managed in accordance with the requirements of the final rule. If control equipment cannot be installed by June 5, 1995, a covered facility must prepare an implementation schedule, including dates for purchase orders for equipment and awarding of contracts and the equipment must be installed no later than December 8, 1997.

**House Passes Regulatory** Moratorium Legislation-Could Affect Lead Rules-The House of Representatives has passed legislation that would freeze most new federal regulations for the rest of the year. The Regulatory Transition Act (H.R. 450) would place a moratorium on regulations issued primarily by the U.S. EPA and the Occupational Safety and Health Administration. Regulations issued after November 20, 1994, would be impacted. President Clinton has threatened to veto the legislation should it pass the Senate because it deters regulations that may save lives.

Democrats argued that rules to protect public health, safety, and the environment should not be delayed and that the legislation was written so broadly that it could immerse urgent rules in litigation.

But almost every time the Democrats offered amendments to exempt health, safety, or environmental rules from the bill, Republicans countered that the legislation provided leeway for the administration to impose regulations whenever there was an "imminent threat." Carol Browner, head of the U.S. EPA, objected to the bill in a letter to Congress. She claims, "although its sponsors say it would exempt public health emergencies, the exemption only applies to situations that are immediately life-threatening." She used the packet of lead-paint abatement rules as an example of those that would cause unnecessary risk to human health if delayed. Those final lead rules adversely affected by the moratorium include requirements for training and certification of lead abatement workers; lead-based paint disclosure rules; lead hazard education before renovation; and the significant new use rule for lead.

Under the bill, the White House director of the Office of Information and Regulatory Affairs will determine whether a rule addresses an imminent threat to safety. Reportedly, administration lawyers are expecting to face lawsuits from affected industries if they attempt to push through many rules using the safety loophole.

The House also overwhelmingly agreed to extend until June 30, 1996, the moratorium on rules that affect businesses with fewer than 100 employees. Under the amendment, once the general moratorium expires, an agency could issue a rule affecting both large and small businesses, but small businesses would not have to comply until mid-1996.

Clean Air Act May Be Reopened—If the chairman of the House Commerce Committee Subcommittee on Oversight and Investigations has his way, the Clean Air Act will be reopened and amended during this session of Congress. Rep. Joseph Barton (R-TX) has recently indicated that he is confident his oversight hearings on the CAA will lead to the reopening and revising of the act.

Reportedly, mobile sources will be targeted for revision, but if there is evidence that stationary sources should also be revised, then there is a possibility a review would occur.

Water Quality Bill Introduced in the House—On February 15, 1995, Rep. Bud Shuster (R-PA) introduced H.R. 961, which would reauthorize the Clean Water Act. The bill is essentially an updated version of the bipartisan CWA proposal which was offered during the last congressional session. The current bill differs from the bipartisan proposal in two key respects: (1) it would create an administrative appeals process for wetlands determinations; and (2) would authorize the state revolving loan fund program at \$2 billion annually. The bill specifically addresses several issues of interest to the paint industry. One provision in the bill would exclude facilities whose stormwater does not come into contact with industrial or commercial activities, materials, or products. The bill would also require EPA to prepare risk and cost/benefit analyses for each standard, effluent limitation (paint industry effluent guidelines are expected to revised in 1996) or other regulatory requirements under the CWA.

The Senate Environment and Public Works Committee is expected to tackle stormwater reform legislation again this year, using the bill (S. 2507) that was introduced last October by then committee Chairman Max Baucus (D-MT). An industry coalition has recently formed to lobby Congress on reforming the entire stormwater permit application program.

Meanwhile, EPA's Office of Water is expected to increase its focus on implementation of projects in several areas, including: (1) development of rules to maximize risk reduction; (2) report on the condition of drinking water in the United States; (3) watershed protection; (4) common sense approaches to regulate smaller sources of stormwater runoff; and (5) streamlining monitoring required under the existing stormwater program.

The EPA plans to issue a final multi-sector model general permit for industrial stormwater discharges once the agency receives confirmation from other federal agencies that the permit complies with the Endangered Species Act and the National Historic Preservation Act. The U.S. Fish and Wildlife Service is expected to comment to EPA on the permit by mid-March, which would result in the publication of a final general permit no sooner than May.

The EPA is under a court-ordered deadline of March 15, 1995, to finalize its Great Lakes Initiative (GLI), which seeks to control toxic pollutants in the Great Lakes.

Dischargers of bioaccumulative heavy metals, especially mercury, as well as other compounds are targeted for reduction and elimination. The measure is expected to cost municipalities around the Great Lakes a minimum of \$2.5 billion for compliance. According to a survey conducted by the Council for Great Lakes Governors, the GLI will have a significant impact on regional economic growth and could lead to major employment loss due to decreased levels of available capital. Paint manufacturers and other industries would be impacted through increased costs for wastewater treatment as well as expected higher taxes on businesses and residents in that region of the country to help pay for the excessive capital operating and maintenance costs expected with implementation of the GLI. The outcome of the GLI is expected to establish a precedent for EPA programs to reduce toxic chemicals in the waterways throughout the country.

#### States Proposed Legislation and Regulations

#### ALASKA

Air Quality (Regulation)—The Alaska Department of Environmental Conservation (DEC) has issued final regulations implementing the state's clean air operating permit program. The rule, which would regulate the daily compliance of affected facilities, is effective on approval by the U.S. EPA. Contact DEC, (907) 465-5100.

#### ARIZONA

Air Quality (Regulation)—The Environmental Service Department of Maricopa County, Arizona, has proposed a wood furniture coating volatile organic compound (VOC) regulation.

Lead—AZ S. 1208 (Buster) provides for the certification of lead-based paint abatement programs. On February 23, the bill was amended and released from the Senate Committee on Rules.

Solid Waste—AZ H. 2197 (Bowers) requires state environmental cleanup standards to be technically feasible. The bill passed the House on February 16 and was referred to the Senate.

#### CALIFORNIA

Environmental Marketing—CA S. 426 (Leslie) provides that a person may not make any untrue or misleading environmental marketing claims about a product or package sold in the state. The bill, which was introduced on February 15, is under consideration by the Senate Judiciary Committee.

Lead—CA A. 329 (Bustamante) requires the identification of high-risk children for lead poisoning and establishes procedures for lead abatement activities. On February 23, the bill was referred to the Assembly Committee on Health.

Hazardous Waste—CA S. 352 (Wright) exempts a solid waste or a recycling facility that accepts and processes empty aerosol cans and *de minimis* quantities of nonempty aerosol cans from the requirement to obtain a hazardous waste facilities permit. On February 23, the bill was referred to the Senate Committee on Toxics and Public Safety Management.

CA S. 845 (Leonard) requires the adoption of regulations which establish a permitting, inspection, and enforcement program for the collection and handling of household hazardous waste. The bill was introduced on February 23 and has been referred to the Senate Committee on Toxics and Public Safety.

Packaging—CA S. 605 (Mello) exempts rigid plastic containers which are manufactured for use in the shipment of hazardous materials current packaging requirements. The legislation, which was introduced on February 22, is currently under consideration by the Senate Committee on Governmental Organization.

Solid Waste—CA A. 1647 (Richter) authorizes the adoption of a regulatory framework that provides for different levels of regulation for solid waste facilities, based on the threat of potential environmental harm posed by the facility. The legislation was introduced on February 24.

#### COLORADO

Air Quality (Regulation)— A proposal of the Colorado Department of Public Health and Environment (DPHE) would set criteria for the control of emissions from hazardous air pollutants (HAPs). Contact Joseph Palomba, Jr., DPHE, (303) 692-3100.

#### CONNECTICUT

Labeling—CT H. 6635 (Committee on Housing) requires labels warning of the potential dangers of lead-based paint on paint scrapers and other paint related tools that may be used to scrape and sand paint. The bill was introduced on February 14 and sent to the Joint Select Committee on Housing.

Lead—CT H. 5016 (Godfrey) extends state financial assistance for

lead-based paint removal and/or abatement. On February 22, the legislation was approved and released from the Joint Committee on Planning and Development.

CT H. 6631 (Committee on Housing) prohibits municipalities from adopting lead abatement practices more stringent than those adopted by the state. Introduced on February 14, the legislation has been referred to the Joint Select Committee on Housing.

CT H. 6636 (Committee on Housing) provides limited liability to owners and secured lenders of housing which may contain lead-based paint. Introduced on February 9, the legislation was sent to the Joint Select Committee on Housing.

CT H. 6858 (Committee on Public Health) relates to lead poisoning prevention and compliance with federal standards. Introduced on March 1, the legislation was sent to the Joint Committee on Public Health.

Toxics-in-Packaging—CT H. 6880 (Committee on the Environment) allows the use of packaging which incidentally retains small amounts of heavy metals in the components. Introduced on March 2, the legislation was referred to the Joint Committee on the Environment.

#### **FLORIDA**

Advance Disposal Fee—FL S. 738 (Committee on Natural Resources) provides for the cessation of the imposition of the advance disposal fee on June 30, 1997, and for termination of the program on July 1, 1998. Legislation was introduced on March 7.

A proposed rule of the Florida Department of Environmental Protection (DEP) would clarify how companies can demonstrate that they have met the recycling goal of the advance disposal fee program, and therefore are exempted from the fee. Contact Celso Alaisa, DEP, (904) 488-0300.

Air Quality (Regulation)— The Florida DEP has proposed to extend the deadline dates for submitting operating permit applications. Contact Larry George, DEP, (904) 488-0114.

Lead—FL S. 930 (Turner) requires the certification of lead abatement workers and sets specific certification requirements. The legislation was introduced on March 7.

Pollution Prevention—FL H. 1681 (Tedder) repeals the tax on solvent mixtures. The bill was introduced on March 13.

#### GEORGIA

Lead—GA H. 585 (Thomas) provides that the owner of any rental dwelling shall be liable for expenses incurred in treating the effects of lead poisoning in any child residing in such dwelling if it is determined that the condition is the result of exposure to lead-based paint or other lead hazard. On February 28, the bill received an unfavorable report from the House Judiciary Committee.

Packaging—GA S. 335 (James) prohibits the manufacture and distribution of containers that are not reusable or made of biodegradable and recyclable materials. The legislation was introduced on February 13 and sent to the Senate Committee on Consumer Affairs.

#### HAWAII

Solid Waste—HI H. 394 (Shon) increases the initial solid waste management surcharge per ton of solid waste. The bill was amended and released from the House Committee on Energy and Environmental Protection on February 8.

#### ILLINOIS

Lead—IL H. 921 (M. Davis) allows physicians and health care providers to screen children for lead poisoning in conjunction with the school health exam when, in the physician's judgement, the child is potentially at high risk of lead poisoning. On March 2, the bill was released with a favorable report from the House Committee on Health Care and Human Services.

IL H. 1170 (Black and Granberg) limits the lead blood level screening admission requirement for kindergartens. The legislation was introduced on February 10 and sent to the House Committee on Health Care and Human Services.

IL H. 2330 (Zickus and Lyons) provides that physicians shall screen children through six years of age for lead poisoning who reside in high risk areas. The bill was introduced on February 17 and sent to the House Committee on Health Care and Human Services.

#### INDIANA

Air Quality—IN H. 1598 (Bosma) allows a county having a consolidated city which establishes an air permit program that complies with the federal Clean Air Act to obtain approval from the U.S. EPA as an independent program. The legislation was approved by the House on February 21.

A final rule of the Indiana Air Pollution Control Board (APCB) sets requirements for controlling volatile organic compound (VOC) emissions from major stationary sources in Clark, Porter, Floyd, and Lake counties for which the federal government has not yet specified reasonably available control technology (RACT) standards. Contact Larry Fedor, APCB, (317) 232-8223.

The Indiana Department of Environmental Management (DEM) has announced its intention to initiate a rulemaking that would incorporate federal standards relating to compliance extensions for facilities that have undertaken early reductions of hazardous air pollutant emissions. Contact Timothy Method, DEM, (317) 232-8217.

#### **KENTUCKY**

Hazardous Waste (Regulation)— The Kentucky Department of the Environment (DOE) has proposed a regulation relating to standards for characterizing hazardous waste sites, risk assessment, and risk management options. Contact James Hale, DEP, (502) 564-2225.

#### LOUISIANA

Air Quality (Regulation)— A proposal of the Louisiana Department of Environmental Quality (DEQ) specifies standards for industrial waste water for facilities that emit at least 50 tons of VOCs per year and are located in Ascencion, East Baton Rouge, Iberville, Livingston, Pointe Coupee, and West Baton Rouge parishes. Contact Patsy Deaville, DEQ, (504) 765-0399.

Effective April 20, pollutant fees will be raised from 25 to 79% for virtually all regulated pollutants, including air pollutants.

Lead (Regulation)—The Louisiana DEQ is withdrawing a proposed rule which would have set criteria for the licensure and certification of lead abatement personnel. In its place, the U.S. EPA lead standard will be incorporated into the current state regulation. Contact Patsy Deaville, DEQ, (504) 765-0399.

#### MAINE

Lead—ME H. 560 (Daggett) imposes a tax of 25 cents per gallon of paint sold at retail in the state in order to fund a program to provide low-interest loans for lead abatement. Introduced on March 7, the bill was sent to the Joint Committee on Appropriations and Financial Affairs.

#### MARYLAND

Lead—MD H. 1238 (Guns) establishes programs for lead hazard reduction grants and loans. The legislation was introduced on February 23 and sent to the House Committee on Environmental Matters.

MD S. 311 (Minsky) requires the establishment and administration of a Lead Poisoning Screening Program and of a Lead Poisoning Outreach and Education Program. On February 2, the legislation was referred to the Senate Committee on Economic and Environmental Affairs.

#### MASSACHUSETTS

Air Quality (Regulation)—A final regulation adopted by the Massachusetts Department of Environmental Protection (DEP) allows emissions averaging by controlled sources to achieve compliance with VOC and NOx emission standards. Contact Don Squires, DEP, (617) 292-5618.

Automotive Refinishing—MA H. 3677 (Valianti) restricts the sale of automotive paint products to licensed personnel. On February 16, the legislation was introduced and sent to the Joint Committee on Commerce and Labor.

Hazardous Materials—MA H. 3304 (Hodgkins) defines paint as a hazardous product and requires retailers to label such items and to provide toxicity information to consumers. On February 14, the legislation was introduced and referred to the Joint Committee on Commerce and Labor.

The Massachusetts DEP adopted a final rule which modifies the reportable concentrations on the state's hazardous materials list, including the deletion of titanium dioxide from the list. Contact James Coleman, DEP, (617) 292-5648.

Household Hazardous Waste—MA H. 2616 (Kaprielian) establishes a household hazardous waste collection grant program. On February 8, the bill was introduced and sent to the Joint Committee on Natural Resources and Agriculture.

MA S. 1116 (Keating) relates to household hazardous waste. On February 23, the legislation was introduced and referred to the Joint Committee on Natural Resources and Agriculture.

Labeling (Regulation)— A proposal of the Massachusetts DEP would modify reporting requirements under the Toxic Use Reduction Act to require that products and processes be described in accordance with certain proposed codes. Contact Walter Hope, DEP, (617) 292-5982.

Lead—MA H. 1832 (Local Sponsor) amends the lead paint statutes. On March 2, the Joint Committee on Health Care released the bill with an unfavorable recommendation.

MA H. 2173 (Mariano) establishes a state laboratory for the detection of lead and lead poisoning. On March 2, the bill was released from the Joint Committee on Health Care with an unfavorable report.

MA S. 37 (Birmingham) regulates the removal of lead paint. Introduced on February 17, the legislation was sent to the Joint Committee on Commerce and Labor.

MA S. 527 (Leahy) establishes a lead paint bank. The bill was introduced on February 17 and sent to the Joint Committee on Health Care.

MA S. 1557 (Amorello) promotes safety from lead contamination in housing. Introduced on February 24, the bill was sent to the Joint Committee on Taxation.

The Massachusetts Department of Public Health (DPH) has proposed a rule which would establish a program for the interim control of lead paint. Under the proposal, property owners would have up to two years to gather funds sufficient to pay for full compliance with lead abatement regulations. Contact Roy Petre, DPH, (617) 522-3700.

An amendment issued by the Massachusetts DPH would allow property owners or their agents to perform certain low-risk lead abatement activities without first being licensed as a de-leader. Contact Roy Petre, DPH, (617) 522-3700.

Market Share Liability—MA H. 2205 (Jehlen) authorizes the use of market share liability in civil actions brought against producers or manufacturers of lead constituents used in paint. The legislation, which was introduced on January 5, is being considered by the House Judiciary Committee.

Packaging—MA H. 2618 (Marzilli) prohibits the use of packaging that is not recyclable. Introduced on February 8, the bill was sent to the Joint Committee on Natural Resources and Agriculture.

Spray Paint Restrictions—MA H. 2520 (Owens-Hicks) prohibits the sale of spray paint to minors under the age of twenty-one. Introduced on February 8, the legislation was referred to the Joint Committee on Commerce and Labor.

Toxic Substances—MA H. 2404 (Resor) restricts the use, manufacture, or sale of chlorinated solvents. The bill, which was introduced on February 3, is under consideration by the Joint Committee on Natural Resources and Agriculture.

Toxics-in-Packaging—MA H. 3389 (S. Angelo) prohibits the intentional introduction of lead, mercury, cadmium, or hexavalent chromium into packaging. The legislation was introduced on February 14 and sent to the Joint Committee on Natural Resources and Agriculture.

#### MICHIGAN

Lead (Regulation)— A proposal issued by the Michigan Department of Public Health (DPH) would establish requirements for blood lead analysis reporting. DPH, (517) 335-8885.

Recycling—MI S. 131 (Cherry) requires that 30% of the solid waste generated in the state be recycled and that 60% of such material be recycled by July 1, 2000. It sets material content standards for paper, steel, aluminum, polystyrene, and plastics. The bill was introduced on January 17 and sent to the Senate Committee on Natural Resources and Environmental Affairs.

#### **MINNESOTA**

Air Quality (Regulation)—The Minnesota Pollution Control Agency (PCA) has extended operating permit application deadlines by three months. Contact Andrew Ronchak, PCA, (612) 296-3107.

Environmental Marketing—MN S. 588 (Chandler) requires a manufacturer, packager, or retailer making an environmental claim about a product or its packaging to comply with federal regulations on environmental marketing. Introduced on February 21, the bill was referred to the Senate Committee on Commerce and Consumer Protection.

Packaging—MN H. 1100 (Munger) requires that rigid plastic containers must include 10% postconsumer material by January 1, 1997, and 25% by January 1, 2001. The bill was introduced on March 6 and sent to the House Committee on Environment and Natural Resources.

Pollution Prevention—MN H. 825 (Munger) provides grants to develop or implement pollution prevention projects or practices and exempts facilities which do not release toxic chemicals from pollution prevention plan requirements. Introduced on February 23, the bill has been referred to the House Committee on the Environment and Natural Resources.

Product Liability—MN S. 488 (Betzold) provides a defense in actions brought against distributors, manufacturers, or sellers for damages from an alleged defect in a product by proving that the product conformed to state-of-the-art technology in existence at the time of production or labeling. The bill was referred to the Senate Judiciary Committee on February 16.

#### MISSOURI

Air Quality (Regulation)—The Missouri Department of Natural Resources (DNR) has proposed revisions to the state air control program, including changes to the definition of VOCs and what constitutes construction of a new source, as well as amendments that would allow a source to begin construction prior to approval of a permit under certain circumstances. Contact Roger Randolph, DNR, (314) 751-4817.

Transportation (Regulation)—An emergency regulation issued by the Missouri Department of Economic Development (DED) implements federal requirements relating to the intrastate transportation of goods. Contact DED, (314) 751-7100.

#### MONTANA

Lead—MT H. 498 (Cocchiarella) creates a lead-based paint hazard control program and authorizes the promulgation of rules governing leadbased paint activities. On February 15, the bill was amended and released from the House Committee on Business and Labor.

MT H. 499 (Cocchiarella), among other things, (1) authorizes the establishment of a lead poisoning reduction program; (2) requires the reporting of lead poisoning cases; (3) authorizes the implementation of a case management system; and (4) requires a health care provider who makes a diagnosis of lead poisoning to provide information concerning treatment and follow-up to the victim. The legislation was released with a favorable recommendation from the House Committee on Business and Labor on February 15.

Pollution Prevention (Regulation)— The Montana Department of Health and Environmental Sciences (DHES) adopted final regulations providing special property tax treatment for air and water pollution control equipment. Contact Jeff Chaffee, DHES, (406) 444-2544.

#### **NEVADA**

Air Quality—NV S. 247 (Committee on Natural Resources) authorizes the establishment of a program for the exchange of credits to reduce air pollution. Introduced on March 6, the bill was referred to the Senate Committee on Natural Resources.

#### **NEW HAMPSHIRE**

Lead—NH S. 25 (Lovejoy) relates to case management by the state in lead paint poisoning cases. The bill was approved by the Senate on February 21 and was referred to the House Committee on Health, Human Services, and Elderly Affairs.

NH S. 27 (J. King) relates to lead poisoning prevention and control. On February 21, the legislation was released with amendments from the Senate Committee on Public Institutions, Health, and Human Services.

NH S. 54 (Wheeler) provides that the mere presence of lead-based paint does not constitute negligence on the part of the owner of a dwelling. The legislation passed the Senate on February 21 and was sent to the House Committee on Judiciary and Family Law.

NH S. 128 (Shaheen) establishes a Lead Hazard Abatement Fund. On February 21, the bill failed to pass the Senate.

The New Hampshire Department of Health and Human Services (DHHS) has proposed a regulation which would clarify the procedures for the licensure and certification of lead abatement workers and would establish standards to be followed when performing lead abatement activities. Contact Jean Bergman, DHHS, (603) 271-4501.

The New Hampshire Department of Health and Human Services (DHHS) has proposed amendments to the childhood lead poisoning prevention program, including provisions that would clarify requirements for "in-place management." Contact Jean Bergman, DHHS, (603) 271-4501. Toxics-in-Packaging—NH S. 129 (Rodeschin) exempts certain packages and packaging components containing lead, cadmium, mercury, or hexavalent chromium from compliance with current toxic reduction laws. Exemptions include packaging in which heavy metals have been added in order to comply with federal health requirements or in cases in which there are no feasible alternatives. The bill passed the Senate on February 7 and was sent to the House Committee on the Environment and Agriculture.

#### NEW JERSEY

Lead—NJ S. 1537 (Bassano and Matheussen) requires the screening of children for lead exposure. The bill passed the Senate on February 9 and was referred to Assembly Committee on Health and Human Services.

A final rule of the New Jersey Department of Health (DOH) establishes requirements for certifying lead abatement personnel. Contact Joseph Eldridge, DOH, (609) 984-6710.

Pollution Prevention—NJ A. 903 (DiGaetano and Bryant) establishes a pollution prevention framework, emphasizing recycling and reduction. The legislation passed the Assembly on January 23 and was referred to the Senate Committee on the Environment.

Transportation (Regulation)—The New Jersey Department of Transportation (DOT) has readopted final amendments relating to federal standards concerning the transportation of hazardous materials. Contact Janis Hoagland, DOT, (609) 292-0716.

#### NEW MEXICO

Air Quality (Regulation)—The Air Quality Board (AQB) for Albuquerque/Bernalillo County has issued a final regulation that relates to new source review, operating permits, and permit fees. It also incorporates federal standards concerning emissions of hazardous air pollutants. Contact Glen Dennis, AQB, (505) 768-2641.

#### NEW YORK

Household Hazardous Waste—NY A. 4631 (McGee) establishes a Household Hazardous Waste Disposal Program for the treatment and disposal of hazardous materials used or possessed by individual households in the state. Introduced on February 27, the legislation was referred to the Assembly Committee on Environmental Conservation.

Labeling—NY A. 3975 (DiNapoli) requires the labeling of the recycled content level of consumer packaging. The bill, which was introduced on February 16, is being considered by the Assembly Committee on Consumer Affairs and Protection.

Lead—NY A. 4329 (Eve) provides that policies insuring against liability for injury shall not exclude coverage for exposure to lead-based paint. On February 23, the bill was introduced and referred to the Assembly Committee on Insurance.

The New York Department of Health (DOH) adopted a final rule dealing with lead poisoning control through environmental assessment and lead abatement. Contact DOH, (518) 474-2011.

Packaging—NY A. 3975 (DiNapoli) requires all manufacturers to disclose the post-consumer recycled content of a package on the label of all consumer commodities. On February 16, the bill was sent to the Assembly Committee on Consumer Affairs and Protection for consideration.

NY S. 2395 (Spano) requires packaging to be reusable or recyclable. Introduced on February 16, the legislation was sent to the Senate Committee on Environmental Conservation.

