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**“Coatings in the Americas:  
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Courtyard Convention Center  
St. Louis, MO  
October 9-10-11, 1995

**23** Epoxy Nucleophile Catalyzed Transesterification (First Place Winner in the 1994 Roon Awards Competition)—C.P. Craun

*A novel and useful application of a well-known reaction is introduced. Epoxy is used as a crosslinker/initiator for transesterification reaction between polyacrylates and polyhydroxy compounds.*

**31** Initiators for High-Solids Polyacrylates Resins: Selection Guidelines and Performance Review—G.G. Myers

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**47** Styrenation of Oils Based on Secondary Esters of Castor Oil—O.S. Kabasakal et al.

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# C O M M E N T

## Don't be Left Out



The Federation's educational gears have been grinding rapidly over the last few months to prepare a slate of programs for 1995. After much investigation, review and discussion, three events have been developed and will be held in the first six months of 1995.

The schedule starts on Tuesday and Wednesday, March 21-22, 1995 with the "Formulating for the New Clean Air Act" at the Airport Marriott in Cleveland, OH. An impressive array of speakers will discuss the trends in formulating low VOC coatings to meet the Federal Clean Air Act guidelines. The program's sponsor, FSCT's Professional Development Committee (PDC), is intent on building on the success of last year's event of the same title, and has fine tuned the program to make it more pertinent to the regulatory changes happening in the coatings industry.

The 1995 Spring Week technical program has an international flavor in both location and content. The theme, "Coatings in the Americas: Regulatory and Technological Issues" focuses on three important ingredients for success in the coatings marketplace: the embracing of the new technologies; a commitment to quality and quality-related programs such as ISO 9000; and a thorough understanding of the governmental regulations impacting the industry. This year's seminar will be held at the Fiesta Americana Coral Beach in Cancun, Mexico on Wednesday and Thursday, May 17-18, 1995.

The PDC has also developed the new seminar, "Polymer Chemistry for the Coatings Formulator," scheduled for Tuesday and Wednesday, June 20-21, 1995 in Chicago, IL. Aimed at the paint formulator, this seminar was created after reviewing suggestions from previous FSCT program attendees regarding the kinds of events they would like to see from the Federation. The two day program will feature thirteen noted speakers delivering presentations on topics such as Characterization, Phenolics, and Condensation.

One challenge facing everyone is remaining informed about the necessary data to perform effectively in the workplace. These educational events are one way to prevent yourself from falling into the den of the uninformed. Mark your calendar now to attend one or more of these FSCT educational programs and insure being technically up-to-date.

Michael G. Bell  
Director of Educational Services

Translations provided by: French—Montreal Society Member Mario Côte of Eastman Chemical Canada Inc.-Montreal;  
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## Epoxy Nucleophile Catalyzed Transesterification—G.P. Craun

JCT, 67, No. 841, 23 (Feb. 1995)

Epoxy nucleophile catalyzed transesterification (ENCAT) is a new cure technology, which is particularly well-suited for coatings applications. Acrylic and acrylic-polyester blends in high-solids paints self-cure with ENCAT under normal bake conditions to give an excellent balance of hardness, flexibility, and solvent resistance. Powder paints prepared with ester derivatives of epoxy resins self-cure, and likewise give an excellent balance of properties. The ENCAT catalyst is formed during cure by the combination of a nucleophile with an epoxide moiety. A strong base alkoxide, which is produced when the epoxide ring opens by nucleophilic attack, initiates transesterification. This mechanism is supported by  $^{13}\text{C}$  NMR and gas chromatographic studies. Rate of transesterification is related to the structure of the hydroxyl and ester groups undergoing transesterification. Transesterification rate is directly related to the concentrations of the nucleophile and epoxy co-catalysts, as well as to the basicity and nucleophilicity of the nucleophile.

## Transestérification Catalysé par un Nucléophile Epoxy—G.P. Craun

La transestérification catalysé par un nucléophile epoxy (ENCAT) est une nouvelle technologie de durcissement qui convient particulièrement bien aux applications de revêtements. Les acryliques et mélanges acryliques-polyesters dans les peintures à haute teneur en solides se durcissent par eux-mêmes avec ENCAT sous des conditions de cuisson normale pour donner un excellent mélange de dureté, flexibilité et résistance aux solvants. Des revêtements en poudre préparés avec des dérivés d'esters de résines époxy se durcissent par eux-mêmes et donnent aussi un excellent mélange de propriétés. Le catalyseur ENCAT est formé durant le durcissement par combinaison d'un nucléophile avec la moitié d'un l'époxyde. Une forte base alkoxyde, qui est produite lors de l'ouverture du cycle de l'époxyde par une attaque nucléophile, initie la transestérification. Ce mécanisme est supporté par des études RMN  $\text{C}^{13}$  et chromatographique en phase gazeuse. La vitesse de transestérification est reliée à la structure des groupements hydroxyle et ester participant à la transestérification. La vitesse de transestérification est directement reliée aux concentration du nucléophile et co-catalyseur epoxy, de même que la basicité et nucléophilicité du nucléophile.

## Transesterificaciacion Catalizada con Nucleofilos Epoxicos—G.P. Craun

La transesterificaciacion catalizada con nucleófilos epóxicos (ENCAT) es una tecnologia nueva para el curado, particularmente en aplicaciones para recubrimientos. Las mezclas acrílicas y poliéster-acrílicas en pinturas de altos sólidos curan por si mismas con el ENCAT bajo condiciones normales de horneado, dando como resultado en excelente balance de dureza, flexibilidad y resistencia a solventes. Las pinturas en polvo preparadas con esteres derivados de resinas epóxicas curan por si mismas, y de la misma forma proporcionan un excelente balance en sus propiedades. El catalizador ENCAT se forma durante el proceso de curado por la combinación de un nucleófilo con una molécula epóxica. Un alcóxido de base fuerte, se produce cuando el anillo epóxico se abre mediante un ataque nucleofílico, iniciando así la transesterificaciacion. Este mecanismo se apoya en estudios de cromatografia de gases y resonancia magnética nuclear (RMN) base  $^{13}\text{C}$ . El grado de transesterificaciacion está relacionado con la estructura de los grupos de esteres e hidróxilos participantes. El grado de transesterificaciacion está directamente relacionado a las concentraciones de los catalizadores epóxicos y nucleofilos, así como a la basicidad y nucleofilidad del nucleófilo.

## Initiators for High-Solids Polyacrylates Resins: Selection Guidelines and Performance Review—G.G. Myers

JCT, 67, No. 841, 31 (Feb. 1995)

This paper covers the selection criteria for choosing an effective free-radical initiator for the polymerization of acrylic monomers. Two basic systems have been used to evaluate a large number of organic peroxides, including an original equipment manufacturer (OEM) model system. The systems and resin characterization procedures are described. Results of approximately 100 polymerizations using different initiators and conditions are presented. Table 6 summarizes all of the results obtained and can be used for initiator selection screening. Resin characterization, cost performance, and conversion are used as guidelines in all of the initiator evaluations.



**Initiateurs Pour Résines Polyacrylates à Haut Solides: Guide de Sélection et Revue de Performance—G. G. Myers**

Ce papier aborde le critère de sélection pour choisir un initiateur de radical libre efficace pour la polymérisation de monomères acryliques. Deux systèmes de base ont été utilisés pour évaluer un grand nombre de peroxydes organiques incluant un système modèle équipement original manufacturier (EOM). Les procédures de caractérisation de la résine et des systèmes sont décrites. Les résultats d'approximativement 100 polymérisations utilisant différents initiateurs et conditions sont présentés. La table finale présentée résume tous les résultats obtenus et peut être utilisée pour la sélection d'un initiateur. La caractérisation de la résine, le coût de performance et la conversion sont utilisés comme guide dans toutes les évaluations d'initiateurs.

**Iniciadores para Resinas de Poliácridatos de Altos Sólidos: Guías de Selección y Repaso de Funcionamiento—G.G. Myers**

Este documento contiene criterios de selección para escoger un iniciador efectivo de radicales libres para la polimerización de monómeros acrílicos básicos. Se han usado (dos sistemas básicos) para evaluar en gran número de peróxidos orgánicos, incluyendo un sistema OEM (fabricante de equipo original). Se describen los sistemas de procedimientos de caracterización de resinas. Se presentan resultados de aproximadamente 100 polimerizaciones usando diferentes iniciadores y a diferentes condiciones. La tabla final resume todos los resultados obtenidos y puede ser usado como un monitoreo preliminar en la selección de un iniciador. La caracterización de resinas, el costo y la conversión se usan como guías en las evaluaciones de iniciadores.

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**Novel Organosilane Crosslinking Agents for Powder Coatings—S.F. Thames et al.**

JCT, 67, No. 841, 39 (Feb. 1995)

Organosilane dicarboxylic acids have been substituted for a portion of the carboxyl-terminated polyesters in conventional epoxy-polyester powder coatings and provide improvements in a number of performance characteristics. For instance, environmental scanning electron microscopy (ESEM) studies confirm improvements in flow and leveling; the modified powders permit lower cure temperatures, and chemical and weather resistance properties are improved as is their thermal stability. Adhesion and pencil hardness are essentially unchanged, but minor improvements are noted in some formulations. However, impact strength and flexibility are lowered for most silane-containing formulations, probably a consequence of higher aromatic content in the silane diacids.

**Nouveaux Agents de Réticulations Organosilanes Pour Revêtements en Poudre—S.F. Thames et al.**

Les acides organosilanes dicarboxyliques ont été substitués pour une portion des époxy-polyesters avec terminaison carboxyle dans les revêtements en poudre conventionnels polyester et procure des améliorations dans plusieurs caractéristiques de performance. Par exemple, des études environnementales de microscopie à balayage électronique confirment des améliorations à l'écoulement et au nivellement; les poudres modifiées permettent des températures de durcissement inférieur; et les propriétés de résistance chimique et au climat sont améliorées comme leur stabilité thermique. L'adhésion et la dureté au crayon sont essentiellement inchangées, mais des améliorations mineures sont notées dans certaines formulations. Cependant, la solidité et la flexibilité à l'impact sont réduites pour la plupart des formulations contenant du silane, probablement une conséquence d'un contenu aromatique plus élevé dans les diacide silanes.

**Novedosos Agentes Organosilanos de Entrecruzamiento para Recubrimientos en Polvo—S.F. Thames et al.**

Los ácidos dicarboxílicos de organosilanos han sido sustituidos por una porción de los poliésteres de carboxilo terminal en recubrimientos convencionales en polvo de poliésteres epóxicos y proveen mejoras en varias de sus características. Por ejemplo, estudios de monitoreo ambiental por microscopía electrónica confirman las mejoras en flujo y nivelación; los polvos modificados permiten bajas temperaturas de curado, además se mejoran las propiedades químicas y de resistencia a la intemperie; su estabilidad térmica. La adhesión y dureza permanece básicamente sin cambio. Algunas formulaciones presentan cambios mínimos. Sin embargo, la fuerza de impacto y la flexibilidad disminuyen para la mayoría de las formulaciones que contiene silanos, probablemente como consecuencia de un alto contenido de aromáticos en los diácidos de silano.

**Styrenation of Oils Based on Secondary Esters of Castor Oil—O.S. Kabasakal et al.**

JCT, 67, No. 841, 47 (Feb. 1995)

The synthesis of styrenated oils based on secondary esters of castor oil is described. The steps of the process are as follows: (1) esterification of castor oil with fatty acids of drying and semidrying oils; (2) splitting of castor oil esters; and (3) conversion of the splitting mixture to either triglyceride or partial glyceride. While the triglyceride samples were styrenated by a conventional process, the partial glycerides' thermally labile azo groups were initially inserted into the oil molecule, and then this intermediate was reacted with styrene. The obtained styrenated oils showed good film properties.

**Styrénation d'huiles Basé sur les Esters Secondaires de l'huile de Ricin—O.S. Kabasakal et al.**

La synthèse d'huiles de styrène basé sur des esters secondaires d'huile de ricin est décrite. Les étapes du procédé sont (1) l'estérification de l'huile de ricin avec les acides gras d'huile sèche et semi-sèche, (2) la rupture des esters d'huile de ricin, (3) la conversion du mélange de rupture avec un triglycéride ou une glycérine partielle. Pendant que les échantillons de triglycérides étaient styrénés par procédé conventionnel, dans le cas de glycérines partielles, des groupements azo se modifiant aux changements thermiques étaient initialement insérés dans la molécule d'huile et par la suite cet intermédiaire fut réagi avec le styrène. Les huiles de styrène obtenues ont démontré de bonnes propriétés de film.

**Estirenciació de Aceites Basados en Esteres Secundarios de Aceite de Ricino—O.S. Kabasakal et al.**

Se describe la síntesis de aceites estirenados basados en ester secundarios de aceite de ricino. Los pasos en este proceso son: (1) esterificación del aceite de ricino con ácidos grasos de aceites secantes y semisecantes; (2) división de los ésteres de aceite de ricino, y (3) conversión de la mezcla dividida a un triglicérido o un glicérido parcial. Mientras las muestras de triglicérido fueron estirenadas por un proceso convencional, en el caso de glicéridos parciales térmicamente hábiles, grupos azo se insertaron inicialmente en la molécula de aceite y después este intermedio fue puesto a reaccionar con estireno. Los aceites estirenados que se obtuvieron mostraron buenas propiedades de película.



**NEW** Monographs in the  
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Coating Systems"**

by Jonathan W. Martin, Sam C. Saunders, F. Louis Floyd, and John P. Wineburg

For more information, contact  
Federation of Societies for Coatings Technology  
492 Norristown Rd., Blue Bell, PA 19422  
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## "Gateway City" Prepares for Federation's 1995 Annual Meeting & Paint Industries' Show

Travelling from more than 60 countries, over 8,500 coatings personnel are expected to attend the 73rd Annual Meeting and 60th Paint Industries' Show of the FSCT. The premier event of the industry, the Annual Meeting & Paint Show will be held on October 9-11 at the newly renovated Cervantes Convention Center, St. Louis, MO.

Focusing on the theme, "Creativity + Adaptability = Gateway to Success," programs and exhibits will highlight a wide array of innovative products and exciting new technologies.