Spray Paint Restrictions—NY A. 4893 (Herbst) prohibits the sale of aerosol spray paint cans containing a fluid that is not water-soluble. On March 6, the bill was introduced and referred to the Assembly Committee on Consumer Affairs and Protection.

Toxic Chemical Release—NY A. 3970 (DiNapoli) requires certain facilities to report releases of toxic chemicals on a toxic chemical release form. Introduced on February 16, the legislation was referred to the Assembly Committee on Environmental Conservation.

#### NORTH CAROLINA

Lead (Regulation)— The North Carolina Department of Environment (DOE) has issued final regulations, effective January 1, 1995, which set the standards for determining elevated blood levels. Contact Grady Balentine, Department of Justice, (919) 733-4618.

Toxic Chemical Release—NC H. 308 (Hackney) establishes a state goal for the reduction of releases and transfers of certain toxic chemicals. The bill, which was introduced on February 23, is under consideration by the House Committee on Health and the Environment.

#### OREGON

Air Quality (Regulation)—A proposed regulation of the Oregon Department of Environmental Quality (DEQ) would increase the fees associated with the operating permit program and incorporate by reference the federal standards relating to controlling emissions of hazardous air pollutants. Contact Chris Rich, DEQ, (503) 229-6775.

Household Hazardous Products— OR H. 2741 (Brown) requires retail establishments to display designated shelf signs in the immediate vicinity of household hazardous products. Introduced on February 16, the legislation was sent to the House Committee on Natural Resources.

Hazardous Waste—OR H. 3201 (Brown) requires the development of consumer information on the proper disposal of retail products containing hazardous chemicals and requires retailers to provide customers with this information. The bill, which was introduced on March 3, is being considered by the House Committee on Natural Resources.

Lead—OR H. 2971 (Repine) establishes lead-based paint activities programs to conform to federal law including training, licensing, and registration programs. The bill was introduced on March 2.

Packaging—OR H. 2680 (Naito) requires rigid plastic containers to be composed of plastic that is being recycled at a rate of 50%. The legislation was sent to the House Committee on Natural Resources on February 13.

#### PENNSYLVANIA

Lead—PA H. 1046 (Laughlin) requires the certification of individuals engaged in lead-based paint activities, establishes minimum training requirements, and sets criteria for performing lead-based paint activities. On March 3, the legislation was introduced and sent to the House Committee on Labor Relations.

Solid Waste—PA H. 979 (Michlovic) permits counties to form stormwater management districts for the purpose of regulating stormwater within designated watershed boundaries. The bill was introduced on March 1 and referred to the House Committee on Environmental Resources and Energy.

#### **RHODE ISLAND**

Environmental Marketing—RI H. 6020 (Henseler) makes it unlawful for any persons selling goods to make deceptive claims about their environmental impact.

On February 14, the bill was introduced and referred to the House Committee on Corporations.

Environmental Marketing—RI H. 6564 (Long and Iannitelli) concerns truth in environmental marketing claims. The bill was introduced on February 14 and sent to the House Committee on Corporations.

Lead—RI H. 6507 (Murphy) concerns lead poisoning prevention. The bill, which was introduced on February 14, was sent to the House Committee on Corporations.

The Department of Health (DOH) has issued a final rule establishing standards for lead poisoning prevention, including procedures for blood screening and laboratory testing. Contact DOH, (401) 277-1417.

Spray Paint Restrictions—RI H. 6554 (McCauley) concerns spray paint can access. Introduced on February 14, the legislation was referred to the House Judiciary Committee.

Toxics-in-Packaging—RI S. 533 (Porter) adds an exemption category for reusable packaging to the Toxic Packaging Reduction Act of 1990 and extends the recycling exemption to January 1, 2000. The bill, which was introduced on February 9, is currently under consideration by the Senate Committee on Special Legislation.

#### TENNESSEE

Air Quality (Regulation)—The Tennessee Department of Environment and Conservation (DEC) has issued final regulations removing Knox County from the list of counties in which sources of VOCs and NOx must make specified reports. Contact Malcolm Butler, DEC, (615) 532-0554.

Pollution Prevention—TN H. 345 (Kisber) makes permanent sales tax credits on pollution control equipment for auto paint shops and similar businesses. The bill received a favorable report from the House Committee on Finance. Solid Waste—TN H. 877 (B. Turner) prohibits municipalities from collecting stormwater user fees in a manner not authorized by law. The bill was sent to the House Committee on Conservation and the Environment on February 13.

#### TEXAS

Hazardous Waste (Regulation)— The Texas Natural Resources Conservation Commission (NRCC) has announced the withdrawal of a proposed rule that would have established a comprehensive spill prevention program for discharges of hazardous substances to the environment. Contact Marianne Baker, NRCC, (512) 239-0475.

Lead—TX H. 1373 (Coleman) relates to the creation of a lead-based paint certification program for target housing. The legislation was introduced on February 16.

TX H. 1456 (Van de Putte) relates to the identification of children suffering from lead poisoning and to control measures for lead contamination. On February 21, the legislation was sent to the House Committee on Public Health.

TX H. 1888 (Van de Putte) relates to the regulation of lead-based paint activities and hazards. The bill, which was introduced on March 2, is under consideration by the House Committee on Public Health.

Spray Paint Restrictions—TX H. 2115 (Lewis) creates an offense for the possession of aerosol paint by a person younger than 18 years. The legislation was introduced on March 7 and referred to the House Committee on Criminal Jurisprudence.

TX S. 707 (Rosson) relates to the accessibility of certain paints to business patrons in specific municipalities. The legislation was introduced on February 22 and sent to the Senate Committee on Economic Development.

#### UTAH

Air Quality (Regulation)—The Utah Department of Environmental Quality (DEQ) has proposed amendments to the state's clean air operating permit program, including provisions that would limit the ability to exempt sources as insignificant. The proposal also clarifies the application of reasonably available control technology VOC emission standards for major and minor sources of VOC emissions. Contact Jan Miller, (DEQ), (801) 536-4042.

Hazardous Waste (Regulation)—A final rule of the Utah Department of Environmental Quality (DEQ) changes provisions concerning the circumstances under which certificates of compliance can be revoked for the underground storage tank program. Contact Gary Austin, DEQ, (801) 536-4100.

#### VERMONT

Air Quality (Regulation)—A proposal of the Vermont Agency for Natural Resources (ANR) would revise the state's program for controlling emissions of PM-10 (particulate matter) in areas which are in attainment for this pollutant. Contact Richard Valentinetti, ANR, (802) 241-3840.

Hazardous Materials—VT H. 258 (Gossens) repeals the law that requires shelf labeling where hazardous products are sold at retail. On February 14, the bill was introduced.

Lead—VT H.B. 471 (Kurt) establishes a primary program to prevent childhood lead poisoning and provides state resources to train property owners and managers to reduce lead-based hazards in rental housing and child care facilities. The bill was introduced on March 3.

Solid Waste—VT H. 415 (Rose) repeals the prohibition on the disposal

of solidified water-based paint in landfills. The bill was introduced on March 1.

#### VIRGINIA

Lead—VA H. 2454 (Reid) revises current provisions relating to certification of lead contractors and workers to comply with proposed U.S. EPA regulations. On March 1, the legislation was sent to the governor for signature.

Water Quality—VA S. 763 (Howell). eliminates the requirement that applicants for permits to discharge sewage, industrial wastes, or other wastes, to or adjacent to, state waters provide a certificate from the local government where the discharge will take place. The House approved the legislation on February 17.

#### WASHINGTON

Lead (Regulation)—The Washington Department of Labor and Industry (DLI) issued final regulations concerning employer response to results of medical monitoring for workplace exposures to lead. Contact Marcia Holt, DLI, (206) 956-5530.

Solid Waste—WA S. 5888 (Sutherland) authorizes the consideration of the nature or type of land user in establishing charges for sewer and stormwater control systems. Introduced on February 13, the bill was referred to the Senate Committee on Energy, Telecommunications, and Utilities..

#### WYOMING

Hazardous Waste (Regulation)—A final rule adopted by the Wyoming Department of Environmental Quality (DEQ) establishes requirements for the management of underground storage tanks containing hazardous materials. Contact LeRoy Feunser, DEQ, (307) 777-7096.

## New York Society's Second Annual Symposium Highlights Additives and Modifiers for Coatings

he New York Society for Coatings Technology's Second Annual Symposium "Recent Advances in Additives and Modifiers for Modern Coatings" will be held on May 3-4, 1995, at the Newark Airport Holiday Inn North, Newark, NJ.

The symposium will feature 14 papers that will discuss the following:

"Surfactants—Additives for an Emerging Technology"—Sam Morell, of S.P. Morell and Co.;

"Factors Influencing Defoamer Selection for Coatings and Printing Inks"—Chuck

#### Louisville SCT and PCA Host Spring Conference

On April 19, the Louisville Society for Coatings Technology, in conjunction with the Louisville Paint and Coatings Association, will host the 1995 Spring Symposium, themed "Spectrum of Coatings Science." The event will take place at the Executive West Hotel, in Louisville, KY.

The program for the one-day symposium is as follows:

"Characterization and Analysis of Coatings Rheology and Coatings Flow"—Richard R. Eley, of The Glidden Co.;

"Controlling Metallic and Pearlescent Colors with Sphere Spectrophotometer and Existing Color Formulation Software"—John Mueller, of Datacolor International;

"Tests and Techniques in the Selection of Preservatives for the Coatings Industry"— William Machemer, of Troy Corp.;

"Formulating a Very Low VOC H<sub>2</sub>O Epoxy Maintenance Coating"—Henry Heck, of Dow Chemical North America, Resin Products R&D;

"Recent Developments in Epoxy Resins and Curing Agents"—David Helfand, of Ciba Corp., Polymers Division;

"Improvement of Wetting on Plastics and Related Substrates"—Fred Lewchik;

"Reducing Automotive Paint Costs Via Pigment Selection"—Milton Misogianes, of Ciba Corp., Pigments Division; and

"High-Gloss Alkyd Type Performance Emulsion Polymers: A New Technology"— Fred Giles, of UCAR Emulsion Systems.

The registration fee for the symposium is \$50. The LSCT/LPCA dinner meeting fee is an additional \$25. For more information, contact llona Duvall, of Red Spot Paint Co., P.O. Box 418, Evansville, IN 47703-0418; (812) 428-9337. D'Amico, of Ultra Additives, Inc.;

"Polyacrylate Leveling and Defoaming Agents for High-Solids Coatings"—Rudy Berndlmaier, of King Industries;

"The Use of Hydroxyethyl Ethylene Urea as a Zero-VOC Additive"—Eric Percy, of Percy International Ltd.;

"Description and Applications of a New Line of Specialty Waxes"— Carl J. Cappabianca and Andrew A. Romano, of Ashland Chemical Co., Drew Industrial Division;

"An Excellent Crosslinking Agent for Water-Based Low VOC Coatings"—Gerald V. Delancy, of Sybron Chemicals Inc.;

"Aliphatic Epoxy Emulsion Crosslinker for Waterborne Coatings"—Del Eslinger, of Cook Composites and Polymers;

"Aqueous Suspensions of Cellulosic Thickeners: Function and Mechanisms in Water-Based Coatings"—C.W. Vanderslice, H.F. Haag, and C.L. Burdick, of Aqualon Co;

"A New Ultra-Low VOC, High Performance Dispersant Technology for Modern Waterborne Coatings Systems"—P. Quednau and M. Philipoom, of EFKA Chemicals and B.V. Van Doren, R. Van Doren, and W. Reynolds, of Rheox Inc.;

"Advantages and Selection of Associative Thickeners"—Ron Broadbent, of Henkel Corp.;

> "Ultra Low Viscosity Oxazolidine and Aldimine-Based Reactive Diluents for High-Solids Polyurethane Coatings"—Thomas L. Johnson, of ANGUS Chemical Co.;

"Wax Additives for Coatings: An Overview"—Ramon Pineiro and Michael Frantz, of Daniel Products Co.;

> "Properties of Silica Products for Performance in Coatings"—Kathy Closs, of Dow Corning

Corp.; and

"The Relationship Between Adhesion Promotion and Corrosion Reduction"— Lawrence B. Cohen, of Chartwell International Inc.

The registration fee is \$150 and includes two luncheons, reception, and dinner. For more information, please contact NYSCT, 520 Westfield Ave., Ste. 208, Elizabeth, NJ 07208; (908) 354-3200 or Larry Waelde at (201) 589-2500, ext. 359.

#### "Practical Approaches to VOC Reductions" Theme for Pacific Northwest Society's Spring Meeting

The Pacific Northwest Society for Coatings Technology's 48th Annual Spring Symposium will be held at the Red Lion Lloyd Center Hotel, Portland, OR, on May 4-6, 1995. This year's theme is "Practical Approaches to VOC Reductions." The technical program is scheduled to include:

"Unique Wetting and Dispersing Additives for High-Solids and Waterborne Systems"—Robert McMullin, of BYK-Chemie;

"Wollastonite, Filler of Choice in Waterborne and Low VOC Anti-Corrosive Formulations"—Sara Robinson, of NYCO Minerals;

"New Alkyd Emulsion Technology which Allows Less than .2 lb/gal VOC and Waterborne Dispersions from a Powdered Polyester Resin"—Kelley Lane, of Amoco Chemical Co.;

"Oxsol 100—An Alternative Solvent that Is VOC Exempt and Its Use in Coatings Formulations to Reduce VOC Levels"— Gilbert Nagy, of Occidental Chemical Corp.;

"The Next Generation of Stain Blocking Emulsions for Interior and Exterior that Need No Coalescing Solvents or Stain-Inhibiting Pigments"—Michael Wildman, of Engineered Polymer Solutions (EPS);

"Formulating with Waterborne Resins"— Carol Williams, of Reichhold Chemicals;

"Beyond Multipurpose TiO2, New Pigments Designed to Lower Coating VOCs"— R. Gattman, of E.I. du Pont de Nemours;

"New Developments in Waterborne Dispersion Technology for Wood Surfaces"— Richard Johnson, of McWhorter Technologies; and

"Coming VOC Regulations and the Clean Air Act in the U.S. and Canada"— Carol Niemi, of NPCA/Dow Chemical.

In addition to the technical program, a slate of social activities is featured. Attendees may spend Thursday golfing or skiing. Following the afternoon on the links or the slopes, an evening at the greyhound races is planned. For more information on attending the symposium, contact Ken Wenzel, Chemical Distributors Inc., P.O. Box 10763, Portland, OR 97210; (503) 243-1082. (See registration form on page 22.)

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#### SSCT Annual Meeting Slated for April 19-21 in Savannah, GA

The Southern Society for Coatings Technology's Annual Meeting will be held at the Hyatt Regency, Savannah, GA, on April 19-21, 1995. This year's theme is "Getting Ready for the Next Century."

The technical program is scheduled to include the following presentations:

"Overview of Coming Air Regulations"—Bernie Zysman, of Occidental Chemical;

#### "The Fundamentals of Color" Seminar Schedule Announced

Macbeth, a Division of Kollmorgen Instruments Corp., New Windsor, NY, has announced the 1995 schedule for "The Fundamentals of Color Seminar."

Seminar dates and locations are: April 18-19, Charlotte, NC; May 2-3, Cherry Hill, NJ; May 23-24, Newton, MA; June 6-7, St. Louis, MO; and June 20-21, Rosemont, IL.

An introduction to the basic principles of color science, this seminar has been developed for people involved in the design, production, quality control, or inspection of materials for which color is important.

The seminar is comprised of lectures and practical demonstrations on the use of visual standards, controlled lighting and viewing conditions, and color measurement instrumentation.

The registration fee is \$395 and includes reference materials, a workbook, and lunch. For more information, contact Wanda F. Smith, Macbeth, 405 Little Britain Rd., New Windsor, NY 12553-6148. "Environmental Regulation Impact on Coatings Preservatives/Biocides and Developing Technologies—Doug Simpson, of Olin, Inc.;

"The Next Generation of Stain Blocking Emulsions"—Mike Wildman, of Engineered Polymer Solutions, Inc.;

"New Developments in Dispersion Technology"—Rich Johnson,of McWhorter Technologies, Inc.;

"What ISO 9000 Registration Means to You"—Tom Daquilla, of Daniel Products;

"Low VOC Technology for the Coming Decade"—Steven Cooper, of Rohm and Haas, Inc.; "New Methods for Determining Paint and Coatings Failures"—Rich Brown, of MVA, Inc.;

"Recent Advancements in Waterborne Urethanes"–Gail Pollano, of Zeneca Resins; and

"Design of Experiments for Paints and Coatings—Fundamentals and Applications"—Roger Hester, of University of Southern Mississippi.

For more information on the 1995 Southern Society for Coatings Technology's Annual Meeting, contact Wayne West, Thompson & Formby, Inc., 10136 Magnolia Dr., Olive Branch, MS 38654.

#### Munsell Color Science Lab Offers Two Color Courses; Color Measurement Program Slated for June 5-7

Rochester Institute of Technology's (RIT) Munsell Color Science Laboratory, Rochester, NY, will conduct four industrial courses in color science for electronic imaging systems.

The first course offered by RIT is "Device-Independent Color Imaging," to be held on June 12-14. This course is designed to teach methods of achieving high-accuracy color for electronic imaging peripherals, so called device-independent color. An understanding of visual system (colorimetry), metrology (spectrophotometry, photometry, spectraradiometry), image formation principles (color modeling), and the interaction between observers and the colored image (color-appearance) will be presented.

#### **Thames Valley Section to Address In-Can Tinting**

The society of the so

Due to the increasing need to produce small batches of paint in a variety of colors, accurately, consistently, and efficiently for both the industrial and decorative applications, the Thames Valley Section of the Oil and Colour Chemists' Association (OCCA), is featuring "Aspects of In-Can Tinting Schemes." Slated for April 27, at the Marriott Hotel, Slough, United Kingdom, this symposium addresses these issues with the following presentations:

"Pigment Selection for Tinting Schemes"-Jeurgen Fröschle, of BASF;

"New Developments in Wetting and Dispersing Agents for Color Pastes"—Lionel Morpeth, of BYK-Chemie;

"Computer Formulations for Tinting Systems"—Hayden Williams, Colour Computers Ltd.;

"Focusing on Decorative Tinting Systems into the Next Millennium"—Howard Chadwick, of Fluid Management (UK) Ltd.;

"The History, Development, and Future of Industrial Tinting Systems and Machinery"— Tikkurila Oy;

"The History, Development, and Future of Decorative Tinting Systems"; and

"Benefits of In-Can Tinting"-Roger Nicholls, of Johnstones Paints Ltd.

The registration fee for nonmembers is £176.25. The fee includes abstracts of papers and a buffet lunch. For more information, contact Chris Pacey-Day or Yvonne Waterman, at OCCA, Priory House, 967 Harrow Rd., Wembley, HA0 2SF, United Kingdom.

"Color-Appearance Models: Theory and Practice" is scheduled for June 15-16. This two-day course is designed for scientists and engineers working in the fields of color science, color reproduction, and electronic imaging. The aim of this course is to introduce scientists and engineers in the fields of color science, imaging science, and other related disciplines to the fundamental phenomena, techniques, and models of color appearance.

The third course "Principles of Industrial Color Measurement" will be held on June 5-7 and will focus on the applications of colorimetry for industrial color control. Topics to be discussed include: spectrophotometry principles, geometry selection, and methods of characterizing precision and accuracy; CIE colorimetry—derivation of colorimetry from XYZ through CIELAB; and tolerancing— CMC and TC1-29 equations, deriving visual tolerances from historical pass/fail data, and optimizing lic ratios. Other topics to be explored are terminology, color vision, color order systems, illuminant and observer metamerism, and color TQM concepts.

"Industrial Instrumental Color Matching" is the fourth course offered. Scheduled for June 8, this course presents techniques for computer colorant formulation systems in an industrial environment. Colorant identification via spectral analyses, additive functions of reflectance (Kubelka-Munk) and transmittance (Beert-Lambert), semi quantitative production batch adjustments, principles of computer colorant formulation, methods to get the most of your system, and a problem solving session will be emphasized.

For additional information on any of these course, contact Colleen M. Desimone, Munsell Color Science Laboratory, Rochester Institute of Technology, Chester F. Carlson Center for Imaging Science, 54 Lomb Memorial Dr., Rochester, NY 14623.

## Western Coatings Societies Symposium Provides Attendees With "New Opportunities, Challenges & Solutions"

cousing on "New Opportunities, Challenges and Solutions," members of the Western Coatings Societies held their 22nd Biennial Symposium and Show (WCSSS) at the Hilton Hotel in San Francisco, CA on February 20-22. Sponsored by the Golden Gate, Los Angeles, Pacific Northwest, and Rocky Mountain Societies for Coatings Technology, the WCSSS welcomed 1600 attendees to participate in the event.

In keeping with their charter to promote educational activities to the coatings industry, the WCSSS provided many "new opportunities" for participants to

learn about the most recent innovations in the field. In addition, the Golden Gate Society, hosts of the 1995 event, extended nearly 300 complimentary registration vouchers to laboratory, factory, and sales personnel, and students who otherwise could not attend.

The Symposium offered a varied technical program which featured concurrent sessions focusing on five different aspects of the coatings business. The 39 technical papers presented were well attended and well received.

Registrants also had the opportunity to explore possible solutions to the



Show exhibits featuring the latest products and services attracted 1600 attendees.

The tone of the meeting was set by Keynote Speaker, Sandra Skommesa, President of Ellis Paint Company. Ms. Skommesa predicted that the technical aspects of the coatings industry will be a primary focus for the next five years. Advances in this area will be pivotal in determining the future direc-



General Chairman Gordon Pioch welcomes Entertainment Chairman Barry Adler and his wife, Spousal Program Chairman, Evelyn Adler.

challenges facing the industry with representatives from 86 exhibiting companies. tions of the industry. She pointed out that the theme of the WCSSS, "New Opportu-



Symposium Treasurer Pat Shaw, FSCT Executive Vice President Robert F. Ziegler, and FSCT President Joseph P. Walton at Industry Luncheon.

nities, Challenges and Solutions," could be addressed by the three ingredients which lead to success—vision, attitude, and passion. It takes vision to recognize the opportunities which may be made available; a positive attitude to be open to meeting challenges; and passion to find and implement solutions. Ms. Skommesa's presentation



Co-chairman Don Nolte congratulates Keynote Speaker Sandra Skommesa.

generated a great deal of excitement for the audience of 350 who attended the Opening Session.

Another highlight of the WCSSS was the Industry Luncheon. Duane Garrett, political pundit and radio talk show host, presented his view of national politics for 1996.

The Symposium concluded with a banquet and show, with entertainment by comedian Boss

median Ross Shafer and the music of "The Diamonds"—a "golden oldies" rock band that treated the audience to hits from the '50s and '60s.

Serving as General Chairman of the Symposium was Gordon Pioch, of Eureka Chemical Co. Assisting him were Co-



Luncheon Speaker Duane Garrett.

Chairman—Don Nolte, of John K. Bice Co. and Treasurer—Patricia Shaw, of Radiant Color. Chairmen of the event were: Exhibits—Tom Dowd, of Dowd & Guild; Technical Program—Adrian Adkins, of Schoofs, Inc.; Entertainment—Barry Adler; and Spousal Program—Evelyn Adler.

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## Correlation of Thermal History and Performance in Thermosetting Powder Coatings

K.G. Panjnani, S.D. Pace, and S.F. Thames—University of Southern Mississippi\*

#### INTRODUCTION

P owder coatings represent approximately 5-8% of the U.S. industrial finishing market of which 80-85% are thermosetting powders.<sup>1</sup> Powder coatings offer several advantages including little or no volatile organic content, high utilization rates, energy savings, and elimination of hazardous waste.<sup>2</sup> However, contrary to conventional solvent containing coatings, powder coatings must melt, flow, and fuse into a smooth film when heated.

Most thermoplastic powders are high molecular weight, semicrystalline polymers that melt flow to smooth films upon heating. Therefore, the crystalline melting temperature is a critical factor in the selection of thermoplastic powder coatings.

On the other hand, thermosetting powders primarily are low molecular weight, amorphous polymers whose glass transition temperature ( $T_g$ ) is elevated during curing polymerization. Consequently, the powder  $T_g$  influences physical and chemical stability during storage and rheological properties during production, as well as film formation and internal stress development in the cured film.<sup>3</sup>

Internal stress is known to increase proportionally with  $T_g^4$  and the Young's modulus<sup>5</sup> of a coating. Increases in pigmentation<sup>6</sup> and crosslink density<sup>7</sup> are known to increase  $T_g$  and Young's modulus, and, therefore, the internal stress within a coating composition. On the contrary, plasticization<sup>4</sup> increases flexibility and lowers  $T_g$ , thus effectively reducing internal stress. Stress formation emanates as a result of dimensional changes accompanying film formation, for example, coalescence during drying, shrinkage on curing, or differences in thermal expansion coefficients of the coating and the substrate.<sup>3,4,8</sup>

In most cases, powder coatings do not shrink excessively when cured, since little or no volatile organic compounds are liberated. Therefore, stress development in powder coatings is primarily attributed to high curing temperatures and high cured-film  $T_g$ s. The Williams, Landel, and Ferry equation<sup>9</sup> demonstrates that melt viscosity of a resin depends on the temperature difference above the  $T_g$  of a coating. Consequently, since the  $T_g$  of powder coating resins is normally high, elevated curing temperatures are required to achieve proper melt viscosity. Furthermore, the thermal expansion coefficients of the coating and the substrate are different, and



I he heat history of thermosetting powder coatings necessitates careful control of extrusion conditions. It is common industrial practice to minimize pre-reactions by rapidly cooling powder extrudates. To some degree,

the quenching process controls the formation of internal stress in the finished product. Similar stress development occurs when powder coated films are cured and cooled rapidly.

This study examines polyester-blocked isocyanate, epoxy-dicyandiamide, polyester-triglycidyl isocyanurate, epoxy-phenolic, and acrylic thermosetting powder coatings via differential scanning calorimetry to quantify annealing and quenching effects on the glass transition temperatures. As a part of this study, panels were coated and cured at 150°C for 30 min. Cooling was effected by either rapid quenching or controlled annealing. Thermal, mechanical, and X-ray diffraction analysis data are presented which corroborate a thermal history dependence on powder coating performance.

when cooled the thermal coefficient differences contribute to internal stress formation. While internal stress in a thermosetting coating can be abated by annealing above  $T_g^{10}$  the process confers long-range ordering to the crosslinked polymer and therefore, variations in potential overall performance properties. This study focuses on the effects of annealing and quenching in relationship to the macroscopic and microscopic properties of several powder coating types.

Presented at the 72nd Annual Meeting of the Federation of Societies for Coatings Technology, on October 12-14, 1994, in New Orleans, LA.

<sup>\*</sup>Department of Polymer Science, Hattiesburg, MS 39406-0076.









#### **EXPERIMENTAL**

#### **Materials**

Five commercially available, thermosetting powder coatings were used and included a polyester-blocked isocyanate (Sample A), an epoxy-dicyandiamide (Sample B), a polyester-triglycidyl isocyanurate (Sample C), an epoxy-phenolic (Sample D), and a thermosetting acrylic powder coating (Sample E). Samples A, B, C, and E were provided by Morton International and Sample D was received from Pratt and Lambert, Inc.

#### **Testing Equipment**

Differential scanning calorimetry (DSC) studies were performed with a Mettler DSC 30 measuring cell where the heating and/or cooling sequence was set between 0 and 300°C. For annealing experiments, each powder was subjected to heating and cooling cycles at the rate of 10°C/min. For the quenching experiments, the samples were heated at 10°C/ min and cooled at 100°C/min.

X-ray diffraction studies were performed with a Siemens Xray XPD-700P polymer diffraction system equipped with a two-dimensional position, sensitive air detector. All data was collected in the transmission mode with a sample to detector distance of 10 cm. After background subtraction, the diffractograms (intensity versus 20 plots) were obtained by radially integrating the two-dimensional plots.

All powder coatings were applied by a corona electrostatic spray technique onto two sets of five, 3 in.  $\times$  6 in.  $\times$  0.02 in., cold-rolled, low carbon, mild steel QD-36 panels. Before spraying, the panels were wiped with reagent grade methyl ethyl ketone to remove oil, grease, and other impurities. The coatings were cured at 150°C for 30 min. The quenching experiment was simulated by removing one set of five panels from the oven at the end of 30 min and cooling ambient temperature in approximately 2 min. Annealing conditions were simulated when the curing oven was switched to the off position and allowed to cool to room temperature over a 20 min

period. The cured film thicknessess were  $50 \pm 5 \,\mu\text{m} (2.0 \pm 0.2 \,\text{mils})$ . The coatings were allowed to equilibrate for seven days before testing.

Mechanical testing was performed via standard ASTM tests noted in *Table* 1. Impact resistance was determined with a BYK-Gardner impact tester, using a 1.82 Kg (4 lb), 1.27 cm (1/2 in.) diameter steel pin. Pencil hardness was expressed in terms of pencil leads of known hardness. Adhesion was assessed by applying and removing a pressure-sensitive tape over a crosshatch constructed by 11 cutting blades. Flexibility was measured by bending coated panels on a Paul N. Gardner, Model MN-CM conical mandrel.

#### **RESULTS AND DISCUSSION**

The thermal, mechanical, and X-ray diffraction analysis data are included in *Table* 1. Differential scanning calorimetric studies demonstrate that annealing the polyester-blocked isocyanate (Sample A), epoxy-dicyandiamide (Sample B), and epoxy-triglycidyl isocyanurate powder coating (Sample C), elevates their respective  $T_g$  by 2.7, 3.4, and 6.1°C (*Figures* 1ac). The epoxy-phenolic powder coating (Sample D) developed  $T_gs$  at 59.2 and 75.6°C when quenched, although annealing gave a single  $T_g$  at 76.3°C (*Figure* 1d). The thermosetting acrylic powder coating (Sample E) gave no change in  $T_g$ on either quenching or annealing, although the quenching process developed an endothermal relaxation (*Figure* 1e).

Wide angle X-ray studies were performed to determine thermal history effects on the microscopic properties of the cured powder coatings. *Figures* 2a-e represent the X-ray diffractograms (intensity versus 2 $\theta$  plots) for Samples A-E, respectively. In terms of absolute intensity value, the X-ray diffractogram of the annealed sample is an exact duplicate of the quenched sample, for all the powder coatings studied. For each powder coating, the diffractograms are offset vertically to demonstrate that cured samples (both quenched and annealed) are amorphous. Moreover, no significant change in microscopic structure (chain packing or short-range ordering) occurs when the samples are annealed, indicating that inter-

Table 1—Thermal, Mechanical, and 2	X-ray Analysis of Powder Coatings
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Sample	G	Glass Transition °C	X-Ray Analysis	Impact <sup>a</sup> Direct/Reverse (Joules)	Pencil Hardness <sup>b</sup>	Adhesion <sup>c</sup>	Flexibility <sup>d</sup> (cm)
A	Quenched Annealed		Amorphous Amorphous	<2.3/<2.3 <2.3/<2.3	B B	5B 5B	0.25 0.25
В	Quenched Annealed		Amorphous Amorphous	18.1+/18.1+ 18.1/15.9	HB F	5B 5B	<0.125 <0.125
С	Quenched Annealed		Amorphous Amorphous	18.1+/15.9 15.9/13.6	B HB	5B 5B	0.125 0.125
D	Quenched Annealed		Amorphous Amorphous	18.1+/18.1+ 15.9/13.6	HB F	5B 5B	<0.125 <0.125
E	Quenched Annealed		Amorphous Amorphous	18.1/<2.3 15.9/<2.3	F	5B 5B	<0.125 <0.125

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nal stress development upon quenching of the powder coatings is negligible.

Mechanical property testing further confirms the absence of significant internal stress development upon quenching. The thermal history of the polyester-blocked isocyanate powder coating (Sample A) does not affect its mechanical properties. However, annealing epoxy-dicyandiamide, polyestertriglycidyl isocyanurate, epoxy-phenolic, and thermosetting acrylic powder coatings (Samples B, C, D, and E, respectively) decreased direct and indirect impact resistance and slightly improved pencil hardness, while adhesion and flexibility remained unchanged.

The deterioration of impact resistance on annealing in Samples B, C, D, and E is attributed to increase in internal stress resulting from increased crosslink density arising during post-cure. The higher crosslink density increases the  $T_g$  of the powder coatings. Similar results have been reported for solvent-based thermosetting coatings.<sup>7,11</sup>

The data (Table 1) confirms a relationship between the mechanical properties and Tg differences for all the powder coatings studied. Annealing the polyester-blocked isocyanate powder coating (Sample Å) gives a  $T_g$  increase of 2.7°C, which is within experimental error,<sup>12</sup> indicating no significant increase in crosslink density and is consistent with the lack of variations in mechanical properties. However, changes in Tg for the epoxy-dicyandiamide (Sample B) and polyestertriglycidyl isocyanurate (Sample C) were 3.4 and 6.1°C, respectively, a difference attributable to alterations in crosslink density, and consequently changes in mechanical properties. The development of two T<sub>a</sub>s on quenching the epoxy-phenolic powder coating (Sample D) suggests an interruption in the crosslinking reaction giving rise to incomplete crosslinking, and therefore less internal stress development. On the other hand, when Sample D is annealed, the crosslinking reaction is completed and internal stress develops resulting in lower impact resistance. Similar results have been reported for a solvent based benzylcellulose-phenolic resin coating.13 Thermal analysis of the thermosetting acrylic powder coating (Sample E) shows an endothermal transition at the T<sub>g</sub> on quenching and no post-curing on annealing. Endothermal transitions at the Tg have been attributed to internal stress development.3 However, the resulting mechanical property variations are inexplicable at this time and will be the subject of further investigation.

#### CONCLUSIONS

The effect of annealing and quenching on performance properties for several powder coatings has been established. Rapid cooling (quenching) does not significantly alter the mechanical properties of powder coatings. However, impact resistance decreases when cured powder coatings are annealed. This effect becomes more evident as the  $T_g$  differences of quenched and annealed powder coatings increase. Wide angle X-ray diffraction studies indicate no significant change in the short-range ordering of polymer chains on annealing indicating negligible internal stress development on quenching. However, annealing allows post-curing reactions to occur which increase the crosslink density and therefore, induce internal stress in the thermosetting powder coatings.

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# Depth Profiling of Acrylic/Melamine Formaldehyde Coatings

G. Haacke, J.S. Brinen, and P.J. Larkin-Cytec Industries Inc.\*

# INTRODUCTION

The physical and chemical properties of thermoset coatings crosslinked with melamine formaldehyde resins have been extensively investigated.<sup>14</sup> In most of the published work, bulk coatings were studied. Less attention has been paid to the question of whether certain materials' parameters change with depth. This question, however, becomes important in coatings in which gradients of specific properties perpendicular to the surface could be of practical significance. For example, in two-layer coatings, one may want to have in the topcoat high surface hardness for good scratch resistance but a softer, more pliable region near the interface with the basecoat to reduce the likelihood of delamination.

In this paper, experiments carried out to follow the depth dependence of selected properties of typical automotive clearcoat layers are discussed. The parameters investigated include the triazine/styrene infrared absorbance ratio, the nitrogen/carbon ratio, the glass transition temperature (T<sub>o</sub>), and the crosslink density, XLD. To measure the depth dependences of these properties, the coatings were microtomed into thin sections using equipment and techniques described recently.<sup>5,6</sup> The parameters then were measured on each individual section. The nitrogen/carbon ratios were determined using X-ray photoelectron spectroscopy (XPS) and elemental analysis. The crosslink densities were derived from dynamic mechanical analysis (DMA). The T<sub>o</sub>s were measured using either DMA or differential scanning calorimetry (DSC) and the infrared absorbance ratios were determined from Fourier transform infrared (FTIR) spectra.

Depth profiling of polymeric materials using the microtome technique has been reported previously. The purpose of these earlier investigations was to understand the depth distribution of polymer additives, such as antioxidants or light stabilizers. In some cases, the distribution profiles were used to study additive diffusion in polymers.<sup>7-9</sup> Other authors investigated additive profiles to generate information concerning the light-stability of automotive coatings.<sup>5,9,10</sup> The work discussed in this paper is not concerned with the distribution of additives but with the depth dependence of certain physical and chemical properties of the polymer matrix. To our knowledge, this has not been reported before. The depth dependences of the glass transition temperature, the crosslink density, and the triazine/ styrene and nitrogen/carbon ratios have been investigated in acrylic/melamine coatings. Depth profiles were determined by using a microtome technique which produced slices large enough  $(5 \times 5 \text{ cm})$  to employ as analytical tools dynamic mechanical analysis, differential scanning calorimetry, and elemental analysis. The triazine/styrene ratios were derived from infrared absorption spectra. Depending on the cure conditions, gradients perpendicular to the coating surface were observed for the glass transition temperature, the crosslink density, and the triazine/ styrene infrared absorbance ratio. For 30 min cure time, these gradients existed in coatings cured below 160°C. The nitrogen/carbon ratios of the crosslinked resins, determined from elemental analysis and Xray photoelectron spectroscopy, stayed constant in samples in which the other parameters investigated changed with depth. The gradients of the glass transition temperature and crosslink density are believed to originate from a shift of the chemical reaction equilibrium of the crosslinking reaction. Trapped isobutanol, a reaction by-product, is assumed to be responsible for this shift.

Changes with depth of the chemical composition of coatings have been investigated by several groups using either chemical analysis,<sup>11,12</sup> or attenuated total reflectance (ATR), a non-destructive technique.<sup>13,14</sup> Utilizing ATR, for example, the authors of reference 14 derived the depth dependence of the melamine/polyester ratio in polyester/melamine coatings. Our experimental FTIR absorbance ratios, taken on acrylic/

Presented at the 72nd Annual Meeting of the Federation of Societies for Coatings Technology, on October 12-14, 1994, in New Orleans, LA. \*Research & Development, 1937 W. Main St., P.O. Box 60, Stamford, CT 06904-0660.

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melamine samples, are in agreement with the spectral results of this work. However, in contrast to polyester-type layers, our data do not indicate that the depth distribution of melamine is nonuniform. As we shall see later, in acrylic/melamine coatings, it is the crosslink density that changes with depth.

# **EXPERIMENTAL**

# **Coating Preparation**

All coatings investigated were of the same composition and consisted of an acrylic backbone (Joncryl 500) and a melamine crosslinker (Cymel® 1168) resin. Joncryl 500 resin (SC Johnson Co.) is a styrene acrylic oligomer with an average molecular weight Mn = 1300, a poly-dispersity Mw/Mn = 1.7, and a hydroxyl number of solids of 140. Cymel 1168 crosslinker (Cytec Industries Inc.) is a methylated-isobutylated melamine formaldehyde resin containing approximately 50 mol% methoxymethyl and 50 mol% iso-butoxymethyl functionality. The backbone to crosslinker ratio was 65:35 by weight. The OH/NOR ratio was 1:2.3. The catalyst was Cycat® 4040 p-toluene sulfonic acid.

The coatings were drawn onto  $10 \times 30$  cm (4 × 12 in.) Aluminum 3003 test panels purchased from Advanced Coating Technologies. A #60 wire-cator rod was used to achieve 50 micrometer (2 mil) layer thickness after cure. For some measurements, 100 micrometer thick coatings were required. These were drawn using a #1470 wire-cator rod. The wer coatings were equilibrated in air and subsequently cured in a forced-air convection oven.

### Microtoming

After cure,  $5 \times 5$  cm ( $2 \times 2$  in.) squares were cut from the test panels and the resin layers carefully lifted off the substrate with a sharp razor blade. The resin samples then were mounted

on the support block of the microtome with double-sided tape. The microtome was a Reichert-Jung Polycut E which is capable of cutting sections as thin as 1 micrometer. For most measurements, the section thickness was 4 or 5 micrometers. Thicker slices were required for DMA measurements as will be discussed later.

# **Analytical Techniques**

The crosslink density was obtained from DMA measurements using a Rheometrics solids analyzer RSA II. In this technique the sample is subjected to an oscillating strain over a temperature range including  $T_g$ . The instrument determines the resulting stress and the phase lag between stress and strain. From the maximum imposed strain, the maximum stress, and the phase lag, the DMA system calculates and plots as a function of temperature the three dynamic mechanical parameters storage modulus E', loss modulus E'', and loss tangent E''/E'. The crosslink density is calculated<sup>15</sup> from the relation

$$XLD = E'/(3RT)$$
(1)

where E' is the minimum value of the storage modulus in the rubbery plateau, R is the gas constant, and T is the absolute temperature at E'. For the measurements reported, the strain frequency was 10 Hz and the heating rate was  $5^{\circ}$ C/min.

The glass transition temperature was determined mostly from DSC scans. Although  $T_g$  can be derived from DMA as the temperature at which E''/E' reaches a maximum, DSC is less time consuming. In DMA it also can happen that the thin microtomed sections tear, necessitating the time-consuming preparation of new samples for repeat measurements. The DSC scans were made in a Perkin-Elmer differential scanning calorimeter Model DSC-4 with microprocessor controller and data station. The scans covered the temperature range between -40°C and +120°C at a rate of 20°C/min. The sample weight was kept close to 5





mg. The  $T_g$  was defined as the midpoint of the endothermic shift displayed by the DSC scan.

Elemental nitrogen/carbon ratios were determined by XPS measurements and by elemental analysis. XPS spectra were measured using an HP 5950A spectrometer with monochromatic Al K $\alpha$  radiation. The spectrometer has been modified with an SSL detector, aperture, and data system. Atomic surface compositions were determined using Wagner's<sup>16</sup> formalism and theoretical cross sections calculated by Scofield.<sup>17</sup> For the HP spectrometer, the percent atomic concentration may be expressed as

$$%A = \frac{I_A / \sigma_A}{\sum_X I_X / \sigma_X} \bullet 100$$
(2)

where  $I_A$ ,  $I_X$  are the intensities under the peaks of atom A and atom X, respectively, and the  $\sigma$ 's are the cross sections for the respective atoms. Atom X is either carbon, nitrogen, or oxygen. The thin microtomed slices were placed on gold plated sample mounts for examination. Survey and high resolution scans were obtained for each slice examined from which the nitrogen/carbon ratios were calculated.

The elemental composition of each microtomed section was also obtained using a Perkin-Elmer 2400 CHN elemental analyzer. This instrument subjects the sample to thermal combustion. The combustion products are analyzed for carbon, nitrogen, and hydrogen in thermal conductivity cells capable of 0.3% accuracy. This work was performed at Baron Consulting Co., Milford, CT.

The residual isobutanol concentrations of microtomed slices were measured by extracting the alcohol and analyzing the extract by gas chromatography. Extraction was carried out either by using supercritical carbon dioxide or acetonitrile. Both techniques yielded nearly identical results.

Information on the molecular structure of the crosslinked coatings was obtained from IR spectra. Spectral features pertaining to the styrene group of Joncryl (701 cm<sup>-1</sup>, 766 cm<sup>-1</sup>) or 3026 cm<sup>-1</sup>) and to the triazine ring of melamine (815 cm<sup>-1</sup>) were analyzed to determine the depth dependence of the acrylic/melamine absorbance ratio. The styrene and triazine moieties do not participate in the crosslinking reactions and can be considered a measure of the molecular composition of the coatings. Spectra were plotted using either a Digilab FTS-60 FTIR spectrometer or a Perkin-Elmer PE 1500 FTIR in-strument.

# **RESULTS AND DISCUSSION**

#### **Glass Transition Temperature**

The depth dependence of  $T_g$  was studied on 50-micrometer thick coatings cured for 30 min at one of the five following temperatures: 110°, 127°, 141°, 160°, and 170°C. After cure, the coatings were microtomed into 4 micrometer thick sections and analyzed by DSC. The results are plotted in *Figures* 1 and 2. In *Figure* 1, the  $T_g$  are shown as function of depth for three coatings cured at 127°, 141°, and 160°C, respectively. As expected, the absolute  $T_g$  values at a given depth increase with cure temperature. The straight lines through the data points are least squares fits. Their slopes vary with cure temperature, the largest change occurring at 127°C. No appre-



ciable variation of  $T_g$  with depth is seen at 160° and 170°C (not included in *Figure* 1). The dependence of  $T_g$  on depth for samples cured at 110°C is shown in *Figure* 2. Here the data are plotted for two samples cut from the same panel to demonstrate the reproducibility of these measurements. The slope and absolute value of  $T_g$  for 110°C cure are smaller than for 127°C, reflecting the fact that the lower temperature results in less chemical conversion. A summary of the  $T_g$  slopes versus cure temperature is presented in *Figure* 3.





The observed dependence of  $T_g$  on cure temperature and depth can be understood if we remind ourselves that  $T_g$  is a measure of the extent of chemical conversion taking place during the cure of thermoset systems. The glass transition temperature increases non-linearily with fractional conversion FC whereby the slope of the  $T_g$ -versus-FC curve changes by nearly one order of magnitude from the early curing stages until it reaches full cure.^{18} The steep slope prevailing at higher conversions facilitates the observation of depth dependent  $T_g$  changes.

The chemical reaction proceeding during cure is essentially the formation of crosslinks between the Joncryl 500 resin and the Cymel 1168 crosslinker. The general reaction scheme is shown in Figure 4a. The proton of the hydroxyethyl group of the hydroxyethylmethacrylate unit of the backbone resin reacts with the alkoxy-functionality of the crosslinker forming an ether linkage. If crosslinking occurs via the isobutoxymethyl group the reaction by-product is isobutanol which escapes from the coating as the crosslinking proceeds. The resulting migration of the isobutanol from the coating interior to the surface is significantly influenced by the cure temperature. We assume that at the higher cure temperatures, i.e., 160°C and above, the diffusivity of the alcohol and the segment mobility of the polymer chains are large enough to enable most of the isobutanol molecules to leave the coating. In the lower cure temperature range, however, the reduced diffusivity and smaller segmental chain mobility hinder the alcohol migration and the isobutanol inside the coating is likely to exceed the equilibrium concentration. As a consequence, to maintain the numerator of the reaction equilibrium constant K at constant value (see Figure 4b), the number of ether linkages at locations of higher isobutanol concentration will be smaller than under true equilibrium conditions. The final result is that the chemical conversion and, therefore, the T<sub>g</sub> are lower in the interior of the coating than close to the surface.

This discussion suggests that a residual concentration of isobutanol remains trapped inside the cured coating. In preliminary experiments we have attempted to prove this point by analyzing extracts of freshly prepared coatings by gas chromatography. Using either organic solvent or supercritical carbon dioxide, approximately 0.6 wt% isobtuanol were extracted from coatings immediately after being cured at 120°C.<sup>19</sup> The analysis of microtomed sections is in progress.

As we pointed out earlier, the Cymel 1168 crosslinker has approximately 50 mol% methoxymethyl functionality. The by-product of the crosslinking reaction of this functionality is methanol. The smaller size of the methanol molecule compared to isobutanol affords it a much higher diffusivity even at low temperatures and we would not expect to find significant concentrations of trapped methanol in the cured coatings.

Examination of Gillham's time-temperature-transformation cure diagram<sup>20</sup> uncovers a second potential mechanism for the formation of T<sub>g</sub> gradients in coatings. It follows from the iso-T<sub>g</sub> curves of this diagram that regions which are at a lower cure temperature require a longer time to reach a specified iso-Tg curve than regions exposed to a higher cure temperature. As a result, a temperature gradient existing in a coating during cure should lead to a Tg gradient. To determine whether under our experimental conditions a temperature gradient could exist for a time period comparable to the cure time, we have estimated from rate of heating equations how long it takes for a coating to reach temperature equilibrium from the time the coating is placed into the curing oven. The calculations show that this time is only a fraction of the 30 min cure employed in our work. Temperature gradients in the coatings do not exist long enough to contribute to the observed depth dependence of T<sub>o</sub>.

# **Crosslink Density**

A direct consequence of the chemical conversion during cure is the formation of crosslinks. As  $T_g$  increases so does XLD. Under conditions which lead to  $T_g$  gradients we also should observe XLD gradients. An analysis of the expected depth dependence of XLD must take into consideration that coatings having a  $T_g$  gradient are not fully cured and, there-



fore, are comprised of sol and gel fractions. The linear dependence of  $T_g$  on depth suggests that a simple rule of mixtures<sup>21</sup> applies:

$$T_{g} = w(s) \times T(s) + w(g) \times T(g)$$
(3)

where w(s) and w(g) are the weight fractions of the sol and gel fractions and T(s) and T(g) are the corresponding glass transition temperatures. Whereas both fractions contribute to  $T_g$ , the crosslink density depends only on the glass transition temperature of the gel content:<sup>18</sup>

$$XLD \sim T(g) - T(lp)$$
(4)

where T(lp) is the glass transition temperature of an infinitely long linear polymer. For T(s) we have:<sup>18</sup>

$$T(s) = Mn \times T(lp)/(Mn + const \times T(lp))$$
(5)

where Mn is the number average molecular weight. According to equation (5) T(s) is a non-linear function of Mn approaching T(lp) at high Mn. From *Figures* 1 and 2 we know that under our experimental conditions,  $T_g$  varies linearily with depth. Therefore, we conclude from equations (3) and (4) that XLD is not a linear function of depth. This conclusion follows from the rule that if a linear function  $(T_g)$  consists of the sum of two functions, where one is non-linear (T(s)), the second function also has to be non-linear.