Exhibit space for the Paint Show is being contracted quickly, with nearly 200 companies reserving booth space to date.

Following is a list of companies who have contracted for exhibit space in the 1995 Paint Industries' Show (as of February 1, 1995).

For information about exhibiting or attending this event, contact FSCT Headquarters, 492 Norristown Rd., Blue Bell, PA 19422-2350; phone: (610) 940-0777 or fax: (610) 940-0292.



Aceto Corp.  
ACT Laboratories, Inc.  
Advanced Software Designs  
AI Process Systems Ltd.  
Air Products & Chemicals  
Air Quality Sciences, Inc.  
AKZO Nobel Chemicals &  
AKZO Nobel Resins  
Alar Engineering Corp.  
Alcan-Toyo America, Inc.  
AlliedSignal Corp.  
Allied Colloids  
American Chemical Society  
Industry Relations  
Amoco Chemical Co.  
Angus Chemical Company  
Anker Labelers USA Inc.  
Aqualon  
Arco Chemical Company  
Ashland Chemical, Inc.  
Atlas Electric Devices  
B.A.G. Corporation  
BASF Corporation  
BatchMaster Software, Inc.  
Bohlin Instruments, Inc.  
Brookfield Engineering Laboratories  
Brookhaven Instruments  
Buckman Laboratories  
Burgess Pigment Co.  
BYK-Chemie USA  
BYK-Gardner, Inc.  
Cabot Corp.—CAB-O-Sil &  
Special Blacks Div.  
Caframo Ltd.  
Calgon Corp.  
Cardolite Corporation  
CB Mills, Div. of Chicago Boiler  
CCP Polymers  
Chemical & Eng. News,  
American Chemical Society  
CIBA-GEIGY Corp., Additives,  
Polymers and Pigments  
Coatings Laboratory Inc.  
Color Corporation  
Consolidated Research Inc.  
Cortec Corp.  
Coulter Corporation  
Scientific Instruments  
CR Minerals Corporation  
Crosfield Company

CYTEC Industries Inc.  
D/L Laboratories  
Daniel Products Co., Inc.  
Datacolor International  
Degussa Corporation  
University of Detroit-Mercy  
Disti-Kleen, Inc.  
Dominion Colour Corp.  
Dow Chemical Co.  
Dow Corning Corp.  
Draiswerke, Inc.  
Drew Industrial Division—Ashland  
Dry Branch Kaolin Company  
Eastern Michigan University  
Eastman Chemical Company  
Ebonex Corporation  
Eiger Machinery, Inc.  
Elcometer, Inc.  
Engelhard Corporation  
Engineered Polymer Solutions  
Epworth Mfg. Co., Inc.  
Etna Products Inc.  
Specialty Chem Div.  
European Coatings Journal  
Exxon Chemical Company  
Fawcett Co., Inc.  
F S C T  
Filter Specialists, Inc.  
Fischer Technology, Inc.  
Fluid Management  
FMJ International Publications Ltd.  
Fryma, Inc.  
Paul N. Gardner Co., Inc.  
Georgia Pacific Resins, Inc.  
The BFGoodrich Co.  
Specialty Chemicals  
Goodyear Tire & Rubber Company,  
Chemical Div.  
Guertin Polymers  
Haake, Inc.  
The C.P. Hall Company  
Halox  
Harcros Pigments Inc.  
Heraeus DSET Laboratories  
Hilton Davis Co.  
Hoechst Celanese Corporation  
Horiba Instruments Inc.  
J.M. Huber Corporation  
Huls America, Inc.  
HunterLab

Ideal Mfg. & Sales Corp.  
International Compliance Center  
ITT Marlow Pumps/ITT A-C  
Pumps  
Kady International  
Kemira Pigments, Inc.  
Kenrich Petrochemicals, Inc.  
King Industries, Inc.  
LaQue Center—Kure Beach  
Atmospheric Testing  
Lawter International  
The Leneta Company  
Liquid Controls Corp.  
Littleford Day Inc.  
The Lubrizol Corporation  
Lucas Meyer, Inc.  
Luzenac America  
3M/Zeelan Industries, Inc.  
3M, Specialty Chemicals  
MacBeth—Div. of Kollmorgen  
Malvern Instruments  
McWhorter Technologies  
The Meurl Corporation  
Micro Powders, Inc.  
Micromeritics  
Mid-States Eng. & Mfg. Co.  
Millipore Corporation  
Milwhite, Inc.  
Mineral Pigments Corp.  
MiniFIBERS, Inc.  
Minolta Corporation  
Mississippi Lime Co.  
University of Missouri-Rolla  
Morehouse-COWLES, Inc.  
Morton International—UCD  
Myers Engineering  
Nacan Products Limited  
Nametre Company  
Netzsch Incorporated  
Neupak Inc.  
New Way Packaging Machinery  
North Dakota State University  
Obron Atlantic Corp.  
Ohio Polychemical Co.  
Olin Chemicals  
OSI Specialities, Inc.  
Paar Physica USA, Inc.  
Paint & Ctg. Ind. Magazine  
Peninsula Polymers  
Phenoxy Associates  
Polar Minerals  
Poly-Resyn, Inc.  
PPG Industries, Inc.  
Silica Products

PPG Industries, Inc.—Specialty  
Chemicals Group  
Premier Mill Corp.  
P Q Corporation/Potters Ind.  
Pyosa S.A. De C.V.  
Q-Panel Lab Products  
Raabe Corporation  
Radtech International North  
America  
Ranbar Technology, Inc.  
RHEOX, Inc.  
Rhone-Poulenc  
Rohm and Haas Co.  
Ronningen-Petter  
Charles Ross and Son Co.  
Russell Finex, Inc.  
Sandoz Chemicals Corporation  
Schold Machine Co.  
Semi-Bulk Systems Inc.  
SEPR (Ceramic Beads & Powders)  
Shamrock Technologies Inc.  
Shell Chemical Company  
Sherwin-Williams Chemicals  
Silberline Mfg. Co., Inc.  
Snyder Industries  
Software 2000 Inc.  
South Florida Test Service  
Southern Clay Products Inc.  
Univ. of Southern Mississippi  
Specialty Minerals Inc.  
Startex Chemical Co.  
Steel Shipping Container Institute  
Sub-Tropical Testing Service  
Sud-Chemie Rheologicals  
21st Century Containers Ltd.  
Tego Chemie Service USA  
Thiele Engineering Company  
Troy Corp.  
U.S. Borax, Inc.  
U.S. Polymers, Inc.  
U.S. Silica Company  
Union Process, Inc.  
R.T. Vanderbilt Co., Inc.  
Versa-Matic Pump Company  
Vorti-Siv Division/MM Industries  
Wacker Silicones Corporation  
Witco Corporation  
World Minerals/Celite Corporation  
X-Rite, Inc.  
Zeneca Biocides  
Zeneca Resins

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### **Formulating for the New Clean Air Act**

**March 21-22, 1995  
Cleveland Airport Mattiotti  
Cleveland, Ohio**

**Contact: Federation of Societies for Coatings Technology  
492 Norristown Rd., Blue Bell, PA 19422-2350  
Phone: (610) 940-0777 • Fax: (610) 940-0292**



## FSCT Invites Nominees for 1996 Mattiello Lecturer

The Federation of Societies for Coatings Technology is seeking nominations for the 1996 Joseph J. Mattiello Lecturer. The Mattiello Lecture will be presented at the 74th Annual Meeting of the Federation, to be held October 23-25, 1996, in Chicago, IL.