To determine XLD, dynamic mechanical analysis was carried out on microtomed sections 10 micrometers thick. This thickness was needed to reduce to acceptable levels the likelihood of sample tear during scanning. The DMA samples were 5 cm long and 0.5 cm wide. Representative scans of the storage modulus of the top three sections of a coating cured for 30 min at 120°C are shown in Figure 5. Slice #1 is the first cut. The minimum value of E' clearly decreases with depth which according to equation (1) translates into a decrease of the crosslink density. The full depth dependence of XLD of a 100 micrometer thick coating cured 30'/120°C is plotted in Figure 6. The 100 micrometer thickness for this experiment was chosen to obtain a sufficient number of microtomed samples suitable for DMA. Frequently, the two sections cut next to the substrate are prone to tear and rarely yield useful DMA data. A 50 micrometer thick coating then would give three data points only.

The XLD versus depth curve of Figure 6 follows qualitatively the characteristics of  $T_g$  displayed in Figures 1 and 2. A noticeable decrease with depth is indicated. The major difference between  $T_g$  and XLD is that XLD is a non-linear function of depth which is not unexpected considering the discussion at the beginning of this section. Although the  $T_g$  versus depth results of Figures 1 and 2 were obtained on 50 µm thick coatings, we also have carried out these measurements on 100 µm thick coatings and observed the same linear depth dependence of  $T_g$ .<sup>22</sup> Therefore, it is valid to compare the curves of Figures 1 and 2 with the curve of Figure 6. In agreement with the  $T_g$  data, we found that at 160°C cure temperature the crosslink density remains constant through the complete sample thickness.

# Nitrogen/Carbon Distribution

The interpretation of the polyester/melamine data in reference 14 points to the possibility that a nonuniform melamine distribution perpendicular to the coating surface also could



occur to some degree in acrylic/melamine coatings. Measurement of the nitrogen/carbon ratio with depth should unambiguously demonstrate whether there is indeed an excess of melamine near the coating surface. Using XPS, carbon and nitrogen spectra were measured on 8-micrometer thick slices of a 100 micrometer coating cured at 30'/120°C. From the spectra the N/C ratio was calculated and found to be constant within  $\pm$  5% for all slices. This result was verified by C/H/N elemental analysis of the same coating used for XPS. The wt% of carbon and nitrogen and the N/C ratio determined by the latter analysis are summarized in Table 1. The table confirms that N/C is essentially constant over the coating thickness in agreement with the XPS results. The average value of N/C on a wt% basis is 0.0823  $\pm$  1.6%. The N/C ratios determined from XPS measurements are  $0.067 \pm 5\%$  which is 20% lower than the ratios obtained from elemental analysis. This difference is attributed to the fact that XPS analyzes only the top 10 nanometers of each section. Minor surface contaminants such as adsorbed water or gases may affect the XPS data. In contrast, elemental analysis integrates the entire thickness of a microtomed slice.

Table 1—Results of the Elemental Analysis of 8 µm Thick Sections of a Clearcoat Sample (100 µm thickness). Sample #1 is the First Cut from the Coating Surface; Sample #10 is the Section Closest to the Substrate

Sample	C (wt%)	N (wt%)	N/C Ratio
1	69.93	5.66	0.0809
2	69.69	5.66	0.0813
3	70.42	5.72	0.0812
4	70.11	5.68	0.0810
5	70.71	5.88	0.0832
6	69.05	5.74	0.0831
7	69.20	5.72	0.0827
8	69.25	5.79	0.0836
9	69.71	5.83	0.0836
10	68.84	5.67	0.0824

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# **Infrared Analysis**

Infrared transmission spectra were recorded of the same microtomed sections used for the DSC analysis. Figure 7 shows the absorbance band ratios versus depth of the triazine 815 cm<sup>-1</sup> band relative to the styrene 3026 cm<sup>-1</sup> band for coatings cured at either 127°, 141°, or 160°C. Comparison of Figures 1 and 7 shows that the triazine/styrene absorbance ratios have a similar depth dependence as observed for Tg. At lower cure temperatures the absorbance ratios decrease with increasing depth while at the highest cure temperature the ratio remains constant. An analysis of the individual peak heights corrected for section thickness variations reveals that only the intensity of the triazine band changes with depth while the styrene band remains constant.

Following Beer's Law, the depth dependence of the triazine/styrene absorbance ratio is a result of either variations in melamine concentration or in the triazine band absorptivity. Both XPS and elemental analysis demonstrated that the nitrogen/carbon ratio is constant over the coating thickness indicating a uniform distribution of melamine. Thus, the variation in the triazine/styrene absorbance results from depth dependent changes in the triazine absorptivity and not from an inhomogeneous distribution of melamine in the coating.

The depth dependence of the triazine/styrene absorbance ratio is most likely a consequence of the observed changes in crosslink density. The 815 cm<sup>-1</sup> band is due to the triazine outof-plane ring puckering vibration and is, therefore, sensitive to the conjugation of the triazine ring.<sup>23</sup> Our data indicate that the intensity of the 815 cm<sup>-1</sup> band increases with XLD. Crosslink density increases occur by reaction between the acrylic hydroxyl group and the melamine proton of the alkoxy group. We assume that this reaction twists the external carbon-nitrogen bond of the triazine ring (see Figure 4a) out of the triazine plane. This digression from coplanarity of the C-N group with the triazine ring may alter the triazine ring vibrations and lead to the observed absorbance changes. The triazine ring absorbance would be amplified by this process with increasing XLD. At the highest cure temperatures, XLD remains constant throughout the coating and no change of the triazine absorbance with depth occurs.

# SUMMARY

Microtoming is a powerful tool for probing the interior of coatings. Although this technique is destructive, it is effective for investigating depth dependent materials properties with high resolution throughout a coating. In contrast to earlier instruments, modern microtomes are capable of cutting largearea specimens which allow the utilization of analytical techniques requiring relatively large sample size such as DMA and elemental analysis. The DMA and DSC measurements have led to the recognition that crosslink density and glass transition gradients can exist in clearcoats. The results of the elemental analysis and XPS demonstrated that care must be exercised in the interpretation of measured variations in infrared absorbances. The assumption that intensity changes of IR bands are solely caused by concentration changes of the absorbing species was found not be applicable to the observations reported in this paper.

# ACKNOWLEDGMENTS

The authors are indebted to N. Colthup who suggested the model used for interpreting the IR data and L. Rosati for valuable discussions on polymer architecture. We also gratefully acknowledge the contributions of A. Cronin, who carried out sample preparation and many measurements, and N. Giannopoulos, for DMA measurements.

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# Interpretation of Corrosion Resistance Properties of Organic Paint Films From Fractal Analysis of Electrochemical Noise Data

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# INTRODUCTION

n the paint industry, accelerated laboratory tests are commonly used to assess the corrosion resistance properties of organic coatings. Since corrosion is an electrochemical phenomenon, electrochemical test methods are becoming widely adopted for this purpose. One such method, electrochemical noise analysis (ECN), can be used to measure the voltage and current fluctuations spontaneously produced by freely corroding metals.1-13

Previous work in this laboratory has focused on measuring and analyzing ECN voltage and current data for painted metal substrates in order to assess the corrosion resistance provided by various coatings.<sup>14-19</sup> ECN data were collected in the form of time records or sets of consecutive voltage or current observations. Methods for the analysis of ECN data have included statistical techniques and visual inspection of the time records. These previous studies have suggested that the ECN time records contain valuable information about corrosion control by coatings. However, the statistical and visual methods of analysis have not revealed information about the specific mechanisms of corrosion inhibition.14-19

Various other workers have analyzed the periodicity in the structure of ECN time records for uncoated metals by calculating its spectral density function (SDF), i.e., the square of its frequency transform. The slope of the SDF at higher frequencies typically has the form  $(1/f)^{\beta}$ . Different values of the exponent  $\beta$  have been reported for specific modes of corrosion, e.g.,  $\beta \ge 2$  implies pitting.<sup>4,6-12,20-24</sup> Alternatively, the structure of a time record can be analyzed in the time domain. The recent development of the subject of fractal geometry has been responsible for many advances in the analysis and characterization of time records. For example, the Hurst exponent,<sup>25-29</sup> H, (which is formally related to  $\beta$ )<sup>26,30,31</sup> reveals long-term time dependence in a time record and can be evaluated from the fluctuations occurring in the data. When the variation in the time record over a specific time interval (the lag time) is proportional to the lag time raised to the power 2H, the time record is said to be fractal. The parameter

Time records of electrochemical noise (ECN), i.e., spontaneous voltage and current fluctuations, were collected from coated metal panels immersed in an electrolyte. After 2,000 hr, the panels were rated visually for blistering and corrosion. Visual assessments and statistical analyses were found to be valuable for comparing the performance of the coatings during immersion. However, in order to elucidate mechanisms of corrosion and inhibition, the frequency structures of the time records were investigated. An established technique from fractal geometry was used to evaluate the Hurst exponent (H). The results ranged from  $2H \approx 0$  (coatings where little or no corrosion was observed to  $2H \approx 2$  (coatings where extensive corrosion activity was evident). The Hurst exponent can be used as an index of the level of corrosion resistance provided by a coating. In addition, H can be interpreted in terms of the color and persistence characteristics of the time records. This interpretation forms the basis of a description of the electrochemical processes which give rise to electrochemical noise.

Presented at the 71st Annual Meeting of the Federation of Societies for Coatings Technology, on October 28, 1993, in Atlanta, GA \*601 Canal Rd., Cleveland, OH 44113.

<sup>2</sup>H then describes both the appearance (the "jaggedness") of the time record and the characteristics (the "persistence") of the associated physico-chemical phenomenon, e.g., corrosion.26-29

In the present work, the objective was to determine whether information on mechanisms of corrosion and corrosion inhi-

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bition could be extracted from ECN data for painted metal. Panels were coated with eight different commercial organic coatings. Time records of voltage and current were collected during exposure to an aqueous electrolyte. At the end of the experiment, simple statistics, SDFs, and the Hurst exponent were evaluated from the noise records. From the results obtained, a model has been proposed to interpret the physical significance of the values of 2H in terms of the persistence of the electrochemical reactions.

# EXPERIMENTAL

# **Coatings Samples**

Descriptions of the eight coatings used in this study are given in Table 1. They were selected to represent a wide range of differences in both the mechanisms by which they function, and the extent to which they inhibit corrosion. For ex-

ample, the polyurethane-based coating contained a highly crosslinked resin system that was expected to prevent an electrolyte from coming rapidly into contact with the metal substrate. In other cases, it was anticipated that active pigments (such as zinc chromate) would inhibit corrosion.14-16

Each sample substrate consisted of a 3 in.  $\times$  6 in. mild steel (SAE 1010) test panel. These steel panels were cleaned by



# Table 1-Description of Coatings Tested in This Study

Binder	Corrosion Protection Mechanism <sup>a</sup>	PVC (%)	Dry Film Thickness (µm, +/-10%)
Polyurethane	Barrier No active pigment	25	40
Epoxy-polyamide (1)	Active pigment: Zinc phosphate (10 wt%)	35	100
Epoxy-polyamide (2)	Active pigment: Strontium chromate (6 wt%)	32	36
Vinyl-chloride vinyl-acetate copolymer		39	51
Alkyd (1) (linseed oil)	Barrier No active pigment	40	75
Alkyd (2) (tofa-phthalic)	Barrier No active pigment	53	36
Acrylic-latex	Active pigment Barium metaborate (11 wt%)	28	75
Alkyd (3) (soya-pentaerithritol)	Active pigment Zinc chromate (0.1 wt%)	42	30

first rinsing with deionized water and then with methyl ethyl ketone, to ensure surface uniformity. The coatings were applied with an automated air spray apparatus. Coated samples were allowed to cure for several weeks in a room where the air was filtered, and the ambient conditions were maintained at 21°C and 45% relative humidity.

# **Electrochemical Noise Apparatus**

The electrochemical noise apparatus has already been described in detail.<sup>14,15</sup> Briefly, a plexiglass cell was attached to each coated panel (electrode) with silicone adhesive and filled with electrolyte solution (0.6 M NaCl). A surface area of approximately 39 cm<sup>2</sup> of each coating was exposed to the electrolyte in each cell. Nominally identical pairs of electrodes were coupled electrically through the measurement instrumentation and electrolytically by an agar salt-bridge. A reference electrode (Ag/AgCl) was placed in one of the cells for each pair of electrodes. This experimental cell arrangement is illustrated in Figure 1a. Eight pairs of coupled electrodes were connected to an IEEE programmable switch controlled by a Hewlett-Packard 85 desktop computer. Two sensitive digital volt meters (HP 3478A) and a custom-built, low-noise, zero-resistance ammeter were used to measure the simultaneous voltage and current fluctuations for each coupled electrode pair (Figure 1b). Measurements are made of the voltage for a coupled pair of electrodes with respect to the reference electrode, and of the "coupling current" that flows between the two electrodes.18

# Data Collection

Data were collected in the form of time records or sets of instantaneous measurements of voltage and current for each

electrode pair. For a given pair of electrodes, voltage and current readings were recorded every 16 sec. The computercontrolled instrumentation cycled repeatedly among the eight electrode pairs, switching from one pair to the next pair every two seconds. These time intervals were chosen because corrosion processes under coatings were expected to be fairly slow. Each voltage and coupling current time record was limited to 64 measurements by the memory capacity of the computer in this experimental arrangement.<sup>14,15</sup> It required 1,024 sec or approximately 17 min to obtain each complete time record for all eight electrode pairs.

The initial time record was collected during the first 17 min after the coated electrodes had been exposed to the electrolyte. Subsequently, time record data sets were obtained on an hourly basis, and then less frequently over the following days and weeks. The immersion experiment was allowed to continue for approximately 2,000 hr. At this time, the cells were dismantled and the coated panels were inspected visually for blistering and rust according to appropriate ASTM procedures.<sup>32</sup> The noise data files were transferred from the HP-85 to a VAX 3100 scientific workstation for manipulation and analysis.

# **RESULTS AND DISCUSSION**

### **Rating of Painted Electrodes**

The corrosion protection afforded by the coatings in this study was first assessed by visually rating the appearance of the painted electrodes at the conclusion of the immersion test. Four examples of the range of performance observed are shown in *Figure 2*. Standard ASTM<sup>32</sup> methods were used to evaluate the degree of surface rusting (ASTM D 610-85), the extent of paint blistering (ASTM D 714-87), and the general appearance of the painted surfaces (ASTM D 1654-92). For each method, the ratings ranged from 10 (best) to 1 (worst). Using the format [D 610; D 714; D 1654], the respective ratings for the examples shown were: (a) epoxy-polyamide (2) [10;10;10]; (b) vinyl-chloride/vinyl-acetate [5;4-medium;8]; (c) alkyd (3) [1;4-dense;1]; and (d) acrylic-latex [3;2-dense;5]. The blisters ranged from relatively small and uniform in size (Figures 2a and 2b), to somewhat larger (Figure 2c) and more distended (Figure 2d) morphologies. The difference in appearance of the blisters indicates that the blister growth processes varied for these coatings. Moreover, Figure 3 shows two examples of the corroded electrode surfaces after the coatings were stripped away. Many small corrosion pits were formed under the alkyd (3) paint (Figure 3a), but a smaller number of larger pits developed beneath the acrylic latex (Figure 3b). The contrasting appearances of these substrates at the end of the experiment reflect differences in the corrosion-inhibiting behavior of these coatings. In order to assess coating performance during the course of the experiment, it was necessary to analyze the time records of ECN data that had been collected intermittently during the immersion test.

# **Visual Inspection of ECN Time Records**

Examples of voltage and current time records for three of the test paints are shown in *Figures* 4-6. These results are representative of the following: a paint with consistently good corrosion resistance properties and a lack of visible degrada-





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tion (epoxy polyamide [2], Figure 4); a paint with good corrosion resistance but slight degradation (vinyl-chloride/vinylacetate copolymer, Figure 5); and a paint with modest corrosion resistance and extensive blistering under these immersion test conditions (acrylic latex, Figure 6). Representative time records are shown from the early (6 hr immersion) and late (approximately 1,536 hr) stages of the 2,000 hr experiment.

The general appearance or structure of the voltage and current time records appeared to differ for the various coatings. In the case of the epoxy-polyamide, which protected the substrate from corrosion, the time records were rather jagged throughout the experiment (Figures 4a and 4b). Relatively high-frequency fluctuations were also observed for the vinylchloride/vinyl-acetate copolymer, but only during the early stages of exposure (Figure 5a). After the coating began to degrade, the fluctuations in the time records exhibited some lower frequency undulations (Figure 5b). For the acrylic latex, the time records seemed rather smooth even after only a few hours of immersion (Figures 6a and 6b). In general, for all eight coatings in this study, relatively high frequency fluctuations corresponded to effective protection against corrosion,



while relatively low frequency fluctuations signaled the inability of the coating to prevent corrosion. In order to substantiate this result, a more quantitative analysis of the time records was carried out.

# Statistical Analysis of ECN Time Records

Statistical methods were found to be particularly useful for the analysis of ECN data in previous studies.<sup>14,15</sup> In this work, each time record consists of N consecutive measurements:

$$X_1, X_2, X_3, X_4, \dots, X_N$$
 (1)

of voltage (V) or current (I) data. The expected value of the mean ( $X_{AV}$ ) of a time record can be calculated from:

$$X_{AV} = \{1/N\} \sum_{i=1,...,N} X_i$$
 (2)

Furthermore, the expected value of the variance  $(\sigma_{X}{}^{2})$  can be evaluated from:

$$\sigma_{X}^{2} = \{1/N\} \sum_{i=1,...,N} (X_{i} \cdot X_{AV})^{2}$$
(3)

and the expected value of the standard deviation obtained from:



$$\sigma_{\rm X} = \{\sigma_{\rm X}^2\}^{1/2} \tag{4}$$

This statistical analysis also includes the estimation of the derived noise resistance,  $R_{N^{\lambda}}$  in terms of the standard deviations of the voltage and current data,

$$R_N \sim \sigma_V / \sigma_l$$
 (4)

from a definition proposed elsewhere in the literature.<sup>1,19</sup>

Results from the statistical analysis were plotted against the time elapsed during exposure to the electrolyte. The following figures are representative of the eight paints listed in *Table* 1 and include the results for the coatings whose time records are pictured in *Figures* 4-6. The mean voltage (*Figure* 7) tended to remain relatively "noble" (more positive) for coatings that appeared to inhibit corrosion. However, in cases where corrosion and coating degradation were observed at the end of the experiment, the mean voltage typically decreased during the experiment (often approaching a limiting value of approximately –650 mV (Ag/AgCl), which is similar to the value anticipated for bare steel corroding under the same conditions). Variations in the mean voltage in *Figure* 7 are comparable to those published elsewhere in the literature.<sup>33</sup>

Mean coupling current values\* tended to increase in magnitude as coating degradation and corrosion progressed (*Figure* 8). Typical values ranged from approximately 10<sup>-11</sup> A/cm<sup>2</sup> (for the best coatings) to values of the order of 10<sup>-6</sup> A/cm<sup>2</sup> in the less protective cases. In general,  $\sigma_V$  decreased as paint failure took place, while  $\sigma_I$  tended to increase for certain coatings and showed relatively little variation for others. Calculated values for  $R_N$  are given in *Figure* 9. These four coatings ranged from good (high  $R_N$ ) to very modest (low  $R_N$ ) corro-

\*The absolute value of the mean coupling current is reported in this work since the "sign" (positive/negative) indicates merely the relative direction of the current flow between two nominally identical coupled electrodes.







sion resistance. During exposure to the electrolyte, the  $R_N$  values progressively decreased. Among the statistical results,  $R_N$  appeared to provide the most clear and reliable ranking of coating performance.

These statistical results were valuable for the direct comparison of all eight coatings in this study. However, neither the visual assessment nor the statistical analysis of the ECN time records provide direct mechanistic information regarding inhibition, coating degradation, and corrosion. Further analysis of the frequency structure of the ECN time records was deemed necessary to access this information.

# ANALYSIS OF ECN TIME RECORDS IN THE FREQUENCY DOMAIN

The frequency structure of a time record can be obtained from its spectral density function (SDF). The SDF may be calculated from the square of the Fourier or maximum entropy<sup>24</sup> transform of the time record, which is then divided by the bandwidth (in Hz).\* Log-log plots of the SDF versus frequency

\*In the ECN literature, the SDF is sometimes referred to as the "power spectral density" or PSD.<sup>3,8</sup> In some studies, the square root of the SDF is reported instead, and is referred to as the "spectral density of the amplitude,"<sup>11</sup> the "noise voltage density,"<sup>16</sup> the "noise amplitude,"<sup>12,22,23</sup> or the "amplitude.<sup>34</sup>

Figure 10—An example of a regular fractal, the Sierpinski gasket, whose structure resembles a nested pattern at various levels of magnification.



for uncoated electrodes typically exhibit a level "plateau" at low frequencies, with a cross-over to a "roll-off" slope at higher frequencies. Various authors have suggested that the cross-over frequency and the steepness of the roll-off slope correlate with the mode of corrosion activity observed on uncoated metals. In the present work,  $\beta$  has been introduced as the notation for steepness of the roll-off slope, i.e.,  $(1/f)^{\beta}$ . Other authors have instead used the notation  $\alpha$  for the high-frequency slope of the SDF of ECN time records. However,  $\beta$  is more consistent with the conventions of fractal geometry and the literature for many other cases of 1/f-noise, e.g., electronic noise.<sup>26-28</sup> Since the coupling current is (presumably) generated by fluctuations in the corrosion current, it is reasonable to expect that there is a correlation between  $\beta$  and the mode of corrosion activity at the electrolyte/metal interface on coated metal as well.

In this work, the SDFs for selected voltage and current time records were calculated<sup>34</sup> with a commercial fast Fourier transform routine.<sup>\*</sup> However, these SDFs were limited to a narrow frequency range because the time records were relatively small (only 64 observations versus 1,024 used by others<sup>24</sup>). There was also a considerable degree of scatter in the SDFs for the same reason. Thus, it was not possible to evaluate  $\beta$  unambiguously because each SDF resembled only a fragment of the form described (i.e., plateau, cross-over, and roll-off). However, since the ECN time records obtained in this work visually resembled the form of many fractal time records already published in the literature, an alternative analysis was carried out using some techniques of fractal geometry.

# FRACTAL ANALYSIS OF ECN TIME RECORDS

# Fractal Geometry and the Hurst Exponent

The development of fractal geometry by Mandelbrot<sup>27</sup> has provided a description and a mathematical model for many apparently complex forms found in nature. Objects with "complex structure" of many nested levels of detail that become visible at increasing scales of magnification are termed fractals. This structure is readily apparent in the regular fractal called the Sierpinski gasket (Figure 10). Many naturally occurring objects (coastlines, clouds, etc.) with similar (but not identical) levels of detail are random fractals. Time records of fluctuating quantities such as the population of a species, stock market values, and the output of certain electrical devices, may also be fractals.<sup>26-28</sup> When such a time record is a fractal, its "average" structure is independent of the time (or frequency) scale. For example, when a recording is made of an audible noise which is a fractal (e.g., white noise), the pitch of the noise would be unchanged when it is played back at various speeds. The noise will "sound the same" after the volume is adjusted.<sup>27-28</sup>

One method for analyzing the structure of a time record is by using the Hurst<sup>25-29</sup> exponent,  $H.^{\dagger}$  Mandelbrot<sup>27</sup> demonstrated that H characterizes the statistical relationship be-

<sup>\*</sup>Mathematica<sup>®</sup>, Wolfram Research, Inc., Champaign, IL.

<sup>&</sup>quot;This parameter was named after a British civil servant who studied the fluctuations of the water-level of the Nile River while helping to plan the Aswan High Dam. In his "rescaled range" analysis, the results for H revealed an unexpected long-term dependence in the annual discharge of the river, the result of which is extended periods of drought and flooding.



Figure 11—Log-log plots of  $(\Delta V(m\delta t))^2_{AV}/(\Delta V(\delta t))^2_{AV}$  and  $(\Delta l(m \delta t))^2_{AV}/(\Delta l(\delta t))^2_{AV}$  versus lag time "m $\delta t$ " seconds for the epoxy-polyamide (2) coating which provided good protection against corrosion. Results were calculated from time records of (a) current and (b) voltage ECN data (equation (8)) for the following periods of exposure: ( $\blacktriangle$ ) 1.5 hr; ( $\blacksquare$ ) 5hr; ( $\square$ ) 28 hr; (X) 504 hr.



Figure 12—Log-log plots of  $(\Delta V(m\delta t))^2_{AV}/(\Delta V(\delta t))^2_{AV}$  and  $(\Delta I(m \delta t))^2_{AV}/(\Delta I(\delta t))^2_{AV}$  versus lag time "m $\delta t$ " seconds for the alkyd, (2) coating which provided intermediate/modest protection against corrosion. Results were calculated from time records of (a) current and (b) voltage ECN data (equation (8)) for the following periods of exposure: ( $\blacktriangle$ ) 1.5 hr; ( $\blacksquare$ ) 5hr; ( $\square$ ) 28 hr; (X) 504 hr.



periods of exposure: (▲) 1.5 hr; (■) 5hr; (□) 28 hr; (Ⅹ) 504 hr.

tween two values of the time record separated by a time increment. If there is a fixed time interval  $\delta t$  between consecutive elements in a time record, then the "average" relationship between values  $X(\{i+m\}\delta t)$  and  $X(i\ \delta t)$  that are separated by a "lag time" m  $\delta t$  can be evaluated as follows:

$$[\Delta X(\mathbf{m} \ \delta \mathbf{t})]^2_{\mathrm{AV}} = \{1/(\mathrm{N} \cdot \mathbf{m})\} \sum_{i=1,\dots,\mathrm{N} \cdot \mathbf{m}} \{X(\{i+\mathrm{m}\}\delta \mathbf{t}) - X(i \ \delta \mathbf{t})\}^2 \\ \alpha [\mathbf{m} \ \delta \mathbf{t}]^{2\mathrm{H}}$$
(6)

In circumstances where a log-log plot of  $[\Delta X(m \ \delta t)]^2_{AV}$  scales linearly with the lag time, then 2H is the scaling exponent and the time record itself is a fractal. A fractal time record is also characterized by its fractal dimension  $d_{f_{t}}$  which can be calculated from the scaling exponents as follows:<sup>26,27</sup>

$$d_f = 2 - H = (5 - \beta)/2 \tag{7}$$

### Evaluation of the Hurst Exponent

Equation (6) was evaluated from the voltage and current time records obtained for all eight coatings in this work. Representative log-log plots from three of the paints (good, intermediate, and modest corrosion protection) are shown in *Figures* 11-13. For ease of presentation, the results of this analysis were normalized as follows:

$$[\Delta X(m \, \delta t)]^2_{AV} / [\Delta X(\delta t)]^2_{AV}$$
(8)

As can be seen in Figures 11-13, each log-log plot approaches a linear form with a slope 2H that describes the dependence of  $[\Delta X(m \ \delta t)]^2_{AV}$  upon the lag time. In other words, since the scaling relationship in equation (6) is satisfied, the ECN time records are fractals.

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For each of the eight coatings, similar values of 2H were obtained for both the current and voltage time records. A strong correlation was also observed between 2H and the visual assessments of the coated panels after testing, as in *Figures* 2 and 3. In general, for all of the paints in this study, higher values of 2H were observed when a coating provided less effective resistance to corrosion. The results for all eight coatings from this study can be summarized as follows:

(1) 2H = 0.0.5 for coatings that provided good protection against corrosion [epoxy-polyamide paints (1) and (2), and the polyurethane];

(2) 2H was initially low and increased to 0.5-1.5 during exposure for the coatings that afforded an intermediate level of protection [vinyl-chloride/vinyl-acetate copolymer, and alkyds (1) and (2)]; and

(3) 2H = 1.5-2 for the coatings that offered relatively modest corrosion protection [acrylic latex and alkyd (3)].

The accuracy of 2H here is limited by the relatively small size of the ECN time records used in this work. In addition, the evaluation of 2H (or  $\beta$ ) can be affected when the ECN fluctuations are superimposed on a steadily increasing (or decreasing) dc voltage or current.<sup>28,35</sup> However, subtracting a linear or quadratic fit from the time record<sup>24</sup> can also bias the analysis of 2H.<sup>35</sup>

In this study of eight paints, the Hurst exponent ranged in value from 2H  $\approx$  0 (relatively jagged noise) to 2H  $\approx$  2 for smoother, undulating noise. This is consistent with the formal relationship between 2H and  $\beta$ , which describes the relative contributions of low- and high-frequency periodic components in the frequency domain. That is, higher values of these exponents indicate a predominance of relatively

lower frequency fluctuations and, consequently, a time record with smoother structure. The results for the Hurst exponent demonstrate that the ECN time records are fractals. Thus, the observed relationships between 2H, the structure of the time records, and coating performance can be discussed from the perspective of fractal geometry.

# Fractal Interpretations of H and Corrosion Inhibition

The structure of a time record can also be described in terms of its "color," in analogy to the color of visible light. White light and noise contain equal contributions of components at all frequencies. Darker colors have fewer contributions from higher frequency components. Lower values of 2H indicate the presence of higher frequency contributions to a time record, and a lighter color. A few specific cases have been studied extensively, e.g., brown noise (2H = 1) is characteristic of a "random" process such as Brownian motion. However, as in a rainbow, there is a continuous spectrum of colors of noise. At the present, the infinitely many shades of noise can be understood only in terms of Mandelbrot's model of "fractional Brownian motion."<sup>26,27</sup>

Fractional Brownian motion can be considered as a generalization of the well-known case of Brownian motion (Appendix 1). Fractional Brownian motion is simply the extension of Brownian motion (brown noise) to cases where there is a long-term relationship among the increments. The increments of persistent noise (2H>1) tend to accumulate, while those of anti-persistent noise (2H<1) tend to alternate and cancel one another. The overall result is that persistent noise has a relatively smooth or undulating appearance (as in *Figure* 6), while antipersistent noise appears rough or jagged (for example, *Figure* 4).

According to the model of fractional Brownian motion, the value of 2H measures the persistence (or antipersistence) in the increments of a time record. This persistence is presumably related to the processes (chemical, physical, etc.) that are responsible for generating the time record. In this study, the appearance of the ECN time records, as well as the values of 2H, are thought to be manifestations of the electrochemical reactions that take place at the electrolyte/substrate interface and which can give rise to corrosion. If so, then this is the reason that 2H is sensitive to the relative level of corrosion resistance provided by the various coatings.

Corrosion begins when an aggressive ion attacks a vulnerable site on the surface of a metal substrate.<sup>36</sup> Apparently, a corrosion pit spreads gradually as aggressive ions attack and oxidize adjacent sites on the metal substrate.<sup>36,37</sup> On the basis of this model for uninhibited pit growth, it is expected that 2H = 2 and the ECN time records would be strongly persistent. Inhibition of pit growth, however, would be expected to reduce the persistence of the electrochemical noise (2H<2). Mechanisms of inhibition might include passivation or the restriction of the diffusion of aggressive ions to the substrate by a "barrier-type" coating. This qualitative model is consistent with the results of this study, where the voltage and current underwent relatively high frequency fluctuations (strongly antipersistent noise,  $2H \approx 0$ ) when a coating provided a high level of protection against corrosion. Thus, the analysis of fractal time records of ECN data with the Hurst exponent shows prom-

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ise as a means to access information concerning specific mechanisms of corrosion and inhibition, as well as providing an indication of coating performance.

# SUMMARY

Time records of electrochemical noise (measurements of voltage and current fluctuations) were collected while coated steel panels were immersed in an electrolyte solution. The appearance of these time records corresponded qualitatively to the extent of the blistering and corrosion observed after 2,000 hr of immersion for the different coatings. The time records that were obtained intermittently during immersion were then analyzed with statistical techniques. In particular, the "derived noise resistance" was valuable for comparing the protection provided by the coatings. However, these results did not provide direct information regarding the mechanisms of corrosion and corrosion inhibition.

Visual inspection of the noise data suggests that mechanistic information may be contained in their frequency structure (jagged versus undulating appearance). Therefore, fast Fourier transforms were used to calculate the spectral density functions of the time records. However, the results obtained for these relatively small sets of data (64 observations per time record) were too limited to allow a meaningful comparison of the structure of the time records for the different coatings.

The voltage and current data appeared to resemble time records that are known to be fractals. Therefore, these electrochemical noise time records were analyzed with an established technique from fractal geometry to evaluate the Hurst exponent (H). The results ranged from  $2H \approx 0$  (coatings where little or no corrosion was observed) to  $2H \approx 2$  (coatings where extensive corrosion activity was evident). Intermediate values of 2H were obtained for coatings that provided a moderate level of protection against corrosion. The Hurst exponent showed promise as an index of the level of corrosion resistance provided by each organic coating.

Fractal geometry also provides a basis for a more detailed interpretation of the frequency structure of electrochemical noise data. The results for 2H can be considered in terms of "color" and "persistence" characteristics of the time records. In turn, this allows the initial development of a mechanistic model to describe the electrochemical processes which give rise to electrochemical noise.

# ACKNOWLEDGMENTS

The authors would like to thank the management of The Sherwin-Williams Co. for supporting the research described here and for permission to publish this work. They also acknowledge the technical support provided by their colleagues Guy Wilson, Carl Shields, Brian Mysza, and the staff of the Technical Information Services Department (Sherwin-Williams). Finally, the authors are grateful for the insightful contributions made by David Eden (CML, Ltd., UK).

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# Appendix 1—Fractals

The recent development of fractal geometry by Mandelbrot<sup>27</sup> has provided new mathematical methods for the attack of previously intractable problems, such as the analysis of the structure and scaling exponents of fractal time records.<sup>27,37</sup> A "fractal" is an object with "complex" structure, or many nested levels of detail that become visible at increasing degrees of magnification, e.g., the Sierpinski gasket in *Figure* 10. An ECN time record is a "random" fractal, where the levels of detail are similar but not identical (i.e., they share the same statistical properties). The "fractal dimension"  $d_f$  describes the structure of a fractal, e.g., the "roughness" of an ECN time record.

The fractal approach is essential to this study because it provides the only substantive explanation for the values of  $d_t, 2H,$  and  $\beta$  that are observed for some of the coatings. Specifically, the fractional Brownian motion (fBm) technique of Mandelbort^{27,29} provides a connection between the structure of the time record (characterized by 2H,  $\beta$ , and  $d_f$ ) and the microscopic behavior (oxidation reactions) responsible for corrosion. Fractional Brownian motion is a generalization of "Brownian motion" that is observed under a microscope when a pollen grain in a liquid undergoes a "random walk" of step like random displacements. The mean squared variation of the position of the particle (or the mean squared displacement) is proportional to the lag time raised to the power 2H where 2H = 1. In fBm, 2H > 1 when there is positive correlation (persistence) among the displacements, and 2H < 1 on account of negative correlation (antippersistence).

In summary, the values of 2H,  $\beta$ , and d<sub>f</sub> are the consequence of the degree of persistence among the increments of the noise, which is rooted in the underlying microscopic physico-chemical processes. The inhibition of corrosion by a coating is characterized by the degree of anti-persistence in the ECN time record, which is measured by 2H, etc.

# Effects of Coating Solvents on the Morphology of Thermoplastic Polyolefins (TPO)

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# INTRODUCTION

The consumption of finished plastics in the United States reached an estimated 2.3 billion pounds in 1992.<sup>1</sup> Surveying industrial usages, the transportation industry accounted for roughly 5.7% of the total dollars consumed (Figure 1).<sup>2</sup> Of the types of plastics utilized in transportation, more specifically in the automotive industry, polypropylene (and its alloys) accounts for 19.7% of the consumption (Figure 2). With the increasing use of polypropylene and its alloys, it becomes increasingly important to understand its paintability.

The paintability of thermoplastic olefins (TPO) was found to vary depending upon type of TPO utilized and type (including bake) of coating applied. We studied the effect of chlorinated polyolefin (CPO) solvent-borne adhesion promoters applied as thin films (0.3 mils dry film thickness) which were subsequently topcoated with two-component (2K) polyester/isocyanate basecoat/clearcoat. We noticed within our studies that adhesion failures could manifest themselves as loss of paint adhesion to the substrate or as a loss of cohesive integrity within the painted plastic. We began to suspect that differences in type of adhesive/cohesive loss of paint and/or substrate, respectively, could be attributed to variances in the surface morphology of the TPO material. For that reason, we began to investigate the role of TPO surface morphology on subsequent paint adhesion, and the role that coating solvents and bake temperatures played on resultant adhesive/cohesive properties.

Thermoplastic olefins are blends of polypropylene and rubber with additional fillers and additives. The polypropylene utilized is a semi-crystalline polymer that has a very low surface free energy and is, therefore, difficult to paint. Rubber, usually an amorphous ethylene-propylene or ethylenepropylene-diene copolymer, is blended with the polypropylene to aid in paintability, as well as some physical properties such as low temperature impact. Additives include thermal and process stabilizers, ultraviolet stabilizers, pigments, and fillers.

When TPO is injection molded, a layering of the copolymers within the substrate occurs near the surface due to shearing forces and thermal gradients induced during the molding process (methods utilized to determine the surface structure of TPO will be discussed later in this paper). The

Adhesion to thermoplastic olefin (TPO) substrates is strongly influenced by the type and amount of solvent contained within paint applied. Morphological changes in the TPO substrate are accomplished in the presence of solvent from the coating and vary depending on paint bake times and temperatures. This paper attempts to delineate the influence of paint and paint processes on the adhesion/cohesion and mechanical properties of coated TPO parts. Initial adhesion to TPO appears to be a function of polypropylene crystallinity at the surface only as it relates to the ability of solvents to permeate through and into the rubber-rich layer beneath it. The amount of swelling of the rubber-rich layer can result in a lowered cohesive integrity of the top TPO layer. One must carefully select the correct solvent balance contained within the paints applied to TPO. It is essential to attain swelling within the substrate to afford initial paint adhesion, but the solvent must evaporate and the rubber shrink (deswell) to attain cohesive integrity of the substrate once painted.

surface morphology of TPO (Figure 3) can be depicted as a layering of polypropylene-rich regions of varying crystallinities beneath which lies a rubber-rich layer. If we refer to these layers of varying crystallinity and composition as "boundary" layers, we can begin to ascribe to each of these "boundaries" their influence on the paintability of the TPO and the resulting cohesive strength attained in the substrate once painted.

The polypropylene crystalline boundary is created as a result of nucleation generated by porous irregularities in the molding surface. The polypropylene begins to nucleate and crystallize nonideally due to the rapid cooling effects seen at the "cool" mold surface from the injection melt. The polypro-

Presented at the 21st Annual Waterborne, Higher-Solids, and Powder Coatings Symposium in New Orleans, LA. "Plastics & Trim Products Div., 24300 Glendale Ave., Detroit, MI 48239.







pylene directly beneath the crystalline boundary can crystallize in a more ideal fashion, e.g., in larger crystal spherulites, due to the delocalization of rapid "cooling" as exhibited on the molded surface.

Rubber-rich areas form directly beneath the polypropylene regions. The rubber is often elongated due to the shear forces present in the injection molding process. It is important that the rubber-rich boundaries lie somewhat near the surface since it is believed that this region accounts for the "paintability" of the TPO. In fact, the same TPO blend when compression molded (no shear forces or thermal gradients are imposed on the substrate in this molding operation) afforded no evidence of layering. In the compression molded TPO, it was very difficult to attain even initial adhesion of the paint to the substrate, since the rubber-rich layer had never formed near the surface.

It is postulated that diffusion of "chlorinated polyolefin solvent-borne adhesion promoter" through the polypropylene boundaries with subsequent "mechanical interlocking" with the rubber accounts for adhesion to the substrate.<sup>3</sup> The composition (including crystallinity) and thickness of these varying boundary layers, therefore, will have a tremendous impact on the paintability of the TPO. Thick, highly crystalline boundaries of polypropylene will tend to retard diffusion and, therefore, result in poor adhesion. Thin, amorphous polypropylene boundaries, on the other hand, will aid in swellability and diffusion of the adhesion promoter and its solvents and, therefore, increase paintability. The impact of rubber distribution within the rubber-rich boundary will play a vital role in initial paint adhesion, as well as in the cohesive strength of the painted TPO composite.

Adhesion to TPO substrates is, therefore, strongly influenced by the type and amount of solvent contained within the applied paint. Morphological changes in the TPO substrate are accomplished in the presence of solvent from the topcoat and vary depending upon paint bake times and temperatures. These morphological changes at and near the surface of TPO affect not only the paint adhesion to the substrate but also the cohesive integrity of the painted plastic composite. This paper attempts to delineate the influence of paint and paint processes on the adhesion/cohesion and mechanical properties of coated thermoplastic olefin parts.

# **RESULTS AND DISCUSSION**

In order to understand the influence of paint and paint processing, it is necessary to understand how the morphology of the TPO substrate changes under varying conditions. The utilization of known techniques, e.g., differential scanning calorimetry (DSC), as well as the "novel" utilization of longestablished analytical methods, e.g., differential interference contrast (DIC) microscopy and X-ray microfocus fluoroscopy (microfocus fluoroscope), to characterize the surface have been undertaken.

X-RAY MICROFOCUS FLUOROSCOPY: The surface and bulk densities of the TPO substrate were analyzed by exciting the substrate with narrow beam X-rays and monitoring the fluorescence emission produced. Figure 4 displays the emission obtained from analyzing the top 10 microns of the TPO substrate. The top spectra shows the presence of a thin skin (as depicted by a thin red stripe running across the top of the

# Morphology of Thermoplastic Polyolefins



substrate) which appears to be of higher crystallinity than the bulk substrate beneath it (color intensity represents variances in crystallinity, with red indicating high crystallinity and blue indicating low crystallinity). If one matches the intensity and frequency of the fluorescence emission (bottom spectra), the spectra attained is very similar to that obtained from a known sample of crystalline polypropylene.

If we now analyze the area directly beneath the top 10 microns of the TPO substrate (*Figure 5*) the color, intensity, and frequency of emission change. The spectra shifts to a light blue color (indicating low crystallinity), and the intensity and frequency of emission more closely parallel emission spectra attained from known samples of ethylene-propylene rubber.

DIFFERENTIAL INTERFERENCE MICROSCOPY (DIC): In order to ascribe the variances in crystallinity as viewed in the X-ray microfocus fluoroscopy spectra to some morphological changes within the substrate, high powered (2400X) optical microscopy with Nomarski interference filters under cross-polarizers was undertaken. *Figures* 6-10 represent the analyses. This type





Figure 6—IPO surface morphology (optical microscopy).



Figure 8—TPO morphology—cavitation effects,



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of analysis was chosen over conventional techniques such as transmission electron spectroscopy (TEM) because it was postulated that it would be difficult to differentiate between stained amorphous ethylene-propylene rubber and amorphous polypropylene (a significant amount of this species would be present in the substrate since semi-crystalline polypropylene is approximately only 50-60% crystalline). (Note: In TEM, staining agents such as osmium or ruthenium tetroxide are used to accentuate morphology; these stains highlight amorphous regions.)<sup>4</sup>

Figure 6 displays the surface morphology of TPO as molded (this substrate was injection molded). It appears that a "droplet" sits at the top surface of the substrate. Upon further analysis (FTIR), this "bubble" was shown to be amorphous polypropylene. It is conjectured that low molecular weight amorphous polypropylene, part of the semi-crystalline polypropylene utilized in the TPO blend, migrates to the surface of the substrate upon cooling from the melt. Since it is a low surface free energy material, it forms a bubble at the air interface to minimize its free energy.

If one then assumes that the polypropylene that was shown to crystallize at the TPO surface from X-ray microfocus fluoroscopy analysis is only semi-crystalline, then upon exposure to continued heat the low molecular weight amorphous content of the surface should continue to migrate and deposit at the surface. *Figures* 7 and 8 depict the effect of morphological

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changes in TPO to heat exposure (121°C). As the "as-molded" TPO is continually exposed to heat, an enhanced concentration of amorphous polypropylene forms at the surface. The amorphous content continues to build through droplet formation until a thin layer of the amorphous polypropylene is deposited. This build-up of amorphous content is accomplished via "cavitation" of amorphous droplets from the top semi-crystalline TPO surface (*Figure* 8). As evidenced in *Figure* 8, a droplet can be seen in the amorphous skin (left photograph) adjacent to a "cratered" void from which it cavitated. Upon analysis of the same top TPO surface via "dark field" optical microscopy the

skin disappears (right photograph). Under the dark field conditions, amorphous regions appear black, thereby indicating the amorphous content of the skin thus formed by heating.

Even more intriguing, if one analyzes the top surface of a painted TPO substrate via the same DIC technique, it can be seen that these "amorphous droplets" can be cause for dewetting of the substrate by the paint (*Figure 9*). The droplets in the asmolded substrate can be seen directly under the paint skin with areas of dewetting adjacent to the material. These "dewetted" areas are voids that when exposed to some external stress (e.g., stone impact) may be cause for paint delamination. It is therefore important to limit, if possible, these areas of cavitated droplets to ensure paint adhesion to the TPO.

The morphology of the entire TPO substrate was also analyzed via DIC (*Figure* 10). Utilizing the knowledge that the polypropylene crystallizes to different degrees under varying temperature gradients that are present in injection molding,<sup>5</sup> we assumed that the polypropylene in the TPO should act in the same vein. From left to right in *Figure* 10, the top surface through the bulk down through the bottom surface of TPO is displayed. The morphology is shown to go through a variation in crystallinity as a function of depth into the substrate. Little evidence of crystallities are too small (0.5 microns) for the resolution of the microscope. Large spheru-







lites (on the order of 50-100 microns) can be viewed in the bulk.

DIFFERENTIAL SCANNING CALORIMETRY (DSC): To justify our claims that crystalline polypropylene does exist on the surface of the molded TPO part, we undertook a DSC study of the top skin of the substrate versus the bulk composition (*Figures* 11 and 12). *Figure* 11 exhibits endothermic transitions at 128.83° and 164.01°C attributable to polyethylene (a process stabilizer) and polypropylene melting, respectively. *Figure* 12, a DSC trace of the bulk TPO, exhibits the same two transitions, but the melting points are shifted slightly upward, and the heats of fusion under the melting points are diminished. From the heats of fusion data, it can be surmised that the skin contains a greater proportion of a particular crystalline "form" of polypropylene. The form of polypropylene crystallinity, however, seems to be somewhat different in the skin than in the bulk as evidenced from the shift in melting point.

The data presented by Fillon et al.<sup>5</sup> on the crystallinity of polypropylene versus nucleation temperature indicates that a shift in melting temperature does occur as a function of the structure of crystalline material formed. The monoclinic form of a polypropylene spherulite (alpha phase) melts at higher temperatures (approaching 165-175°C) than the corresponding hexagonal crystal (beta phase) structure melting at 155-160°C). The crystallization temperature of polypropylene ( $T_c$  is approximately 112-121°C) is dramatically influenced by the rate of cooling. Therefore, the subsequent crystallites that are formed can differ.



Figure 13 displays the effect of polypropylene crystal type (as evidenced from the DSC melting behavior) in TPO as a function of substrate depth. It can be seen that the top and bottom skins of the substrate exhibit a multiple melting transition, with broad melting point ranges from 160-168°C, whereas the middle of the substrate melts sharply at 168°C. It can be equated from this data that the top and bottom of the substrate, which see rapid cooling effects from the melt during injection molding conditions, crystallize in both the alpha and beta phases. Whereas the middle of the substrate, which is insulated from rapid cooling effects and, therefore, cools slowly with optimized crystal formation, crystallizes mainly in the alpha form.

If one now assumes that the subsequent paintability of the TPO is influenced by the type and amount of polypropylene crystallinity present at the surface, one can perform experiments that establish the effect of painting conditions on paint adhesion. Keys to paintability require that the adhesion promoter, namely a chlorinated polyolefin (CPO) dissolved in solvents, diffuses into the substrate and entangles with the rubber phase within the TPO. The influence of solvent in the adhesion promoter (ability to swell the rubber) and bake temperature (ability to "open" the crystalline polypropylene at the surface to allow for solvent and thus polymer diffusion) should play vital roles in paintability.



Adhesion promoter solvents are believed to cause a change in surface geometry. If the solvents are allowed to diffuse through the polypropylene boundaries and into the rubberrich layers, the top morphology of the TPO surface should "roughen" as a result of rubber swelling (*Figure* 14). One can measure swelling in one of two ways: (1) by a change in substrate thickness; or (2) by a change in surface profile.

Figure 15 displays the effect of substrate swelling to a variety of solvents through a measure in percent swelling. The percent swelling ratio is determined by measuring the thickness of the unpainted TPO prior to exposure to a given solvent, and dividing that number by the substrate thickness after exposure to a solvent to gain a percentage. Solvent exposure was accomplished by placing a given quantity of solvent under a watch glass on the TPO surface and exposing the surface to that solvent for 45 min at 25°C. Solvents selected represent those most often utilized in CPO adhesion promoters. It can be seen from the data that solvent selection



Figure 16—Atomic force microscopy (analysis of surface swellability).