The lecture commemorates the contributions of Dr. Joseph J. Mattiello, former President of the Federation, who was instrumental in expanding the application of the sciences in the decorative and protective coatings fields.

The Mattiello Lecture Committee will select a person recognized for outstanding contributions to science, technology, and engineering related to the coatings industry to present a paper on a phase of chemistry, engineering, human relationship, or other discipline fundamental to paint, varnish, lacquer, or related protective and decorative coatings. The Mattiello Lecturer shall embody the standards of technical accomplishment, service to the coatings industry, and leadership established by Joseph J. Mattiello.

All nominations for the Mattiello Lecturer shall include the following information:

- Name, age, and place of birth
- Current position and brief job history
- Education and degrees, with dates
- Brief reference to other significant awards
- Description and significance of accomplishments that are deemed to qualify the nominee for the award.

The items cited should be concerned with coatings, be pertinent or related to coatings, or be concerned with the constituents of coatings. Some activities and accomplishments which are considered to be appropriate for citation in the nomination are the following:

- Publication and communications such as the following:
  - Journal articles
  - Patents
  - Books (authored, edited, or organized)
  - Chapter in books
  - Lectures and presentations
  - Symposia or meetings organized

(Reprints may be submitted with the nomination but should be restricted to those that reflect seminal contributions; teaching

credentials or skills, per se, are not to be considered in this award.)

- Inventions and discoveries
- New scientific principles, understanding or insight
- New or improved products
- New or improved instrumentation or testing methods
- New or improved analytical methods
- Novel uses or applications of products
- New or improved processes for production of resins or coatings, etc.
- Engineering aspects
- New or improved application methods

Please forward all nominations by May 1, 1995, to Patricia D. Viola, Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422-2350.

### Nominations Sought for Armin J. Bruning Award

Dr. Robert T. Marcus, Chairman of the Bruning Award Committee, has announced that nominations are being sought for the 1995 recipient of this award. Established to recognize an individual for outstanding contributions to the science of color in the field of coatings technology, the award commemorates Armin J. Bruning, the inventor of the Davis-Bruning colorimeter. Mr. Bruning was noted for his devotion to the pursuit of the scientific study of color.

A nominee for the award must have contributed significantly to the field of color study. These contributions could include very basic work which increases our understanding of the interaction of colorants, light, and observers (human and instruments). However, the contributions of the nominee could also include various aspects of techniques or theories developed by others, or the teaching of color science or the dissemination of information in a manner which has direct benefit to the coatings industry. The nominee does not have to be a member of a Society or the FSCT.

To nominate an individual for the Bruning Award, please contact Dr. Marcus, Chairman, at Pantone, Inc., 590 Commerce Blvd., Carlstadt, NJ 07072-3098, (201) 935-5500.

The nomination must include documentation concerning the nominee's qualifications and contributions in the area of color science for the coatings industry. Nominations must be received prior to May 12, 1995.

## Panorama™ to Interface with BatchMaster® Software

Panorama™ was developed by the Federation to aid in storing, accessing, and retrieving Material Safety Data Sheets for the coatings industry. This popular CD-based database now contains the MSDS of more than 20,000 raw materials.

Since Panorama was introduced in early 1994, subscribers have asked if the program could be used to create finished product MSDS. Currently, the only way one can use the information in Panorama for new product MSDS is through "cutting and pasting." In the very near future, there will be another solution.

BatchMaster Software Corp., Seal Beach, CA, is in the process of developing an interface between Panorama and BatchMaster. The BatchMaster system "manages the entire manufacturing operation information, from raw materials purchase to production to sales," says Christy Hudson, of BatchMaster. She also states that the software is "finely tuned for paint manufacturers, who work from formulas, and ties in all of the unique aspects of the chemical industry."

BatchMaster automates and integrates many everyday tasks, handling all details from new product development through to finished goods production. Specific modules exist for a variety of applications, including inventory, production, laboratory, MSDS/Compliance, costing, planning, report writ-

ing, labeling, bar code, purchasing, accounts payable and receivable, general ledger, order entry, etc.

In the MSDS/Compliance module, MSDS can be generated on new formulations automatically, based on a variety of product characteristics. This module also determines when customers need new or revised MSDS. The entire MSDS format is under the user's control, so it is guaranteed to comply with present and future requirements regardless of country or locality.

The BatchMaster/Panorama interface will be developed because, as Ms. Hudson states, "Panorama has become extremely popular and many of our customers are asking for it so they can minimize data input and errors." Randy Peck, of BatchMaster, comments, "we've been working hard on it and we're going to continue to do so. This is a priority."

For further details on the BatchMaster/Panorama interface, contact Christy Hudson, BatchMaster Software Corp., 1500 Pacific Coast Highway, Suite E, Seal Beach, CA 90740; phone: (310) 799-8888; FAX: (310) 799-8833.

For more information on Panorama, contact Patricia Viola or Christine DeLissio, Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422; phone: (610) 940-0777; FAX: (610) 940-0292.

# FSCT 1995 SPRING WEEK REGISTRATION FORM

Register Today! Advance housing registration closes March 31, 1995.  
 Complete this form and mail or fax with payment to:  
**FSCT, 492 Norristown Rd., Blue Bell, PA 19422-2350 USA**  
**610-940-0777 ■ FAX: 610-940-0292**



## Seminar Program

Name \_\_\_\_\_ Nickname (For badge) \_\_\_\_\_

Company \_\_\_\_\_

Mailing Address \_\_\_\_\_

City \_\_\_\_\_ State (Province) \_\_\_\_\_ Country \_\_\_\_\_ Mailing Code \_\_\_\_\_

Phone \_\_\_\_\_ Fax \_\_\_\_\_

### COMPANY (Check one)

- Manufacturers of Paints, Varnish, Lacquer, Printing Inks, Sealants, etc.
- Manufacturers of Raw Materials
- Manufacturers of Equipment and Containers
- Sales Agents of Raw Materials and Equipment
- Government Agency
- Research/Testing/Consulting
- Environmental Services
- Educational Institution
- Paint Consumer
- Other \_\_\_\_\_

### POSITION (Check one)

- Management/Administration
- Manufacturing and Engineering
- Quality Control
- Research & Development
- Sales and Marketing
- Consultant
- Educator/Student
- Other \_\_\_\_\_

**FSCT Member Fee** Name of FSCT Society \_\_\_\_\_ **\$225 US per person** \$ \_\_\_\_\_

**Non-Member Fee** **\$295 US per person** \$ \_\_\_\_\_

## Hotel Reservations (Deadline for special group rate is March 31, 1995)

Name of Occupant(s) \_\_\_\_\_

Arrival Date \_\_\_\_\_ Arrival Time \_\_\_\_\_

Departure Date \_\_\_\_\_

**Room Type:**  Single - \$135 US  Double - \$135 US  Smoking  Non-smoking

Special needs: Please specify \_\_\_\_\_

**Deposit Information:** One night's deposit<sup>(1,2,3)</sup> is required **Deposit of \$148.50 US** \$ \_\_\_\_\_

- Note:** (1) Deposit includes one night's room rate (\$135) plus 10% tax.  
 (2) Hotel cancellations received **within seven (7) days** of arrival will incur a penalty of one night's room fee, plus tax  
 (3) **No-shows** will incur a penalty of two night's room fee, plus tax