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The effect of polypropylene crystallinity on solvent diffusion rates (permeation rates) is shown in Figure 19. Permeation rates of an adhesion promoter solvent blend (containing xylene, toluene, and aromatic 100) were studied through polypropylene films (25 microns thick) of varying crystallinities. The permeation rates were measured at 25°C by placing 6 mL of the adhesion promoter solvent blend in a Fisher Payne permeability cup, covering the cup with the polypropylene film to be measured, and determining weight loss (due to solvent evaporation by diffusion through the film) in the cup over a 45 min time span. It can be viewed from the data that the higher the crystallinity in the

polypropylene film, the less the amount of solvent permeability through the film. Therefore, it is safe to say that if highly crystalline polypropylene skins form on the surface of the TPO substrate during molding that it will be more difficult to attain adhesion because of lack of permeation of the CPO adhesion promoter.

Variances in polypropylene crystallinity, as well as phase separation of the tubber phase from the polypropylene phases, can result from differences in molding conditions. Shear forces in the injection molding operation will be maximized at or near the gate (areas where the TPO melt is injected into the mold cavity). For this reason, we studied the effects of distance from the gate and amount of rubber in the TPO blend (*Figure* 20). Unexpectedly, the TPO with the higher amount of rubber swelled less than that with lower rubber content. The more highly sheared samples (near the gate) swelled less than those farther from the gate. The effect of shear therefore



will play a role in the amount of swelling and, therefore, the degree of entanglements of the CPO with the rubber phase.

Surface swelling of the TPO can also be measured by determining the change in surface profile as a function of solvent exposure. Figures 16 and 17 display the effect of solvent swelling on two different TPOs with aromatic 100 at 25°C for 45 min as measured by atomic force microscopy (AFM). The AFM technique utilizes a profilometer stylus that is run across the surface measuring changes in surface "profile." The color scale depicts changes in surface "depth," with red indicating a "high" profile whereas blue represents a "deep" profile. Figure 16 represents the profile of a TPO substrate low in rubber content, thereby displaying little texture as a result of rubber swelling. Figure 17, on the other hand, representing a highly rubber loaded TPO, displays evidence of more texture on the surface as a result of rubber swelling. This change in profile can also be quantified by the

percent swelling technique (*Figure* 18), where the higher rubber content TPO (higher in ethylene propylene (EP) rubber) displays a greater percent swelling ratio.

Knowing that the effect of rubber swellability is an important factor in determining solvent diffusion into the substrate, it is also important to note the effect of the polypropylene surface boundary on solvent diffusion rate. Since we have shown that different types of polypropylene crystals can form at and throughout the TPO substrate, we must determine their effect on diffusion rates, and thus the ability of the CPO adhesion promoter to migrate into the rubber boundaries.

Figure 17—Atomic force microscopy (analysis of surface swellability).



















plays a role in TPO morphology. It appears that not only rubber content but polypropylene crystallinity and its layer thickness may play a role in subsequent solvent swelling.

Surface swelling kinetics, e.g., rate of swelling in aromatic 100 as measured by profilometer over time (0 to 30 min at 725°C), were determined on a series of TPOs exposed to different annealing conditions (Figures 21 and 22). As shown previously, nucleation temperatures do affect the polypropylene crystallinity and should therefore directly affect the amount of solvent diffusion into the substrate and thus the amount of swelling [we assume that the major cause for substrate swelling is rubber swelling in the presence of the solvent (crystalline polypropylene does not swell in the presence of aromatic 100 to any appreciable extent)]. Figure 21 displays the swelling kinetics over time for unpainted TPO annealed at a variety of temperatures prior to exposure to aromatic 100 at 25°C. The TPO substrate (all taken from the same lot) was preannealed in an oven at 20°, 121°, or 170°C for 30 min part temperature prior to testing (graphs are labeled by anneal temperature, e.g., 121°, 170°C, etc.). These temperatures were chosen to determine the effect of the as-molded condition (20°C), of the recrystallization temperature of polypropylene (121°C), and of the melting point of polypropylene (170°C) on the amount and type of polypropylene crystallinity at the surface

Figure 21 shows that as the annealing temperature of the TPO approaches the melting point of polypropylene the swellability decreases. The highly sheared substrates (those near the gate labeled with a suffix "G") also display the same effect, however, they swell to a greater extent. This behavior suggests that as the polypropylene crystallinity increases (reorientation of the spherulites reaches a maximum when recrystallized from the melt at 170°C and allowed to cool slowly at room temperature) the degree of solvent diffusion into the substrate decreases, and the rubber swells less. The fact that the gated samples swell more, even after the polypropylene has been allowed to crystallize more fully, indicates that the polypropylene boundary layer at the surface is thinner in samples taken near the gate than those taken farther from the gate. Therefore, it appears the effects of shear in injection molded TPO manifest themselves in separation of the polypropylene and rubber phases.

Substrate shrinkage (deswelling) after exposure to aromatic 100 was also determined in attempts to delineate the ability of a rubber layer, once swollen, to return to an "unswollen condition." This will become more important when referring to the cohesive strength of swollen and unswollen TPO (discussed later in the Hesiometer section of this paper) when exposed to external stresses. Figure 22 measures the deswelling behavior of the same series of TPO substrates as a function of time after swelling (0 to 30 min after exposure to aromatic 100 at 25°C).

The kinetics of deswelling of a 110,000 psi flexural modulus TPO to aromatic 100 were fit to a second order equation with respect to time:

$$R_q = 1.61 \times E - 4 t^2 + 6.79 \times E - 3 t + 0.057$$
  
where  $R_q = rms$  roughness  
 $t = time$   
 $E = exponential$ 

٢

The correlation coefficient of goodness of fit was 0.992. This



indicates that once a rubber is swollen it will take a long time for it to deswell since this is not a linear equation.

In attempts to determine if swellability was proportional to the amount of solvent diffused into the substrate, a fluorescent dye, namely 7-diethylamino-4-methyl coumarin, was added to the adhesion promoter solvent blend at a concentration of 0.5 wt% based on CPO resin solids. The amount of dye penetration, and thus solvent penetration, was then measured by cryogenically fracturing the substrate and viewing the crosssectional fluorescence on a fluorescence microscope using a violet excitation filter in combination with a dichroic mirror and an additional barrier filter. Photographs were made using high speed color negative film and depths of fluorescence were measured using Sigma-Scan software with known standards. The fluorescence data thus obtained (see a representative fluorescence photograph as viewed in *Figure 23*) corre-





lated well with swellability data, e.g., the deeper the degree of dye penetration the more the swelling of the substrate. This data, therefore, supports the location and concentration of rubber near the surface as the main reason for swelling.

Hesiometer measurements, a type of "peel-strength" adhesion test, were then undertaken to determine the "optimal" swelling required in a TPO substrate to attain adhesion without affecting the cohesive integrity of the substrate. A schematic of the Hesiometer testing apparatus is shown in *Figure* 24 with a schematic of types of failure obtained from such a test shown in *Figure* 25. In this test method, a sample is placed on the translation stage of the Hesiometer and the blade is placed horizontally on top of the substrate. The blade then translates at a constant force across the substrate measuring the energy required to remove designated layers (referred to as removal force). Data obtained from such a measurement can then be translated into the type of failure measured, e.g., cutting, cracking, peeling, etc., as shown schematically in *Figure* 25.<sup>6</sup>

Figure 26 displays the effect of TPO exposure to toluene (45 min at 25°C) on cohesive energy thus attained. The thickness of the boundary layer removed and the resulting energy required to remove the layer is given. It can be seen from the graph that as the boundary layer becomes thicker the adhesion energy required to peel the respective layer off increases. Solvent exposure seems to decrease the energy required to "cohesively delaminate" the substrate up to a thick-





ness of approximately 200 microns, where little variance is seen.

The effect of varying lengths of solvent exposure of the same TPO to aromatic 100 on adhesion energy is shown in *Figure 27*. The same trends occur with this solvent versus toluene, where increased swelling decreases adhesion energy of layer removed up to about 200 microns. Longer solvent exposures, thereby more effective swelling, decrease the cohesive integrity of the substrate. Solvent evaporation rates therefore become a prominent factor in the cohesive energy of the substrate once painted. If the solvent has a slow evaporation rate and remains in the substrate after painting, the substrate is more prone to exhibit a cohesive delamination when exposed to stress than a substrate which was painted with a fast evaporating solvent that leaves the substrate and allows it to reach its equilibrium deswelled condition (which exhibits higher adhesion energies).

The effect of annealing temperature on the TPO substrate was also evaluated by the Hesiometer to determine its effects on cohesive energy of boundary layers. It appears from the data presented in *Figure* 28 that as a TPO substrate is an nealed at its recrystallization temperature and allowed to cool slowly at room temperature (e.g., the crystallinity at the surface increases) the cohesive integrity of the substrate decreases down to a depth of approximately 200 microns into the bulk and then remains relatively constant. This data suggests that reorientation of the polypropylene at the surface of the substrate can severely weaken the substrate's top surface layers.

# CONCLUSIONS

Differential scanning calorimetry, differential interference contrast microscopy, optical cross-polarized birefringence microscopy, and X-ray microfocus fluoroscopy afforded us the ability to determine the amount and type of layered morphology achieved in a thermoplastic olefin substrate as a function of processing conditions. Polypropylene appears to crystallize at the surface of the plastic when injection molded. The amount and crystallinity of the polypropylene at the surface can be varied by changing conditions such as temperature and shear in the molding process, as well as annealing conditions after molding. Rubber, which lies in a layer directly beneath the polypropylene surface, appears to be responsible for

# Morphology of Thermoplastic Polyolefins

paintability of the TPO. The two layers, in conjunction with the paint and its processing parameters, appear to relate more directly to the cohesive integrity of the painted composite.

Initial adhesion to a TPO substrate appears to be a function of polypropylene crystallinity at the surface only as it relates to the ability of solvents to permeate through and into the rubber layer beneath it. We have shown that polypropylene crystallinity is a function of molding conditions, as well as annealing conditions imposed on the part after the molding operation. The more crystalline the polypropylene is at the surface, the less solvent, and, hence, adhesion promoter that can diffuse into the substrate and the lower the ability of a paint to stick (this statement is based purely on the assumption that in order to attain adhesion to TPO, adhesion promoter must mechanically interlock with the rubber molecules in the underlying boundary layer).

Swelling of the rubber in the TPO by solvents contained within topcoats applied to TPO is diffusion controlled. The amount of swelling can result in lowering the amount of energy required to pull the substrate cohesively apart (as determined by Hesiometer data). Therefore, one must carefully choose the correct solvent balance contained within paints applied to TPO. It is essential to attain swelling within the substrate to afford initial adhesion of the paint, but the solvent must evaporate and the rubber deswell to attain cohesive integrity of the substrate once painted. One can therefore choose solvents with selected evaporation profiles to match the topcoat bake parameters in the painting process and thereby insure the proper swelling/deswelling kinetics.

Finally, by increasing the amount of rubber in the TPO, a more elastic layer forms from which a higher cohesion energy can be attained before reaching its yield stress and delaminating. Therefore, the selection of TPO modulus in determining ability to accommodate external stresses is important.

The data supplied thus far allows us to propose a mechanism for cohesive delamination of the substrate based on surface morphology as a function of painting conditions. The integral strength of the substrate is very important when considering the external stresses that can and will be imposed upon painted parts, i.e., gravel impact. Thus, the robustness, and, therefore, the useful life of the painted part, can be predicted prior to field exposure. Although our understanding of the TPO substrate has been further elucidated by the techniques described herein, we have as yet to relate our swellability data to actual field performance of painted parts. The trends developed between swelling and cohesive strength of the TPO substrate, although presented herein, will be further delineated in subsequent papers.

# ACKNOWLEDGMENTS

The authors wish to thank the following employees of the Ford Motor Company without whom this paper would not be possible: Janice Tardiff and Anna McNeight for data supplied from Hesiometer measurements and Clark Thomas, Jeff LaDuke, and Jim Bayley for optical cross-polarized birefringence data. Alex Zinkosky is also gratefully acknowledged for his contribution of X-ray microfocus fluoroscopy spectra.

Gene Sommerfeld and Gene Giuffre of Bostik were instrumental in providing guidance in the differential interference contrast microscopy photographs, as well as the differential scanning calorimetry data, and they are also gratefully acknowledged.

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# Teamwork Brings Innovative Effect Pigments to Light

Rüdiger Iden-BASF AG\*

B ringing an innovative product to market these days requires a good deal more than just chemical research. Sales, marketing, production, and research must all come together in order to turn a technological innovation into a commercial success.

The development history of Paliocrom® products-a line of effect pigments created by a variety of innovative processes-is an excellent window through which to see the teamwork necessary to bring even the most technologically advanced product to fruition. The willingness of the production departments, the open-mindedness of the technical service department and the sense of innovative response to customer requirements in the marketing department all played equally important roles in the success of Paliocrom pigments, which earned the BASF Innovation Prize recognizing the achievements of a team of chemists, engineers, and technical service employees.

# Effect Pigments and How They Work

To understand the development history that follows, a brief discussion of the principle of effect pigments is necessary (see *Figure 1*).

From a physical viewpoint, we are dealing with a complex mixture consisting of the absorption of light, as known to us from conventional dyes or pigments; oriented reflection, a mirror effect similar to what can be observed on the surface of still water; and interference of reflected rays of light, comparable to the oil film on a puddle of water.

When this principle is transferred to a reflective substrate with a platelet shape, on which a further, partially reflective layer is applied, the situation is this: light of a particular wavelength can interfere positively on such a material, and can therefore increase in intensity. If the viewing angle is then altered, the path length of the light within the partially reflective layer also changes. This means that at a different angle of incidence, a different wavelength brings about an intensification of the light. The result of this is the angular dependency of the impression of brightness and color given by effect pigments-the 'effect" which we find so attractive.

Market research has told us, however, that to be commercially viable the pigmen



- Brilliant colored pigments
- Distinct light/dark effects
- High hiding power, good fastness properties

viable, the pigments must also fulfill a variety of further requirements:

✓ The colors must be bright.

 $\checkmark$  Distinct light-dark effects must be present.

 $\checkmark$  The colors must exhibit fastness to light and weathering.

 $\checkmark$  They must exhibit good hiding power.

 $\checkmark$  They must be compatible with a range of binder systems.

# The Hydrothermal Process

Even the very best concept is useless without suitable individual chemicals. In this respect, our hard work was repaid with some degree of luck. In an experiment to stabilize ferrite yellow by aluminum compounds in an autoclave, the temperature control on the autoclave failed. The test sample became overheated and the experiment therefore backfired. However, the isolated product included bright red flakes—and the ancestor of Paliocrom copper was born.

Thin, monocrystalline flakes of doped iron oxide (*Figure 2*) create the

play of multiple light reflection and light absorption that gives this pigment its shimmering, warm copper tone. It is created in a specially equipped hydrothermal plant in Stuttgart, Germany, that consists of two core elements (as shown in *Figure 3*):

- Ferrite yellow production, where the precursor is produced in highly pure form in the precise chemical composition and particle size; and
- (2) The hydrothermal reactor in which, under pressure and at high temperature, crystallization is carried out to give the effect pigment. Certainly not "soft" chemistry, but nevertheless an environmentally compatible synthesis which is sparing in its consumption of resources.

# Figure 2—Paliocrom Copper

- BASF patents Europe Al<sub>x</sub>Fe<sub>2-x</sub>O<sub>3</sub> flakes
- BASF patents worldwide Al<sub>x</sub>Mn<sub>y</sub>Fe<sub>2-x-y</sub>O<sub>3</sub> flakes
- Hydrothermal iron oxide, high hiding power, velvety

<sup>\*</sup>Central Division Color Laboratory, Head of Research, Pigments and Nontextile Dyes, Ludwigshafen, Germany.

### R. Iden





The sales department was quick to recognize the benefits the hydrothermal process offers customers:

✓ True monocrystals without in-built defects.

✓ Controlled particle shape.

✓ Uniform particle size distribution, or a large "useful fraction."

✓ No agglomeration.

✓ Controlled, homogenous composition, representing a high degree of reproducibility in production batches.

# **Gas Phase Reduction**

A completely different kind of preparation is required for Paliocrom blue-silver. In this pigment, the mica support is surmounted by a layer of titanium dioxide, which undergoes chemical modifications in a gas phase reduction by the incorporation of titanium oxynitrides. This results in an angle-dependent deepening in color shade to blue. (This, too, required the development of a unique process—see Figure 4.)

The blue-silver is a pigment with good hiding power, a property which is sought after because it allows less pigment to be

#### Figure 4—Paliocrom Blue-Silver

- BASF patents
- Processes protected worldwide (granted or applied for)
- TiO<sub>x</sub>N<sub>y</sub> coated mica
- Gas phase reduction
- Hiding power

used and thinner coats to be applied without allowing the substrate to show through.

Our production department was able to achieve these properties via a high-temperature reduction carried out using ammonia in a rotary tube furnace (*Figure 5*). This, too, is a process with high environmental compatibility, since the only by-product besides nitrogen and water is hydrogen. The process can be controlled precisely, and consequently gives a large number of additional interesting effect pigments.

# Chemical Vapor Deposition

Undoubtedly, the most fascinating variant is a process known as chemical vapor deposition, or CVD (U.S. Patent No. 4,328,042). CVD, which produces Paliocrom gold, redgold, and red, represents the production of solids from reactive, vaporizable liquids which are deposited from the gas phase onto a solid substrate.

The uniform layer of iron oxide (about 25 nanometers thick) on the aluminum flakes results in properties which are truly unique (*Figure* 6): a gleaming golden metallic effect pigment with high brightness values and a hitherto unmatched hiding power. Like the other Paliocrom products, the potential of these pigments was noted at an early stage in the marketing department, and ultimately led to the new CVD production plants in Ludwigshafen, Germany.

The secret of this success lies in the chemical vapor deposition reactor (*Figure* 7), which in principle looks quite simple but contains state-of-the-art engineering. In the reactor, the aluminum flakes are fluidized in a stream of nitrogen and thus rendered accessible as the reaction medium to the reactor.

tive iron carbonyl, which is passed, in controlled amounts, together with oxygen. On these flakes the iron carbonyl decomposes to give uniform layers of iron oxide. The only waste product formed is carbon monoxide or, after catalytic oxidation, carbon dioxide.

#### Figure 6—Paliocrom Products

# Gold

- BASF patents
- Materials and processes protected worldwide
- $\alpha$ –Fe<sub>2</sub>O<sub>3</sub> via CVD on aluminum
- Particle size <30 μm/20μm</li>
- High hiding power

# Red

- BASF patents
- Processes protected worldwide
- a–Fe<sub>2</sub>O<sub>3</sub> via CVD on mica, aluminum
- Hiding power

This high degree of compatibility with the environment makes chemical vapor deposition a process whose future is assured, from precisely this ecological viewpoint. Other advantages of this innovative technology include:

 $\checkmark$  Application to a wide selection of substrates.

✓ An electrolyte-free coating.

✓ Firmly adhering, homogenous nanolayers.

# **Innovative Effect Pigments**

✓ Simple process control.

 $\checkmark$  Simple realization of multi-layer systems.

# CVD Tomorrow

Looking beyond the color pigments mentioned in this article, the chemical vapor deposition process offers an extremely broad variety of products. For example, it may enable us to melt inorganic and organic pigments with previously unknown potential. We are also investigating the use of CVD for other effects, such as conductive fillers or infrared-reflecting pigments, as a functional material in protection against overheating in vehicles. We are studying CVD-coated components for laser printers and copiers, which might have a considerably longer lifetime than their conventional counterparts.

The development of these processes was not without their setbacks, and often benefited from chance (see Appendix). However, each made its way along the path to industrial maturity due in large part to the cooperation of sales, marketing, production, and research personnel working toward a common goal.



#### Appendix The Innovative History of Paliocrom Pigments Some say that research consists of 90% error and 10% chance; others prefer the definition 90% hard work and 10% chance. The latter played an important part in Paliocrom pigments. HYDROTHERMAL PROCESS "Our Colleague, Mr. Chance" • 1976-78 Search for temperature-stable ferrite yellow (>180°C) -Coating with aluminum compounds -Heat treatment —Overheated autoclave $\rightarrow$ bright red flakes **Research and Development** • 1979 Development of continuous process 1986 Batch process Doped variants aluminum, silicon, manganese • 1989 Paliocrom copper goes into production CHEMICAL VAPOR DEPOSITION (CVD) Start Magnetite on glass (colored in part) -Optical storage media Late 1970s First experiments in color laboratory Research • 1979 First patents • 1983 Paint development • 1987 Water-based paint development • 1992 Development with other substrates, other reactive gases Development • 1986 Development of processes Pre-marketing • 1989 • 1991 Paliocrom gold goes into production

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# **Society Reports**

# CDIC-JANUARY

#### "Acrylic-Polyurethane Hybrid Dispersions"

In a special election, Teresa L. Case, of Fiberglass Evercoat Co., was elected to assume the duties of Treasurer, due to the vacancy left by Steven Prodromo, of Hilton Davis Co.

Environmental Committee Chairman Kenneth Pendleton, of K.A. Pendleton Co., Inc., reported that the land ban provisions of RECRA need to be considered; they will cause drastic changes in the way some paint companies do business. He also noted the effect of upcoming shipping regulations.

According to Scholarship Chairman Andrew Nogueira, of Hunting Industrial Coatings, only three applications for the Lou Larsen Educational Grant were received.

The evening's first speaker was Philadelphia Society member Charles R. Hegedus, of Air Products and Chemicals, Inc. He addressed the members on "ACRYLIC-POLYURE-THANE HYBRID DISPERSIONS AND THEIR USE IN WATERBORNE COATINGS."

Dr. Hegedus acknowledged that acrylic and polyurethane dispersions have been used individually and as physical blends in coatings applications. While the resulting coatings display a balance of properties from the inherent characteristics of acrylics and polyurethanes, the properties of the blended coatings often fall short of those predicted from the traditional "rule of mixtures." The dispersions discussed by the speaker were polymerized in two steps. The first step is production of the urethane polymer by step growth polymerization. The second step consists of swelling the particles of polyurethane with the acrylic monomers, which are then polymerized by chain growth polymerization. This two-step polymerization, similar to that used to prepare interpenetrating polymer networks, results in a hybrid polymer that is dispersed in water after neutralization with amine. This dispersion has properties superior to those predicted by the traditional "rule of mixtures."

Dr. Hegedus presented charts that depicted the mechanical properties of these dispersions. They offer a balance between hardness, flexibility, and toughness. These urethane dispersions are available in three hardness grades, from soft to hard.

The speaker also discussed the molecular structure and the properties of clear and pigmented coatings made with these dispersions.

Q. What about approval for use on gym floors and bowling alleys?

A. While comparative data is being obtained, i.e., Taber abrasion, Tukon hardness, crosshatch adhesion, etc., approval for bowling alleys and gym floors has not been obtained.

The educational speaker was Frank Platek of the U.S. FDA's National Forensic Chemistry Center. He addressed how criminal and civil product purity investigations are conducted, and the analytical techniques and capabilities which are available at the USDA's center.

WILLIAM E. JELF, III, Secretary



Members of the Cleveland Society Technical Committee in attendance at the Society's January Meeting (from left): Philip Slifko, Fred Anwari, Wally Stipkovich, Mark DiLorenzo, Chris Harding, and Roy Glover.



Also attending the Cleveland meeting were (from left) NPCA's Executive Director Andy Doyle, Federation President Joseph P. Walton, and NPCA President Joseph M. Walton.

# CHICAGO—FEBRUARY

### "Designing for Quality"

It was announced that the Manufacturing Committee's "Process Safety" seminar is slated for March 8, at the Oak Brook Marriott, Oak Brook, IL.

Charles Rooney, of Orr & Boss, delivered the presentation "DESIGNING FOR QUAL-ITY."

Dr. Rooney described a "house of quality" matrix that accommodated customer requirements, product parameters, and quality specifications. He also highlighted a simplex centroid experiment model based on a ladder and factorial design, which minimizes the number of samples prepared to achieve the desired customer requirements.

VICTOR M. WILLIS, Publicity

# CLEVELAND—JANUARY

# "State of the Paint Industry"

FSCT President Joseph P. Walton, of Jamestown Paint Co., was joined in attendance by Joseph M. Walton, President of NPCA and J. Andrew Doyle, Executive Director of NPCA.

President Constance Williams, of The Lubrizol Corp., announced that the deadline for the A.L. Hendry Awards competition is July 3, 1995. In addition, the deadline for the Roon Foundation Awards competition is March 1, 1995.

Ms. Williams also reported that FSCT is sponsoring "Formulating for the Clean Air Act" symposium on March 21-22 at the Cleveland Airport Marriott. In addition, she announced that the CSCT manufacturing seminar "Manufacturing Principals to Survive in the 90s" will take place on April 11, at the Cleveland Hilton South. The 38th Technical Symposium, "Focus on the Future," is scheduled for May 4-5 at the Quaker Square Hilton in Akron, OH. Contact Sharie Moskaluk, of The Sherwin-Williams Co., for more information.

Ben Carlozzo, of Mameco International, presented Certificates of Merit to the Society's Technical Committee members.

The technical presentation entitled "STATE OF THE PAINT INDUSTRY" was delivered by John Danziesen, of Glidden/ICI.

Mr. Danziesen stated that the paint industry revolves around change, and he used ICI and ICI Paints to provide examples. The speaker noted that ICI is not only a paint company, but is involved in various businesses globally.

Mr. Danziesen discussed ICI Paints' focus, which includes decorative paints, auto body, and coatings for food and beverages. Geographically speaking, the paint market consists of the following: North America— 37.5%; Europe—37.5%; and Asia/Australia/New Zealand—25%. According to the speaker, the world paint market is 14 billion liters and \$50 million with one-half being decorative paints. If the markets are split by region, it would be North America—31%; Western Europe—31%; Asia Pacific—31%; and Latin America—7%.

During the last 10 years, a shakeup has occurred in the top leaders of world paint manufacturers since the industry has undergone change. The speaker cited China, Southeast Asia, and Asia as developing countries; Japan and Korea are also growing.

Mr. Danziesen detailed the concentration of paint manufacturers and global market share. They are: multi-national—9%; multi-regional—27%; and national—64%. The world total major product type is mostly decorative paints.

The speaker concluded by giving the following critical success factors for a paint company: create shareholder values, innovate, welcome change, and emphasize customer service.

RICHARD A. MIKOL, Secretary

# LOUISVILLE—FEBRUARY

### "Silica Flatting Agents"

Society President Mike Moilanen, of Süd Chemie Rheologicals, informed the members of the following upcoming events: March 22—Louisville Chamber of Commerce Seminar on Groundwater Protection Plans; April 11—Cleveland Society Manufacturing Symposium; and April 18-19— Washington Paint Technical Group Symposium.

The evening's technical speaker was Baltimore Society member Bernadette Plichta, of W.R. Grace & Co., who spoke on "Break-Through Slica Flatting for Waterborne Systems."

Ms. Plichta stated that regulatory compliance has resulted in advancements in waterborne coatings for demanding industrial applications. Due to improved waterborne technology, raw material suppliers have been provided with new opportunities and challenges.

The speaker introduced a silica hydroxyl gel that is a novel flatting agent designed for waterborne systems and includes a porous silica flatting agent with water-filled pores. When evaluating a product for the coatings industry, quality, appearance and performance are areas of consideration/importance.

According to Ms. Plichta, this silica hydroxyl gel has flatting efficiency in waterborne systems while preserving clarity, chemical and stain resistance, and rheology. Other benefits of the product are handling, formulating, and performance.

The speaker concluded by commenting that the development of this product demonstrates how raw material suppliers can formulate new technologies.

DAN FORTNEY, Secretary

# NORTHWESTERN—FEBRUARY

# "Acid Rain"

FSCT President-Elect Darlene R. Brezinski, of Consolidated Research Inc. attended the meeting. Dr. Brezinski presented possible areas in which the Federation could improve member services and strengthen its position in the coatings industry. She informed the membership that FSCT is inves-

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CDIC [Second Monday—Location alternates between Cincinnati, Columbus, Dayton, and Indianapolis]. WILLIAM JEIF, III, Akzo Nobel Coatings, Inc., P.O. Box 147, Columbus, OH 43216-0147.

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

CLEVELAND (Third Tuesday—Roadhouse, Independence, OH). RICHARD A. MIKOL, Tremco Inc., 10701 Shaker Blvd., Cleveland, OH 44104.

DALLAS (Second Thursday following first Wednesday—Radisson Hotel, Dallas, TX). CHIP NEWCOME, 1448 N. Joe Wilson Rd., Cedar Hill, TX 75104.

DETROIT (Second Tuesday—meeting sites vary). JAN SPALDING, BASF Corp., 26701 Telegraph Rd., Southfield, MI 48086-5809.

GOLDEN GATE (Monday before third Wednesday—alternates between Francesco's in Oakland, CA, and Holiday Inn in S. San Franscisco). DON MAZZONE, Dowd & Guild, Inc., 14 Crow Canyon Ct., #200, San Ramon, CA 94583.

HOUSTON (Second Wednesday—Medallion Hotel, Houston, TX). Guy Sullaway, Courtaulds Coatings, P.O. Box 4806, Houston, TX 77210.

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LOS ANGELES (Second Wednesday—Steven's Steakhouse, Commerce, CA). JOSEPH B. EVANS, Trail Chemical Corp., 9904 Gidley St., El Monte, CA 91731.

LOUISVILLE (Third Wednesday—Executive West Motor Hotel, Louisville, KY). DAN FORTNEY, American Dispersions Inc., P.O. Box 11505, Louisville, KY 40211.

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

MONTREAL (First Wednesday—Le Bifthèque Steakhouse, St. Laurent, Quebec). Luc MILIETE, Frank E. Dempsey & Sons Ltd., 2379-46nd Ave., Lachine, Que., H8T 3C9, Canada.

NEW ENGLAND (Third Thursday—Sheraton Lexington Hotel, Lexington, MA). GENE C. ANDERSON, Chemcentral Corp., 38 Spindlewick Dr., Nashua, NH 03062. NEW YORK (Second Tuesday—Landmark II, East Rutherford, NJ). JOHN W. DU,

Hüls America, Inc., P.O. Box 365, Piscataway, NJ 08854.

NORTHWESTERN (Tuesday following first Monday—Jax Cafe, Minneapolis, MN). JOSEPH J. MILLS, Milsolv® Corp./Minnesota, 2340 Rose Pl., Roseville, MN 55113.

PACIFIC NORTHWEST (PORTLAND SECTION—Tuesday before third Wednesday—Tony Roma's, Mall 205, Portland, OR; SEATLE SECTION—Third Wednesday— Wyndham Garden Hotel, Sea-Tac, WA; VANCOUVER SECTION—Thursday after third Wednesday—Abercorn Inn, Richmond, B.C.J. EDward Linton, Cloverdale Paint Co., 6950 King George Hwy., Surrey, B.C., V3W 4Z1, Canada.

PHILADELPHIA (Second Thursday—Williamson's Restaurant, GSB Bldg., Bala Cynwyd, PA). THOMAS G. BROWN, Consultants Consortium, 209 Fox Ln., Wallingford, PA 19086.

PIEDMONT (Third Wednesday—Ramada Inn Airport, Greensboro, NC). Roy MoDJEWSKI, Akzo Nobel Coatings, Inc., 1431 Progress St., High Point, NC 27261. PITTSBURGH (Second Monday—Montemurro's Restaurant, Sharpsburg, PA).

JAMES REDISKE, Miles Inc., Mobay Rd., Pittsburg, PA 15205.

**ROCKY MOUNTAIN** (Monday following first Wednesday—Zangs Brewery, Denver, CO). CHARLES SHROEDER, Fel-Pro Inc., 6120 E. 58th Ave., Commerce City, CO 80022.

ST. LOUIS (Third Tuesday—The Salad Bowl Restaurant, St. Louis, MO). MICHAEL P. HEFFERON, Walsh & Associates, Inc., 1801 S. Hanley Rd., St. Louis, MO 63144.

SOUTHERN (GULF COAST SECTION—third Thursday; CENTRAL FLORIDA SECTION third Thursday after first Monday; ATLANTA SECTION—third Thursday; MEMPHIS SEC-TION—bi-monthly on second Tuesday; and MIAMI SECTION—Tuesday prior to Central Florida Section). WAITER R. NAUGHTON, JR., Scott Paint Corp., 7839 Fruitland Rd., Sarasota, FL 34240.

TORONTO (Second Monday—Meeting sites vary). NATAUE JANOWSKY, Degussa Canada Ltd., 4261 Mainway Dr., Burlington, Ont., L7R 3Y8, Canada. WESTERN NEW YORK —MARKO MARKOFF, 182 Farmingdale Rd.,

WESTERN NEW YORK —MARKO MARKOFF, 182 Farmingdale Rd. Cheektowaga, NY 14225. tigating holding a Paint Show in 1996 in Mexico City, Mexico.

Technical Committee Chairman Ed Ferlauto, of The Valspar Corp., reported that he will attend the Federation's Technical Advisory Committee Meeting in Baltimore, MD, on March 16-17. Mr. Ferlauto also noted that the Technical Committee's paper on acid rain has been provisionally accepted by the JOURNAL OF COATINGS TECH-NOLOGY. He also stated that the paper on formaldehyde is underway.

Educational Committee Chairman H. Mustapha Bacchus, of The Valspar Corp., reported that student presentations for April's Education Night are being prepared.

The first speaker of the evening, Ed Ferlauto, presented the Northwestern Society's Technical Committee's paper "The STUDY OF THE EFFECTS OF ACID RAIN ON ALKYD, POLYESTER, AND SILICONE MODIFIED HIGH-SOLIDS."

Mr. Ferlauto stated that acid rain has been recognized as a factor in the degradation of applied coating materials. Mechanisms of degradation have shown a synergism when acid rain, sulfur dioxide, and UV light interact. In this study, the Society evaluated chemical as well as physical changes that occurred when alkyd, polyester, and silicone modified polyester highsolids coatings were exposed for nearly three years at five precipitation monitored sites in the United States and Canada. Also, a simulated acid rain chamber that precludes the influence of UV light and a high concentration of sulfur dioxide was used to expose the nanels

According to the speaker, precipitation pH, surface pH, anions and cations of panel washings, gloss changes of the coatings and surface analysis using ion scattering spectroscopy and secondary ion mass spectrometry were evaluated. Correlation coefficients were calculated to assess similar effects under different conditions. The correlations were employed to identify common factors that appear to influence degradation of the coatings.

Mr. Ferlauto revealed the results of the study. He said that degradation was enhanced as the average pH of rainfall decreases. Evidence indicated a difference in the chemical changes on the surface of coatings exposed to simulated acid rain compared with outdoor exposure. This observation was consistent with reports of synergistic behavior in the presence of UV light and sulfur dioxide.

The second speaker of the meeting was Eileen Eichten-Carlson, of Eichten's Hidden Acres Cheese and Bison Farm. She discussed "European Style Cheese Making in MINNESOTA."

JOSEPH J. MILLS, Secretary

# People in the News





S. Haduch

J.R. Simmons

Stan Haduch was promoted to Vice President and General Manager of Ranbar Technology Inc., Glenshaw, PA. He will be responsible for sales, marketing, manufacturing, and transportation.

In other news, Jay R. Simmons has joined the company as Manufacturing Manager. In this capacity, Mr. Simmons is responsible for all manufacturing activities, including plant management, engineering, maintenance, and transportation.

Gerald Katz has accepted the position of Senior Vice President of Corporate Development, Witco Corp., Greenwich, CT. Mr. Katz will be responsible for the worldwide search and evaluation of acquisition opportunities.

The company has also hired **Paul Lipsitz** as its Human Resources Director. In this capacity, Mr. Lipsitz will negotiate and administer collective bargaining agreements. Jeff Pillsworth has been promoted to Technical Service Supervisor, Resins, Nacan Products Ltd., Brampton, Ont., Canada. The Toronto Society member will be responsible for product application and technical service for the paint and coatings industry.

Lorrie M. Adamz was promoted to Plant Manager for Zeneca Resins' Napa, CA, production facility. She was previously a production engineer with Zeneca Resins' sister company, Zeneca Specialties, in Wilmington, DE.

E. Kears Pollock has been named Vice President, Coatings and Resins, PPG Industries, Inc., Pittsburgh, PA. In his new role, Mr. Pollock assumes operating responsibility for coatings and resins.

In other news, **Richard Zahren** was appointed Vice President of Automotive Products, Coatings, and Resins, filling the position vacated by Mr. Pollock.

The company has also made the following promotions: William H. Hernandez— Senior Vice President, Finance; Dan W. Kiener—Controller; and Gary W. Weber— Vice President, Science and Technology.

OSI Specialties, Danbury, CT, has appointed Anthony V. Parassio Vice President/Sales, North America. His responsibilities include sales and customer service in the United States and Canada.

# Obituaries

George E.F. Brewer, formerly employed by Ford Motor Co.'s Manufacturing Development Center, passed away on November 13, 1994. The Detroit Society member was 85.

In the early 1960s, Dr. Brewer carried out research for Ford in which he formed the concept of electrodeposition of the solid portion of water-reducible paint in a bath or tank. This research led to the issuance in 1965 of the first of a series of patents on electrodeposition of paint.

Dr. Brewer left Ford in 1972 to launch his consulting business, in which he remained active until his death. He was also the author of more than 100 articles in technical and educational journals and held 23 patents in electrodeposition and other areas of coatings technology.

In 1973, Dr. Brewer delivered the prestigious Joseph J. Mattiello Lecture, entitled "Electrodeposition of Paint: Achievement Through Parallel Effort," at the 51st Annual Meeting of the Federation, in Chicago, IL.

Edward G. Bobalek, an Honorary Member of the Cleveland Society, died on October 13, 1994 at the age of 79.

Mr. Bobalek concluded his professional career as Senior Chemical Engineering Advisor for the Environmental Protection Agency in Research Triangle Park, NC. He had also been Gottesman Professor and Department Head of Chemical Engineering at the University of Maine in Orono.

He is survived by his wife, Marie; five sons, Stephen, John, Thomas, Charles, and Philip; a daughter, Margaret King; and seven grandchildren. Charles Kahn, of the New York Society, died on January 2, 1995. He was 59 years old.

Mr. Kahn was President of Kahn Tech, Inc. and a partner in Kahn Tech Specialty Packaging. He was previously employed by Rohm Tech, Inc., Specialty Chemicals, which he joined in 1982.

Mr. Kahn was also a member of the American Chemical Society, the Sales Association of the Chemical Industry, and the Society of Cosmetic Chemists.

He is survived by his wife, Joan; and a daughter, Nicole.

Allan R. Coletta has been named Materials Manager for ICI Surfactants—Atlas Point Site, New Castle, DE. He will be responsible for warehousing, purchasing, domestic transportation, and import/export of raw materials and finished goods.

Engelhard Corp., Iselin, NJ, has named William E. Nettles Vice President and Chief Financial Officer. During a transition period of several months, he will work with Senior Vice President and Chief Financial Officer Robert L. Guyett, who is retiring.

William L. Staron has accepted the position of Vice President and General Manager of the Chemical Catalysts Group to fill the vacancy left by Mr. Nettles.

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# **Senior Faculty Position**

The Coatings Research Institute at Eastern Michigan University announces a tenure-track faculty position at associate or full professor rank with appointment as early as summer 1995. The Institute is committed to developing relevant scientific knowledge for understanding and expanding the technology of paints and coatings. Ph.D. with minimum three years industrial coating experience required. Candidates must demonstrate their ability to develop a vigorous, well-funded, internationally recognized research program and their commitment to excellence in teaching at undergraduate and graduate levels. Applications accepted until position filled. Submit cv, list of publications and professional awards, description of research interests and record of research funding, transcripts, and names of five references to Dr. John Massingill, c/o Position F9515, 204 King Hall, Eastern Michigan University, Ypsilanti, MI 48197.

# **New FSCT Members**

# BALTIMORE

# Active

Davis, Kelly D .- Duron, Inc., Beltsville, MD.

- Gavins, Laura S.-Duron, Inc., Beltsville. Gibson, Charles E .- Duron, Inc., Beltsville.
- Grieb, Ronald, S .- Duron, Inc., Beltsville.
- Higgs, William T .- Duron, Inc., Beltsville.
- Radman, John M.-Trace Laboratories, Linthicum, MD
- Reilly, Kevin M .- Duron, Inc., Beltsville.
- Sharma, Sunita R.-Duron, Inc., Beltsville.
- Stopyro, Jeanne M .- Duron, Inc., Beltsville.
- Thomas, Denise-Bruning Paint Co., Baltimore, MD.
- Vera, Ernesto-Union Carbide Corp., S. Charleston, WV.

#### Associate

- Cleavenger, Stephanie J.-Eastman Chemical Co., Horsham, PA.
- Firouzgar, Sam-KTH-Handels GmbH USA, Falls Church, VA.
- Morgan, John L .- Duron, Inc., Beltsville, MD.

# CHICAGO

#### Active

Palenik, Skip J .- Microtrace, Elgin, IL.

- Rodgers, Christopher-INX International, Elk Grove Village, IL.
- Wyers, John D .- Chase Products Co., Maywood, II.

#### Associate

- Goliak, Joseph W.-Witco Corp., Bolingbrook, Π.
- Hanrahan, Brian D .- Union Carbide Corp., S. Elgin, IL.

# DALLAS

Active Blast, Daniel B .- Jones-Blair Co., Dallas, TX.

# **GOLDEN GATE**

#### Active

- Buchman, Jeffery I .- Morton International, Dixon, CA.
- Hilbert, Theodore A .- Reichhold Chemicals, Inc., Durham, NC.
- Weernink, Jan—Technical Coatings Co., Santa Clara, CA.

#### Associate

Strahan, Cathy D.-Hockmeyer Equipment Co., Walnut Creek, CA.

# LOS ANGELES

### Active

- Burkes, Louie E .- Thoro System Products, Hayward, CA. Crawford, James K .- EDOCO, Long Beach, CA
- Desai, Harshad P .- Polycoat Products, Santa Fe Springs, Ca.
- Rice, Cassandra S .- Epmar Corp., Sante Fe Springs.

Uyeho, Howard Y .- EDOCO, Long Beach.

#### Associate

Collins, Edna-Search West Inc., Ontario, CA. Tijerina, Pedro-Pyochem, Inc., Yorba Linda, CA

# LOUISVILLE

Active

Kilian, Lori D .- Hampshire Chemical, Ownsboro, KY.

# NORTHWESTERN

Active

Frick, Darryl A .- Conklin Co., Inc., Shakopee, MN Pavlik, Kyle R.-Sierra, Minnetonka, MN.

# **PACIFIC NORTHWEST**

Active

Grint, Gary D .- Cardinal Industrial Finishing, Woodinville, WA. Sharman, Roger Leonard-Insul-Mastic, New

Westminster, B.C.

#### Tan, William Y.C .- Tenco Westech Ind., Inc., Richmond, B.C.

# **ROCKY MOUNTAIN**

#### Active

Scott, Vincent C.S.-Arizona Oxides Inc., Phoenix, AZ.

Cox, Gene-Sun Tech Coatings Manufacturing, Glendale, AZ.

# SOUTHERN

#### Active

- McHugh, David S .--Spirit Enterprises Inc., Snellville, GA
- Walsh, Robert E .- Gilman Paint, Chattanooga, TN.
- Wiggins, Gary A.-LRW Coatings Inc., Flomaton, AL.

#### Associate

Alberstadt, Richard E.-Magnolia Chemicals, New Orleans, LA Daigle, Frank-Mehaffey & Daigle, New Orleans.

# Educator/Student

Hariharan, Rajan-University of Southern Mississippi, Hattiesburg, MS.

# **Future Society Meetings**

# CDIC

(May 8)—"EVALUATION OF NEW GENERATION COALESCING AGENTS FOR INDUSTRIAL ACRYLIC LA-TEXES"—Thomas M. Larson, Exxon Chemical Co.

# Chicago

(May 12)—Annual Awards Banquet.

# Cleveland

(May 4-5)-Educational Symposium. (May 16)—Awards/Past-Presidents'/Spouses' Night.

#### Golden Gate

(May 15)-"HIGH-SPEED DISPERSION TECH-NIQUES"-Rocky Courtain, Morehouse Industries, Inc

(June 19)-Manufacturing Committee Seminar.

# Los Angeles

(May 10)—Awards Night. "HIGH-SPEED DIS-PERSION TECHNIQUES"-Rocky Courtain, Morehouse Industries, Inc. (June 14)—Annual Meeting.

### Montreal

(May 3)—"ADHESION PROMOTERS"—Eastman Chemicals

# **New England**

(May 25)—"New Chemistries in Defoamers AND WETTING AGENTS"-Fred Lewchik, BYK-Chemie.

### **Pacific Northwest** (Puget Sound Section)

(May 4-6)--48th Annual Spring Symposium, Portland, OR.

(May 17)-Joint Meeting with Puget Sound Paint and Coatings Association. "HIGH-SPEED DIS-PERSION TECHNIQUES"-Rocky Courtain, Morehouse Industries, Inc.

#### Pittsburgh

(May 8)-"COLOR TRENDS FOR THE COMING YEAR"-PPG Industries, Inc. Past-Presidents' and Spouses' Night.

#### Toronto

(May 8)-"Non-Toxic Anticorrosive Pigments in Aqueous Media"-Robert E. Snyder, A.R. Monteith (77) Ltd. (Toronto Society Technical Committee Presentation).



# **New Products**



# **Gloss** Topcoat

New from Sherwin-Williams is industrial enamel HS, a high-solids, low VOC alkyd gloss topcoat for interior or exterior industrial use. This high-gloss coating reportedly resists chipping, flaking, and abrasion as well as dirt and yellowing in industrial environments. Recommended applications include tanks, bridges, pipes, vessels, handrails, machinery, and equipment.

Circle No. 30 on Reader Service Card

# **Coating System**

A new coatings technology is designed to eliminate rust or corrosion for decades. Potential applications include aircraft, bridges, automobiles, ships, chemical storage facilities, and waste treatment facilities. The 21st Century Coatings Inc. products are reportedly able to perform as a corrosion barrier against all but 100% sulfuric acid, and have been used in U.S. Navy submarines.

Circle No. 31 on Reader Service Card

# Surfactant

Dynol<sup>™</sup> 604 surfactant is a low VOC, low foam, nonionic wetting agent which reportedly promotes substrate wetting of waterborne systems such as coatings, inks, and adhesives by lowering the equilibrium (static) and dynamic surface tension of aqueous systems. The product isable to act as an alternative for difficult-to-wet substrates requiring flow and leveling under diverse application conditions. A brochure on the Air Products and Chemicals Inc. surfactant describes its physical properties, applications, and performance.

Circle No. 32 on Reader Service Card

# Aqueous Colorants

A line of high-solids, solvent-free, low VOC aqueous color dispersions is designed for coloring of water-based systems. Aurasperse II™ colorants are formulated without alkyl phenol ethoxylates and reportedly provide fullcolor value with a minimum of mixing. The lead-free EngelhardCorp. dispersions are used primarily for architectural coatings.

Circle No. 33 on Reader Service Card

# Monomer

A monomer designed to provide automotive clearcoat finishes with depth of image in the paint matrix is introduced. Rohm Tech's D1053 methacrylate reportedly provides coatings with flexibility, strength, and toughness. This product also provides UV absorption qualities to help coatings resist degradation due to sunlight exposure.

Circle No. 34 on Reader Service Card

### Waxes

Luwax<sup>®</sup> montan and other waxes are used in such applications as automobile, floor, and shoe polishes as well as in wood and plastics processing. A technical bulletin, "Luwax Montan and Other Waxes," highlights these BASF Performance Chemical agents. This literature provides descriptions of products ranging from a hard wax used as an emulsifier in shoe and automobile polishes to a specialty wax suitable for solvent-based wood and stone finishes as well as for leather finish solutions.

Circle No. 35 on Reader Service Card

# Foam Control

A new foam control agent, antifoam emulsion SE 26, is engineered for providing longterm foam control in hot, high-alkaline systems. This 20% active ingredient solution can be added directly to most foaming systems, or can be diluted with either water or a portion of the foaming system. Good dispersibility in water and alkali resistance are reported of this Wacker Silicones Corp. agent.

Circle No. 36 on Reader Service Card

#### Benzophenone

Velsicure<sup>®</sup> benzophenone, available in technical flake and powder, has a purity of 99.9%. The benzophenone technical flake is used as an ultraviolet photoinitiator, an ultraviolet absorber, an intermediate for pharmaceuticals, and a food additive. This high purity product is manufactured by Velsicol Chemical Corp.

Circle No. 37 on Reader Service Card

# Industrial Colorants

The Chroma-Chem<sup>®</sup> 844 colorants line provides a choice of 180 colors for in-plant and machine tinting of high performance, nonaqueous industrial and maintenance coatings. These intermix colorants may be used at retail or factory levels. Hüls America Inc. recommends these products for use in applications including acrylics, alkyds, cellulosic lacquers, chlorinated rubber, epoxies, polyesters, polyurethanes, and vinyl lacquers.

Circle No. 38 on Reader Service Card



# Color Data Transfer

X-Rite, Inc. introduces ColorMail<sup>™</sup> color data transfer program. The system works with AT&T's EasyLink network, an already-established global electronic network, to facilitate color data transfer. This program operates by translating data from a spectrophotometer into an electronic file, which is then placed in an electronic mailbox and downloaded onto the recipient's computer.

Circle No. 39 on Reader Service Card

### **On-Line Monitoring**

On-line color monitoring systems which have application in the paper, plastic, film, coil coating, and coated glass industries are available. These HunterLab systems are reportedly able to minimize off-color products, reduce manufacturing costs, and improve processes. A VHS video describes savings in reduced inventories and production and efficiency as related to these systems.