## Post Seminar Excursions and Airport Shuttle Transportation

May 19 — Tulum and Xcaret No. of Persons \_\_\_\_\_ @ **\$79 US per person** \$ \_\_\_\_\_

May 20 — Markets to Malls No. of Persons \_\_\_\_\_ @ **\$15 US per person** \$ \_\_\_\_\_

Airport Shuttle Transportation No. of Persons \_\_\_\_\_ @ **\$10 US One-Way** \$ \_\_\_\_\_

**Airline** \_\_\_\_\_ **Flight No.** \_\_\_\_\_

**Note:** Excursion and Airport Shuttle cancellations received after **May 9** are non-refundable

## Calculate Total Payment Due

**Please note choice of payment:**

Check enclosed. Make payable to FSCT in U.S. Funds

Please charge to the following card:  MC  VISA  AMEX **Total Payment Due FSCT:** \$ \_\_\_\_\_

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# Coatings Industry Honor and Remembrance Fund

In mid-1992, the Coatings Industry Education Foundation (CIEF) was proud to announce the establishment of the Coatings Industry Honor and Remembrance Fund, which is administered by the Trustees of the CIEF. The concept of an "Honor and Remembrance" fund was new to our industry, and was first suggested by 1993 FSCT President Colin Penny, who felt that many of us would like to honor friends, spouses, respected colleagues and outstanding employees in a public and substantive way. Colin felt that—by establishing a fund dedicated specifically to the honor and/or remembrance of those special people—the CIEF would be creating a meaningful and lasting tribute to their work. Since the intent of the Trustees is to use the earnings from the principal of this fund for educational assistance in the form of scholarships, fellowships, and grants to colleges and universities with coatings programs, anyone making a donation will also have the additional satisfaction of knowing that their gift will be used to help educate those who will carry on the tradition of scientific and technological excellence in the coatings indus-

try—and who may very well be honorees of this same fund, someday.

By recognizing gifts to the Honor and Remembrance Fund in the JOURNAL OF COATINGS TECHNOLOGY, it is the Trustees' intent to give international recognition to both the donors and the honorees, as well as to focus on the educational benefits being derived from such gifts. This is a bold new venture which will enable every individual, corporation, and society associated with the coatings industry to really "make a difference," by recognizing specific individuals' contributions to our industry, while furthering the educational efforts of the CIEF at the same time. Gifts in any amount (made payable to "CIEF—Honor and Remembrance Fund," and sent to the Federation Office) will be recognized annually in the JOURNAL OF COATINGS TECHNOLOGY, and will be divided into the following five categories:

- ◆ Gifts up to \$249
- ◆ Gifts from \$250-\$499
- ◆ Gifts from \$500-\$999
- ◆ Gifts from \$1000-\$9999
- ◆ Gifts of \$10,000 and greater

All gifts to the Honor and Remembrance Fund will be tax deductible to the extent that the law allows, and may be made as a direct gift to the Fund, as a gift in honor of a living person, or as a gift in remembrance of one who is deceased. Examples:

- ◆ The Acme Corporation
- ◆ The Ohio Coatings Society, in honor of Maynard Q. Browning
- ◆ Mr. and Mrs. Pierre M. Lundquist, in remembrance of John Z. Edwardson

It is currently the intention of the Trustees to reprint the list annually, with some indication of gifts which have been added since the last printing. The Trustees also placed a plaque, in 1994, in the Federation Office in Blue Bell, listing all donors of record, with sufficient room to add additional donors in the future. As of December 1994, the Honor and Remembrance Fund has received generous, and deeply appreciated, gifts from the following individuals:

## The Coatings Industry Honor and Remembrance Fund

(Donations through December 31, 1994)

### \*Gifts of \$10,000 and Greater\*

*Len and Neta Schaeffer, in remembrance of  
Fred and Ruth Daniel*

### \*Gifts of \$1000-\$9999\*

*Mrs. Herbert L. Fenburr, in remembrance of  
Dr. Herbert L. Fenburr*

*The Houston Society for Coatings Technology, in remembrance  
of Loren B. Odell*

*The Dallas and Houston Societies for Coatings Technology*

*John J. Oates, in remembrance of Elias Singer*

### \*Gifts of \$500-\$999\*

*Akzo Coatings Inc. (Columbus), in remembrance of our  
employees who died in 1992*

*The Baltimore Society for Coatings Technology, in remembrance  
of our deceased members*

*The Baltimore Society for Coatings Technology, in remembrance  
of Richard D. McCloskey\**

### \*Gifts of \$250-\$499\*

*Colin D. Penny\**

*Saul Spindel, in honor of Sidney B. Levinson\**

### \*Gifts up to \$249\*

*The Birmingham Paint, Varnish, and Lacquer Club, in  
remembrance of Ray Mowl and Ken Cooke*

*Sidney Lauren, in remembrance of Fred G. Schwab*

*Mr. & Mrs. George R. Pilcher, in remembrance of  
Helen Skowronska*

*Mr. and Mrs. Hiram P. Ball, in remembrance of  
Fred G. Schwab\**

*Doris S. Schwab, in remembrance of Fred G. Schwab\**

\*Gift received during 1994

## Miles Inc. to Become Bayer Corp.; CEO Outlines Strategies for the Future

At the beginning of each new year, many resolve to make changes and plans for the future. One company making such plans is Bayer Corp., Leverkusen, Germany. In fact, at a news conference in New York City last month, Bayer Chief Executive Officer Manfred Schneider introduced the Bayer Group operations to the United States following the acquisition of the North American over-

### USN Seeks Sources for Fouling Systems

The Carderock Division Naval Surface Warfare Center, Annapolis, MD, is interested in fouling control coating systems for ship underwater hulls with a mode of action by biocide or by a naturally occurring agent or chemical analog of a natural agent, that are suitable for use on steel, aluminum, and rubber and that are VOC compliant. The following characteristics are also desirable:

- ⇒ Contains reduced amounts of copper, or no copper, and does not contain any other heavy metal toxicants;
- ⇒ Available in gray (FED-STD-595 Color Nos. 26270 and 26173);
- ⇒ Flash point not to be less than 70°F;
- ⇒ Shelf-life of at least two years;
- ⇒ Satisfactory spray, brush, and roll properties;
- ⇒ Satisfactory recoatability and storage stability; and
- ⇒ Compatible with cathodic protection.

Any offeror having an existing or developmental coating system which meets the above characteristics in whole or part should submit the following information: system description (including recommended anti-corrosive primer system); product data and application sheets, Material Safety Data Sheets; one-year panel data at a tropical fouling site (pictures are helpful); release rate data and method used to obtain the data, for any component that is released into the aquatic environment; EPA registration number for the active agent(s), if available, or status of registration process; and POC name and telephone number.

Respond to Karen Poole, Naval Surface Warfare Center, Carderock Division, Annapolis Detachment, 3A Legget Circle, Annapolis, MD 21402-5067. Questions may be addressed to Karen Poole at (410) 293-2275, or fax (410) 293-3052.

the-counter (OTC) business of Sterling Winthrop.

Joining Dr. Schneider in New York was Helge H. Wehmeier, President and CEO of Miles Inc., Pittsburgh, PA. As part of the acquisition, Bayer regained the rights to its name and trademarks in the United States, Canada, and the Caribbean. Therefore, Bayer's U.S. subsidiary, Miles Inc., will change its name to Bayer Corp. on April 1.

In 1994 Bayer's investment in the future included \$2 billion of capital investments and another \$2 billion on research and development. An additional \$1.4 billion was spent on acquisitions, including \$1 billion for the North American Sterling OTC business and the Bayer rights. "These investments," said Dr. Schneider, "are to secure future growth." Bayer plans to increase worldwide sales next.

Dr. Schneider stated that Bayer has been involved in the U.S. market since 1865, and the U.S. will continue to play a key role in Bayer's corporate plans by expanding "our activities here still further." This priority is due to continued growth in North America in 1994, along with considerable improvements in South America and Asia. He was optimistic for 1995 as well.

Three reasons were cited for Dr. Schneider's optimism: (1) business prospects in the major markets are good; (2) wide-ranging restructuring measures have been implemented; and (3) internal potential for growth. Opportunities for growth lie in sev-

eral areas including pharmaceuticals, consumer care, and diagnostics business groups.

In the health care segments, Bayer has two drugs with sales of some \$1.3 billion each. These drugs are Cipro, an anti-infective, and Adalat, a cardiovascular therapeutic. Bayer hopes to add more products by the year 2000 to increase sales.

Other areas have been targeted as well. Bayer will continue to expand its role as a supplier of polymeric materials. Dr. Schneider highlighted plastics such as Makrolon polycarbonate and its blends, synthetic rubber and rubber chemicals, and raw materials such as a MDI and TDI for polyurethanes as growth areas.

Miles Inc. has been a strong contributor to the Bayer Group. According to Mr. Wehmeier, Miles contributed nearly a quarter of the Bayer Group's worldwide turnover. Eighty percent of products sold by Miles were manufactured in the United States.

Miles spent some \$500 million in capital investments in 1994, major projects included a tripling of production capacity for maleic anhydride—precursor for coatings raw materials and plastics—at Baytown, TX.

Bayer Corporation's immediate plans include an advertising campaign aimed at opinion leaders, the business community, and political leaders. Mr. Wehmeier added, "While 90% of all people in this country recognize our new name, most think of it as only aspirin." Bayer is working to change that perception.

### Ralph Stanziola Learning Center Dedicated

Chroma Corp., McHenry, IL, recently dedicated its new customer conference facility as the "Ralph Stanziola Learning Center."

For the past eight years, Mr. Stanziola has been conducting the Chroma Customer Color Seminars which covered topics such as basics of color technology, in-plant technology, in-plant color control, and the ABCs of establishing color tolerances.

A member of the Philadelphia Society, Mr. Stanziola is President of Industrial Color Technology, Neshanic Station, NJ. In addition, he serves as a member of the JOURNAL OF COATINGS TECHNOLOGY Editorial Review Board and is Chairman of the Inter-Society Color

Council Committee of the Federation. Mr. Stanziola has been in the coatings field for 30 years and specializes in industrial color control.



Robert D. Swain (right), President of Chroma Corp., congratulates Ralph Stanziola at the dedication of Chroma's new Customer Conference Facility.



## NPCA Challenges U.S. Court of Appeals on CERCLA

In a brief filed with the Supreme Court, the National Paint and Coatings Association (NPCA), Washington, D.C., urged the U.S. Court of Appeals to review a ruling that could discourage useful recycling of industrial by-products by imposing the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA).

NPCA joined the American Petroleum Institute, National Association of Chemical Recyclers, and National Association of Printing Ink in support of granting ASARCO Incorporated's petition for Writ of Certiorari in *ASARCO v. Louisiana Pacific*. The question is whether or not manufacturers who recycle industrial by-products by selling them in good faith to other parties for productive use can be found liable under CERCLA for "waste disposal," where those by-products turn out to be defective for their intended use.

According to the brief filed by the associations, the decision of the U.S. Court of Appeals for the Ninth Circuit jeopardizes the manufacture, sale, and use of valuable products. In general, the brief emphasizes the importance of recycling and cites the "chilling" impact that expanding CERCLA

liability to this situation will have on the recycling industry and activities.

The associations argue that the lower court's decision does not sufficiently address the public policy implications of its decision for recycling, materials handling, and, specifically, ignores the practical, adverse im-

pact its decision will have on the Congressional goals of waste minimization or materials recovery. The brief also states that the lower court does not deal with the implications of its decision on consumers of valuable products made with and from secondary by-products.

### Mergers & Acquisitions

**AlliedSignal, Inc.**, Morristown, NJ, acquired the product and application technology of **Exxon Chemicals'** Escomer® line of synthetic polyethylene waxes, with the exception of one specialty grade.

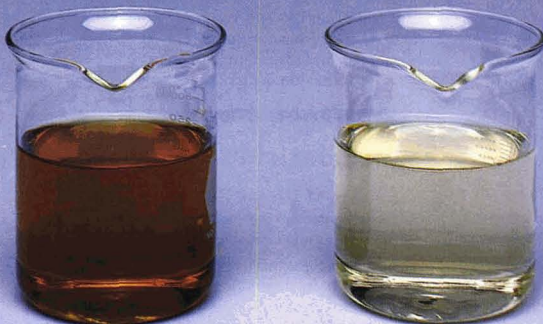
Under terms of the agreement, AlliedSignal will acquire all Escomer technology except for Escomer H101 product grade.

**Velsicol Chemical Corp.**, Rosemont, IL, acquired the benzyl alcohol business from Akzo Nobel Chemicals. The sale includes some capital equipment located at Akzo's, Edison, NJ, production facility.

**United States Filter Corp.**, Rockford, IL, acquired **L'eau Claire International (LCI)**, Inc. Based in Lacombe, LA, LCI specializes in upflow media filtration.

Selected assets of **Colorgen, Inc.**, have been purchased by **X-Rite, Inc.**, Grandville, MI. The acquired assets include the custom database software and paint formulations used by hardware and paint stores, retail chains, and paint manufacturers.

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# Regulatory Update February 1995

**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, Washington, 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.

## **Environmental Strategy Is Uncertain for the 104th Congress—**

The window of opportunity for passage of any major environmental legislation appears to be closed. The Democratic-controlled 103rd Congress was the best opportunity for environmental initiatives, yet due primarily to political maneuvering, nothing was passed. With the Republicans in charge of both the House and the Senate, it is unlikely that environmental issues will be a top priority.

For the first 100 days of the 104th Congress, the Senate and the House will be dealing with the *Contract with America* bills (see the following). Perhaps after those are completed and Congress settles into a routine, certain environmental statutes may be considered for reauthorization. Those statutes that have proven costly to industry may be the first to be reconsidered.

Among the issues affected by the change in legislative control are those summarized in the following.