Circle No. 40 on Reader Service Card



# **Mapping Stage**

The Advantage Motorized Mapping Stage provides precise positioning of samples for microanalysis on Spectra-Tech's FTIR microscopes. The stage has a travel range of 1.18 x2.76 in. and is designed for mapping or other types of planned sampling sequences in FTIR microscopy. Applications include quality control and surface defect analysis as well as mapping of polymers, electronic components, and geological samples.

Circle No. 41 on Reader Service Card

# Fluid Level Monitor

Bernhard's Tank Safe 712 fluid level monitor and overflow prevention instrument is designed to prevent overfilling of aboveground tanks by automatically shutting off flow to the tank. This monitor is mounted on top of the tank and measures liquid levels via an ultrasonic probe. A three-pole double throw relay allows the device to be wired to a pump or control valve so that when a high level condition is reached, flow is instantly cut off.

Circle No. 42 on Reader Service Card

# **Plasma Mass Spectrometer**

The Elan<sup>™</sup> 6000 Inductively Coupled Plasma Mass Spectrometer is a fully automated, computer-controlled system that provides routine analysis of inorganic elements. This instrument features a compact design for easy system installation in most laboratory settings. All major subsystems, such as the sample introduction system, plasma, interface, lens, and vacuum system, are under computer control in this Perkin-Elmer device.

Circle No. 43 on Reader Service Card

# **Remote Tank Management**

AllTank<sup>®</sup> L.L.C. introduces its inventory monitoring system for supply chain management. The turnkey system includes monitors, modems, hookups, software, and sensors, and is used in aboveground tank management and delivery scheduling. Using standard nondedicated phone lines, this system automates tank level, fill, and leak detection information gathering from multiple remote sites to one central location.

Circle No. 44 on Reader Service Card

# **Molecular Weight Analysis**

Shimadzu Scientific Instruments has combined matrix-assisted-laser-desorption-ionization with a sensitive time-of-flight mass measurement system to produce the Kompact MALDI molecular weight analyzer. A range of sample substrates may be selected to provide results from a variety of different sample types. This instrument is designed for use in the biotechnology, industrial polymer, and pharmaceutical markets.

Circle No. 45 on Reader Service Card

# Color Spectrophotometer

A dual-beam, diffuse 8° sphere reference color measurement spectrophotometer is available. This Datacolor International instrument is equipped with matching, dual, 128 photodiode arrays and optical analyzers. The Spectraflash\*SF600™ is designed to provide high resolution color measurement and true dual beam drift correction for consistent color values.

Circle No. 46 on Reader Service Card



# Weathering Testing

GM Boxes offer an indirect weathering method to determine the durability and/or colorfastness of any material used for automobile interiors. These Heraeus DSET mechanisms feature an azimuth tracking method designed to avoid sample over-temperature conditions. The devices expose a variety of products and materials under glass, including air bag covers, door panels, seat cushions, and more.

Circle No. 47 on Reader Service Card



# **Cavity Pump**

Netzsch Inc.'s Nemo<sup>®</sup> Nesp progressing cavity pumps, engineered for reliability and low maintenance, feature open-throat suction housing with breaking/mixing paddles, permitting full flow of materials into the pump. The breaking/mixing paddles are powered by an auxiliary motor to ensure that materials do not bridge in the opening. An auger force feeds the material from the throat of the pump into the rotor-stator section.

Circle No. 48 on Reader Service Card

# TECHNICAL **MANAGER** -PAINTS Luzenac Inc., the Canadian subsidiary of Luzenac Group the largest talc company in the world, is renowned for quality and customer service. The company is seeking a dynamic and innovative Technical Manager to play the lead role in the development of its paint related products. Reporting to the Manager of Sales and Marketing, you will be responsible for product development, technical service and sales/marketing liaison. Based in Oakville, Ontario, you will travel extensively throughout Canada and the United States, with regular trips to France. You possess a technical degree, preferably in Chemistry and a minimum of five years experience in developing and evaluating raw materials in paint formulations. An

understanding of the function and application of minerals in paint would be an asset. You are experienced in client service and are a seasoned problem-solver at the customer level. You are a self-starter, able to achieve personal and corporate goals with minimal supervision and rise to the challenge of "running your own show".

If you are looking for an exciting opportunity, please forward a resumé, outlining your qualifications to Rita Bishop, Coopers & Lybrand Consulting, 21 King Street West, 2nd Flr. Hamilton, Ontario, Canada L8P 4W7 (905) 525-0009 ext. 247 Fax (905) 529-0463

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DD		Sales Agents for Raw Materials and Equipment
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# **Calendar of Events**

# **FEDERATION MEETINGS**

For information on FSCT meetings, contact Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422 (610) 940-0777, FAX: (610) 940-0292.

# 1995

(May 17-21)—FSCT Spring Week. Spring Seminar on the 17th and 18th; Board of Directors Meeting on the 20th; Incoming Society Officers Meeting on the 21st. Fiesta Americana, Cancun, Mexico.

(June 20-21)—"Polymer Chemistry for the Coatings Formulator." Seminar sponsored by the Professional Development Committee. Schaumburg Marriott Hotel, Schaumburg, IL.

(Oct. 9-11)—73rd Annual Meeting and 60th Paint Industries' Show. Cervantes Convention Center, St. Louis, MO.

(Nov. 6-7)—"Formulating for the New Clean Air Act." Seminar sponsored by the Professional Development Committee. Denver, CO.

#### 1996

(Oct. 23-25)—74th Annual Meeting and 61st Paint Industries' Show. McCormick Place North, Chicago, IL.

# 1997

(Nov. 5-7)—75th Annual Meeting and 62nd Paint Industries' Show. Georgia World Congress Center, Atlanta, GA.

# SPECIAL SOCIETY MEETINGS

# 1995

(Apr. 19)—"Spectrum of Coatings Science." Sponsored by the Louisville Society. Executive West Hotel, Louisville, KY. (Ilona Duvall, Red Spot Paint Co., P.O. Box 418, Evansville, IN 47703-0418; (812) 428-9337).

(Apr. 19-21)—"Getting Ready for the Next Century.' Southern Society Annual Meeting. Hyatt Regency, Savannah, GA. (Wayne West, Thompson & Formby, Inc., 10136 Magnolia Dr., Olive Branch, MS 38654).

(Apr. 22)—"Springfest '95." Sponsored by the Northwestern Society. (Robin Norcutt, George C. Brandt, Inc., 2975 Long Lake Rd., Roseville, MN 55113; (612) 636-6500).

(May 3-4)—"Recent Advances in Modifiers for Modern Coatings." Symposium sponsored by the New York Society. Holiday Inn North, Newark Airport, Newark, NJ. (Larry Waelde, Troy Chemical Corp., c/o NYSCT Office, 520 Westfield Ave., Elizabeth, NJ 07208; (908) 354-3200).

(May 4-5)—"Focus on the Future." 38th Annual Technical Symposium sponsored by the Cleveland Society. Quaker Square Hilton, Akron, OH. (Sharie Moskaluk, The Sherwin-Williams Co., 601 Canal Rd., Cleveland, OH 44113-2498; (216) 566-3661).

(May 4-6)—48th Annual Spring Symposium. Sponsored by the Pacific Northwest Society. Red Lion Lloyd Center Hotel, Portland, OR. (Ken Wenzel, Chemical Distributors, Inc., P.O. Box 10763, Portland, OR 97210; (503) 243-1082).

(May 8-9)—Eastern Training Conference and Show. Sponsored by the Philadelphia Society for Coatings Technology. Valley Forge Convention Center, Valley Forge, PA. (Wayne Kraus, Hercules Inc., Research Center, 500 Hercules Rd., Wilmington, DE 19808; (302) 995-3435. Booth reservations: Larry Kelly, Eastech Chemical, Inc., 5700 Tacony St., Philadelphia, PA 19135; (215) 537-1000).

#### 1997

(Feb. 18-20)—Western Coatings Societies' 23rd Biennial Symposium and Show. Sponsored by the Golden Gate, Los Angeles, Pacific Northwest, and Rocky Mountain Societies. Disneyland Hotel and Convention Center, Anaheim, CA. (Bruce Cotton, Pluess-Staufer (California), Inc., P.O. Box 825, Lucerne Valley, CA 92356; (619) 248-7306; or Ron Elliott, J.R. Elliott Enterprises, Inc., 300 Thor Pl., Brea, CA 92621; (714) 529-0711.

# OTHER ORGANIZATIONS

(Apr. 23-25)-ISCC Annual Meet-

# 1995 — North America



ing. Sponsored by Inter-Society Color Council, Greensboro, NC. (Ron Oldchurch, ISCC Poster Papers Committee, 1680 N. Hwy. 101, ≠11, Leucadia, CA 92024).

(Apr. 24-27)—"Introduction to Coatings Science." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Shelby F. Thames, Director, USM, Box 10037, Hattiesburg, MS 39406-0037).

(Apr. 24-28)—"Applied Rheology for Industrial Chemists." Short course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Director, Professional Development Institute, P.O. Box 1792, Kent, OH 44240).

(Apr. 24-28)—"Introduction to Paint Formulation." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(Apr. 28-May 2)—"International Ultraviolet and Electron Beam (UV/ EB) Curing Conference and Exhibition." Seminar sponsored by RadTech International. Opryland Hotel, Nashville, TN. (Chris Dionne, RadTech International North America, 60 Revere Dr., Ste. 500, Northbrook, IL 60062).



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(May 3-4)—1995 International Symposium on Developing Plastics Technologies. Sponsored by the Canadian Plastics Institute. Queen's Landing Inn, Niagara-on-the-Lake, Ontario. (The Canadian Plastics Institute, 1262 Don Mills Rd., Unit 48, Don Mills, Ont. M3B 2W7, Canada).

(May 8-12)—"Dispersion of Pigments and Resins in Fluid Media." Short course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Director, Professional Development Institute, P.O. Box 1792, Kent, OH 44240).

(May 15-19)—"Introduction to Paint Formulation." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(May 15-20)—"Interpretation of IR and Raman Spectra: Lectures, Interpretation Workshops, and FTIR Laboratories." Course sponsored by the Fisk Infrared Institute. Vanderbilt University, Nashville, TN. (Clara D. Craver, Fisk Infrared Institute, 1000 17th Ave. North, Nashville, TN 37208-3061).

(May 22-24)—Eighth International Symposium on Polymer Analysis and Characterization (ISPAC-8). Sanibel Island, FL. (ISPAC Registration, 815 Don Gaspar, Sante Fe, NM 87501).

(May 22-25)—"Coatings Science for Coatings Technicians." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Shelby F. Thames, Director, USM, Box 10037, Hattiesburg, MS 39406-0037).

(May 22-26)—"Adhesion Principles and Practice for Coatings and Polymer Scientists." Short course sponsored by Kent State University, Kent, OH. (Carl J. Knaus, Director, Professional Development Institute, P.O. Box 1792, Kent, OH 44240).

(June 5-8)—"Coatings Science for Coatings Chemists." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Shelby F. Thames, Director, USM, Box 10037, Hattiesburg, MS 39406-0037).

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Circle No. 241 on the Reader Service Card

(June 5-9)—"Advances in Emulsion Polymerization and Latex Technology." Short course sponsored by Lehigh University, Bethlehem, PA. (Mohamed S. El-Aasser, Emulsion Polymers Institute, Lehigh University, 111 Research Dr., Bethlehem, PA 18015).

(June 12-15)—"Coatings Science for Coatings Formulators." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Shelby F. Thames, Director, USM, Box 10037, Hattiesburg, MS 39406-037).

(June 25-29)—"Coating Work in Nuclear Facilities." Symposium sponsored by The American Nuclear Society and The National Board of Registration for Nuclear Safety Related Coating Engineers and Specialists. Marriott Hotel, Philadelphia, PA. (Dean M. Berger, Executive Secretary, NBR, P.O. Box 56, Leola, PA 17540).

(July 17-19)—"Applied Powder Mechanics." Short course sponsored by Pennsylvania State University, State College, PA. (Particulate Materials Center, 147 Research Building West, University Park, PA 16802-6809).

(July 17-19)—"Basic Coatings for Sales, Marketing, and General Personnel." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(Aug. 7-10)—"Introduction to Powder Coatings Technology." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Shelby F. Thames, Director, USM, Box 10037, Hattiesburg, MS 39406-0037).

(Sept. 11-15)—"Basic Composition of Coatings." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(Sept. 14-15)—"Advanced Radiation (UV/EB) Curing Marketing/Technology." Seminar sponsored by Armbruster Associates Inc. Newport Beach Marriott Hotel and Tennis Club, Newport Beach, CA. (David Armbruster, Armbruster Associates Inc., 43 Stockton Rd., Summit, NJ 07901).

(Sept. 19-21)—"Finishing '95." Conference and exposition sponsored by the Society of Manufacturing Engineers (SME). Albert B. Sabin Convention Center, Cincinnati, OH. (Mary Krome, SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121-0930).

(Sept. 19-22)—"New Horizons '95." Conference jointly sponsored by the American Oil Chemists' Society (AOCS) and the Chemical Specialties Manufacturers' Association (CSMA). Omni Sagamore Resort, Bolton Landing, NY. (Rebecca Richardson, AOCS, P.O. Box 3489, Champaign, IL 61826-3489).

(Sept. 20-21)—"Advanced Radiation (UV/EB) Curing Marketing/Technology." Seminar sponsored by Armbruster Associates Inc. Newark Airport Marriott Hotel, Newark, NJ. (David Armbruster, Armbruster Associates Inc., 43 Stockton Rd., Summir, NJ 07901).

(Sept. 20-22)—"Accelerated and Natural Weathering Techniques for Coatings and Polymers." Short course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Director, Professional Development Institute, P.O. Box 1792, Kent, OH 44240).

(Sept. 25-29)—"Introduction to Paint Formulation." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (Cynthia N. Campbell, UMR Coatings Institute, Dept. of Chemistry, 142 Schrenk Hall, Rolla, MO 65401-0249).

(Sept. 26-29)—"Introduction to Coatings Technology." Short course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Director, Professional Development Institute, P.O. Box 1792, Kent, OH 44240).

(Sept. 29-Oct. 1)—"Fall Decor 1995." Sponsored by the National Decorating Products Association (NDPA). McCormick Place North, Chicago, IL. (Teri Flotron, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

(Nov. 6-9)—11th Annual Advanced Composites Conference and Exposition. Sponsored by ESD-Engineering Society and SAE International. Hyatt Regency, Dearborn, MI. (Wael Berrached, The Engineering Society, 2350 Green Rd., Ste. 190, Ann Arbor, MI 48105).

(Nov. 7-9)—5th Annual Advanced Coatings Technology Conference and Exposition. Sponsored by ESD-Engineering Society and SAE International. Hyatt Regency, Dearborn, MI. (Wael Berrached, The Engineering Society, 2350 Green Rd., Ste. 190, Ann Arbor, MI 48105).

(Nov. 10-16)—1995 International Conference and Exhibition. Sponsored by the Steel Structures Painting Council (SSPC). Dallas, TX. (Dee Boyle, SSPC, 4516 Henry St., Ste. 301, Pittsburgh, PA 15213-3728).

# Asia

(May 23-24)—First Pacific Coating Forum. Sponsored by the Japan Coating Technology Association. Shonan Kokusai Village, Kanagawa, Japan. (Secretariat, Japan Coating Technology Association, Daitchi Naka-Bidg. 4F, 3-4. Nihomkabi Kohunacho Chuouku. Tak



Nihombashi-Kobunacho, Chuouku, Tokyo 103, Japan).

# Australia

(July 20-22)—Surface Coatings Association Australia Conference and Exhibition. Southern Cross Hotel, Melbourne, Australia. (Kahren Giles, Conference Manager,



SCAA Conference and Exhibition, The Meeting Planners, 108 Church St., Hawthorn, VIC 3122).

# 1996 — Australia

(Jan. 18-25)—International Schools and Conference on X-ray Analytical Methods—AXAA '96. Sponsored by the Australian X-ray Analytical Association (AXAA), Inc. Sydney, Australia. (The Secretariat, AXAA '96, GPO Box 128, Sydney, NSW 2001, Australia).

#### Europe

(Apr. 27)—"Aspects of In-Can Tinting Schemes." Symposium Sociation of the Oil & Colour Chemists' Association (OCCA). Marriott Hotel, Slough, United Kingdom. (Yvonne Waterman, OCCA, Priory House, 967



Harrow Rd., Wembley HA0 2SF United Kingdom).

(May 15-18)—"Recycle '95." Forum and Exposition. Sponsored by Maack Business Services. Swissôtel, Zürich, Switzerland. (Maack Business Services, Moosacherstrasse 14, CH-8804 AU/Zürich, Switzerland).

(June 12-14)—"17th Annual International Conference on Advances in the Stabilization and Degradation of Polymers." Luzern, Switzerland. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(June 19-22)—"Science and Technology of Pigment Dispersion." Luzern, Switzerland. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(June 20-23)—"Polymer Blends and Alloys." Luzern, Switzerland. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(July 10-14)—"21st Annual International Conference in Organic Coatings Science and Technology." Athens, Greece. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(Aug. 21-25)—"Advances in Emulsion Polymerization and Latex Technology." Davos, Switzerland. (Gary W. Poehlein, Interdisciplinary Programs, Georgia Institute of Technology, Atlanta, GA 30332-0370).

(Sept. 11-15)—"Epitaxial Film Deposition and Characterization of Semiconductors." Short course sponsored by Continuing Education Institute (CEI)-Europe. Seehof Hotel, Davos, Switzerland. (Tina Persson, CEI-Europe AB, P.O. Box 910, S-612 25 Finspong, Sweden).

(Sept. 11-16)—"CMOS/BiCMOS Process Integration and Engineering." Short course sponsored by Continuing Education Institute (CEI)-Europe. Seehof Hotel, Davos, Switzerland. (Tina Persson, CEI-Europe AB, P.O. Box 910, S-612 25 Finspong, Sweden).

(Sept. 11-19)—"Thick and Thin Film Microelectronics and Related Technologies Including Quality and Reliability." Short course sponsored by Continuing Education Institute (CEI)-Europe. Seehof Hotel, Davos, Switzerland. (Tina Persson, CEI-Europe AB, P.O. Box 910, S-612 25 Finspong, Sweden). (Sept. 11-21)—"Silicon Semiconductor Materials and Process Technology." Short course sponsored by Continuing Education Institute (CEI)-Europe. Sechof Hotel, Davos, Switzerland. (Tina Persson, CEI-Europe AB, P.O. Box 910, S-612 25 Finspong, Sweden).

(Sept. 12-13)—"Merging of Precision Engineering with Micro Fabrication." Short course sponsored by Continuing Education Institute (CEI)-Europe. Seehof Hotel, Davos, Switzerland. (Tina Persson, CEI-Europe AB, P.O. Box 910, S-612 25 Finspong, Sweden).

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(Sept. 16-23)—"Metallization Systems for VLSI and ULSI Circuits." Short course sponsored by Continuing Education Institute (CEI)-Europe. Seehof Hotel, Davos, Switzerland. (Tina Persson, CEI-Europe AB, P.O. Box 910, S-612 25 Finspong, Sweden).

(Sept. 18-21)—"Yield and Reliability in VLSI Development and Manufacturing." Short course sponsored by Continuing Education Institute (CEI)-Europe. Seehof Hotel, Davos, Switzerland. (Tina Persson, CEI-Europe AB, P.O. Box 910, S-612 25 Finspong, Sweden).

(Sept. 19-21)—"Eurocoat '95." Congress-Exhibition organized by AFTPV. Eurexpo Conference Center, Lyons, France. (E. Andre, UATCM, 5 rue Etex, F-75018 Paris, France).

(Sept. 19-22)—"VLSI Lithography and Nanolithography." Short course sponsored by Continuing Education Institute (CEI)-Europe. Seehof Hotel, Davos, Switzerland. (Tina Persson, CEI-Europe AB, P.O. Box 910, S-612 25 Finspong, Sweden).

(Sept. 26-28)—"Surcon '95." Biennial International Conference sponsored by the Oil & Colour Chemists' Association. London, Heathrow. (Chris Pacey-Day, 967 Harrow Rd., Wembley HAO 2SF, United Kingdom).

(Oct. 16-20)—First International Congress on Adhesion Science and Technology (ICAST). Amsterdam, The Netherlands. (ICAST '95, P.O. Box 346, 3700 AH Zeist, The Netherlands).

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NOTE: The Advertisers' index is published for the convenience of our readers and as an additional service to our advertisers. The publisher assumes no liability for errors or omissions. ohn Warner, our faithful fun pun correspondent, sent a copy of a column by Clarence Petersen of the *Chicago Tribune* which celebrates the Pundit, a newsletter published by the International Save the Pun Foundation. Norman Gilbert, Chairman of the Bored of the Foundation, featured the "best" of 1994, which appeared in the Petersen column. Cautious, as always, of dire consequences, we give you half of them this month. When I think I have given enough time for recovery we'll publish the balance.

➡ Maurie Cook of the TV show "Sugar and Spice" signs off this way: "Well, it looks like my thyme's up. I'd like to thank Basil my producer, Rosemary my technical adviser, and Ginger my director. Tune in tumeric for another kitchen caper. Dill we meet again, have a spice day."

• Attorney Balderson and Wanda Divorce are discussing the dissolution of the latter's marriage.

"Now, what are your grounds?" asks Balderson.

"Oh, we have some, two acres on the edge of town," Wanda replies.

"No, no. Do you have a grudge?"

"Yes, it fits two cars and is attached."

"I have to allege some wrongful act. Does your husband beat you up?"

"Oh, no sir, I'm out of bed every morning before he is." "Wanda, why do you want a divorce?"

"Well, you see, we have this problem communicating." Wanda eventually got the divorce on grounds of incompatibility.

Her husband lost his income and she lost her patability.

A tutor who tooted a flute Tried to tutor two tooters to toot. Said the two to the tutor, "Is it easier to toot or To tutor two tooters to toot?"

➡ Gary, the kayak paddler of the Iceland Olympic Team, was training in the Arctic. His feet became so cold that to warm them he built a fire in the bow of the boat. The kayak burned and sank. Gary now knows that you can't have your kayak and heat it, too. In related news, Bill took Kate as his wife while still married to Edith. Sentencing him for bigamy, the judge declared, "You can't have your Kate and Edith, too."

 "We saw the man commit the crime!" The witness related. The jury agreed, and he in time to jail was relegated. When someone later confessed He was dis-criminated.

# \*\*\*\*\*\*

arry Hill, the paint industry's gift to Australia, in October's issue of *Surface Coatings Australia* had some delightful examples of why his column "Overspray" has become so popular Down Under. For example, Larry writes: —Up here in Queensland, anything can happen because ... well, Queensland is different. I heard recently of a local politician whose career was failing dismally, with a string of lost causes behind him. He was to the point of despair when Old Nick (in Australian—"the Devil"—*Ed.*] appeared to him. "I will give you a series of incredibly complex issues which you will win against your most gifted opponents. Your career will flourish, you will enjoy riches and respect. You will become a cabinet minister and finally Prime Minister of Australia. But in exchange I must have the souls of all your children."

The politico's eyes narrowed in distrust. "What's the catch?" he asked.

—Then there was the executive who quit the rat race and joined a silent order of monks. They were only allowed to speak two words a year. After the first twelve months the novice knelt before the abbot. "Food bad," he said. A year later, again on his knees, he said, "Bed hard," Finally, at the end of his third year, he knelt before his superior and said, "I quit."

To which the abbot replied, "I'm not surprised—you've done nothing but bitch since you've been here."

My thanks to Frank Borrelle for keeping me supplied with photocopies of "Overspray."

# \*\*\*\*\*\*\*



riting of the "Save the Pun Foundation" and the story above, reminded me of one I had read a long time ago in "The Lion":

➡ When hard times came to the monastery, the abbot decided to set up a fish and chips stop at the entrance.

Brother James was put in charge of frying the fish and Brother Luke was put in charge of frying the potatoes. The local citizens soon learned to ask for one man or the other when they wanted fish or just chips. One night a newcomer to the area went in to the monastery. He wanted just fish and said to the man behind the counter, "I say, are you the fish friar?"

"No," said Brother Luke, "I'm the chip monk."

And in the same issue:

 The owner of a big corporation called his personnel director and said, "My son will be graduating from college soon and will need a job. He's going to be your new assistant, but he's not to be shown any favoritism. Treat him as you would any son of mine.

→ A tourist, camera in hand, climbed the pasture fence and headed toward a grazing bull. "Hey, you there!" he called to the farmer. "Is this bull safe?"

"A durn sight safer than you are!" the farmer replied. —The Lion, October 1990

> —Herb Hillman, Humbug's Nest, P.O. Box 135, Whitingham, VT 05361.



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