**Superfund**—Valiant efforts to overhaul the Comprehensive Environmental Response, Compensation, and Liability Act (Superfund) during the 103rd Congress screeched to a halt at the end of the last session. Time-consuming political bickering was to blame. Killing the House bill was a last minute Davis-Bacon wage-rate amendment, while the Senate suffered overall partisan politics.

The legislation considered during the 103rd Congress would likely have been the starting point for the 104th had the Democrats retained control. In fact, Rep. John Dingell (D-MI), ranking minority member of the House Commerce Committee (formerly Energy and Commerce) has already introduced a Superfund bill based on legislation considered during

the last Congress. A spokesperson for the new committee chairman, Rep. Thomas Bliley (R-VA), has said that the chairman may bring up bipartisan bills from the last congress after the *Contract with America* bills are addressed.

Though the level of enthusiasm has diminished somewhat, there still appears to be enough interest in Superfund reform to warrant hearings in the committees with jurisdiction. Regardless of interest in reform, the tax authority for Superfund technically expires in 1995 and must be reauthorized. According to the Office of Management and Budget, Superfund can probably exist until the first part of 1996 on excess appropriations, but the taxing authority will have to be reauthorized during the second session of the 104th Congress.

If Congress does choose to debate Superfund, perhaps the biggest change from the last session's bills will be the emphasis on the elimination of retroactive liability. Last year, though specific retroactive liability bills were introduced, support was minimal. Now, Rep. Bliley as Chairman of the Commerce Committee and Rep. Bud Shuster (R-PA) as Chairman of the House Transportation and Infrastructure Committee, and as patrons of the elimination of retroactive liability, the issue may get the necessary backing.

**Clean Water Act**—If rewrites to the Clean Water Act are to be considered during the 104th Congress, they will likely be much narrower in scope than previous attempts. In the meantime, EPA will reportedly focus on implementation of various water initiatives including the regulation of storm water discharges.

Several water programs will be impacted by the reforms called for under the *Contract with America* bills.

In fact, if EPA is required to fulfill the *Contract's* requirements for cost/benefit analyses, many rules will come to a halt.

In the Senate, Environment and Public Works Chairman John Chafee (R-RI) has expressed an interest in certain aspects of clean water reform. Those include the reauthorization of the state revolving loan fund program, wetlands, storm water, and other types of pollution runoff.

The House, on the other hand, will use a bipartisan bill from the 103rd Congress as a starting base. That bill, drafted by House Transportation and Infrastructure Committee Chairman Bud Shuster (R-PA), reportedly addresses cost/benefit analyses, risk assessments, and unfunded mandates. A new proposal will also address the reauthorization of the state revolving loan fund, regulatory matters, and wetlands.

**Clean Air Act**—Despite rumors to the contrary, there appears to be a general consensus that the Clean Air Act will not be reopened to Congressional scrutiny. The primary reason being that the amendments of 1990 took their toll on those involved. However, while the act itself may not be reconsidered, the programs developed as a result of the amendments must be reviewed by a Congress obsessed with deregulation.

Federal agencies are already under an executive order from President Clinton to review existing regulations. Reportedly, the order states that regulations are to be implemented only if necessary and in the most cost-effective way.

Industry remains cautiously optimistic that the Republican Congress will closely scrutinize EPA. Needless to say, there are still regulations to be addressed by EPA in 1995 that industry will be watching



closely. Among them, the National Emission Standard for Hazardous Air Pollutants (NESHAP) for wood furniture manufacturing, off-site waste operations, coatings operations in shipbuilding and repair, and polymers and resins.

**Toxic Substances Control Act (TSCA)**—It is unlikely that TSCA legislation will be pursued during this Congress. Due to political maneuvering, Sen. Harry Reid (D-NV), the single most vocal proponent of TSCA initiatives, will not keep his position as ranking minority member on the Environment and Public Works Subcommittee on Toxic Substances, Environmental Oversight, and Research and Development, which has jurisdiction over TSCA issues. He has, however, stated that he will try his best to attach TSCA amendments to bills offered by his fellow colleagues on the Appropriations Committee. Sen. Reid has been a particular advocate of comprehensive lead initiatives.

The House lost its TSCA champion with the 1994 primary defeat of Rep. Mike Synar (D-OK).

Meanwhile, EPA's Office of Pollution Prevention and Toxics (OPPT) has designated lead paint rules as its top priority for 1995. Worker standards for lead-paint hazard activities, proposed September 2, 1994, will be the first rule issued under the lead program. October is the target date for adoption of the rule. Two other lead-paint rules are expected in the spring. According to EPA, the real estate disclosure rule and the remodeler disclosure rule will result in an estimated 28 million households annually receiving notifications of lead-paint hazards. Proposed standards on lead-based paint hazards and dangerous levels of lead in interior dust and soil are not expected before 1996.

**Regulatory Moratorium Bill Introduced**—Legislation that would place a temporary hold on all recent regulations, guidance, and policy statements except for deregulatory or emergency actions was introduced on January 9.

H.R. 450, introduced by Rep. Tom Delay (R-TX), requires that the implementation of any rule or guidance issued or scheduled to be issued since November 1994, be delayed until July 1. Any deadline occurring during the moratorium would be extended five months or until July 1, whichever is later. The

moratorium would not apply to regulatory waivers, exemptions or deregulatory efforts, and includes an exemption for actions addressing immediate threats to health or safety.

Reportedly, Republican staff are calling the bill the first step in minimizing government and improving regulations—part of the *Contract with America* promise. Not unexpectedly, the Clinton administration is opposing the bill. Instead of an across-the-board moratorium, the administration would prefer to identify and roll back regulations that are determined to be overly burdensome, while at the same time emphasize rules that are beneficial to industry and the economy.

Predictions are that the bill will sail through the House only to be filibustered by Senate Democrats.

#### How to Obtain Congressional Documents

##### Bills, Reports and Public Laws—

There is no charge for House and Senate bills, reports and public laws, but there is a limit to the number that may be obtained at one time. Policies differ in the House and Senate.

**Senate**—Senate bills, reports, public laws and conference reports are available in the Senate Document Room, B-04 Hart Senate Office Building. As many as six different items may be obtained per request, and only one request per person per day will be filled. Multiple copies of one item are not available. Documents may be obtained in person or by mail. Phone orders are not accepted. Mail orders may be sent to: Senate Document Room, B-04 Hart Building, Washington, D.C. 20510. For information on status and availability of legislative documents, call the Senate Document Room Information line at (202) 224-7860.

**House**—House bills, reports and public laws, and conference reports are available in the House Document Room, B-18, House Annex No. 2. The public may obtain as many as six different items per person. Up to 12 items may be requested by mail and six items by telephone. Multiple copies of one item are not available. Only one request per person per day will be filled. Mail orders may be sent to: House Document Room, B-18, House Annex No. 2, Washington, D.C. 20515.

For information on status and availability of legislative documents or

to order documents by phone, call the Document Room at (202) 225-3456.

#### Department of Transportation Research and Special Programs Administration

**December 29, 1994—59 FR 67390**  
**Implementation of the United Nations Recommendations, International Maritime Dangerous Goods Code, and International Civil Aviation Organization's Technical Instructions for the Safe Transport of Dangerous Goods by Air**

##### Action: Final rule

The U.S. DOT's Research and Special Programs Administration (RSPA) has issued a final rule amending the Hazardous Materials Regulations to maintain alignment with corresponding provisions of international standards.

Because of recent changes to the International Maritime Dangerous Goods Code, the International Civil Aviation Organization's Technical Instructions for the Safe Transport of Dangerous Goods by Air, and the United Nations Recommendations on the Transport of Dangerous Goods (U.N. Recommendations), RSPA determined that these revisions are necessary to facilitate the transport of hazardous materials in international commerce.

During the proposed rule stage of this rulemaking, one of the many commenters suggested that RSPA provide a nonbulk packaging exception for "resin solution" comparable to the exception provided for paint under section 173.173 of the HMR. According to the commenter, an exception from the performance packaging requirements provided for certain resin solutions under the U.N. Recommendations and the IMDG code. The commenter also stated that U.S. companies need the exception for resin solutions to compete in the international marketplace. RSPA agreed with these suggestions and is adding "resin solution" to the Section 172.101 table.

RSPA is also amending the section heading and the introductory text of paragraph (b) of Section 173.173 to include resins.

The effective date of this rule is October 1, 1995. Compliance with the regulations, as amended herein, is authorized as of January 1, 1995.

For further information, contact Bob Richard, Assistant International Standards Coordinator, (202) 366-0586, Beth Romo or John Gale, Office of Hazardous Materials Standards, RSPA, U.S. DOT, Washington, D.C.; (202) 366-8553.

Proposed rules under the following statutes have upcoming comment deadlines:

#### Clean Air Act

**National Emission Standards for Hazardous Air Pollutants from Shipbuilding and Ship Repair (59 FR 62681)**—Comment deadline: February 6, 1995. Contact Mohamed Serageldin, Coatings and Consumer Products Group, Emission Standards Divisions (MD-13), U.S. EPA, Research Triangle Park, NC 27711; (919) 541-2379.

**National Emission Standards for Hazardous Air Pollutants from Wood Treatment (59 FR 62652)**—Comment deadline: February 21, 1995. Contact Madeleine Strum, Coatings and Consumer Products Group, Emission Standards Division (MD-13), U.S. EPA, Research Triangle Park, NC 27711; (919) 541-2383.

#### Toxic Substances Control Act

**Disposal of Polychlorinated Biphenyls (59 FR 62788)**—Comment deadline: April 6, 1995. Contact Susan B. Hazen, Director, Environmental Assistance Division (7408), Office of Pollution Prevention and Toxics, EPA Room E-543B, 401 M St., S.W., Washington, D.C. 20460; (202) 554-1404.

## States Proposed Legislation and Regulations

### Alabama

**Air Quality (Regulation)**—A proposed rule of the Alabama Department of Environmental Management (DEM) would ensure consistency with federal and state requirements relating to air pollution sources subject to new source performance standards. Contact Hearing Officer, Office of General Counsel, ADEM, (334) 271-7700.

**Hazardous Waste (Regulation)**—The Alabama DEM issued a final rule which incorporates changes made to federal EPA hazardous waste management regulations, including revisions to land disposal restrictions, and the identification and listing of various hazardous wastes. Contact Steven Jenkins, DEM, (205) 271-7726.

### Arizona

**Air Quality (Regulation)**—The Arizona Department of Environmental Quality (DEQ) announced a proposal to establish procedures for the creation of federally enforceable emission limitations that can be used by an air pollution source, enabling it to avoid classification under federal regulations as a source larger than it actually will be. Contact Martha Seaman, DEQ, (602) 207-2222.

The Arizona DEQ issued a proposal that would allow the DEQ to set emission limitations by permit that are equivalent to the National Emissions Standards for Hazardous Air Pollutants (NESHAP) in the event that the U.S. Environment Protection Agency (EPA) has not established the relevant emission limitation. The proposal also establishes requirements for permit

applications for owners and operators of major sources of hazardous air pollutants and adds provisions for the establishment of maximum achievable control technologies (MACT) requirements. Contact Martha L. Seaman, DEQ, (602) 207-2222.

### Arkansas

**Air Quality (Regulation)**—The Arkansas Department of Pollution Control and Ecology (DPCE) issued a final rule which reduced the initial operating permit fee for existing sources by the amount of their previous annual permit fee and establishes a \$1,000 minimum initial operating permit fee for sources holding an active air permit. The rule also eliminates the annual permit fee for sources emitting less than 10 tons per year of air contaminants and establishes a maximum modification fee for all permits. Contact Doug Szenher, DPCE, (501) 564-7444.

### California

**Air Quality (Regulation)**—The California Air Resources Board (CARB) adopted a final rule which requires local air pollution control and air quality management districts to collect permit fees from major nonvehicular sources of non-attainment pollutants. The rule went into effect on December 28. Contact Don Rake, CARB, (916) 322-7304.

The California CARB adopted an amendment to regulations establishing designation criteria and specifying areas of California as nonattainment, attainment, or unclassified for all pollutants for which state ambient air quality standards are set forth. The

rule went into effect on December 10, 1994. Contact Rich Bradley, CARB, (916) 322-6076.

**Hazardous Waste (Regulation)**—The California Department of Toxic Substances Control (DTSC) issued an emergency rule which grants exemptions from permit requirements for the disposal of wastes based on the percentage of hazardous materials present at the time of disposal. Contact Pamela Thompson, DTSC, (916) 324-9933.

The California DTSC has proposed a rule which would adopt Federal EPA land disposal restrictions standards for the definition of "hazardous waste" and the universal treatment standards for most RCRA hazardous wastes. Contact Pamela Thompson, DTSC, (916) 324-9933.

**Packaging (Regulation)**—The California Integrated Waste Management Board (IWMB) adopted an amendment, effective December 4, which details the responsibilities of product, container, and resin manufacturers to facilitate compliance with the rigid plastic container statutes. The rule would minimize the amount of additional recordkeeping and fiscal resources devoted to documenting compliance. Contact Janice Welch, IWMB, (916) 255-2410.

**Toxic Substances (Regulation)**—The California Environmental Protection Agency (Cal EPA) issued a proposed rule pertaining to methods, policies, and guidelines used in the identification and assessment of chemical toxicity. Contact Thomas McDonald, Cal EPA, (510) 540-2084.



## Colorado

*Air Quality (Regulation)*—A rule adopted by the Colorado Air Quality Control Commission (AQCC) clarifies the exemptions for volatile organic compound (VOC) emissions from surface coating limitations for small job shop operations. The rule became effective on December 30. Contact AQCC, (303) 692-3180.

The Colorado AQCC adopted a final regulation which allows for certified emission reduction credits to be used as an alternative compliance method on a case-by-case basis. The rule went into effect on December 30. Contact Sara Laumann, AQCC, (303) 692-3180.

The Colorado AQCC issued a proposal which would allow the use of emission credit reductions to be used to satisfy a control technique guideline (CTG) requirement limiting VOC emissions from surface coating operations. A hearing will be held on February 16. Contact Joseph Palomba, Jr., AQCC, (303) 692-2000.

The Colorado AQCC announced that it is considering adding acetone, cyclohexane, malathion, tetrahydrofuran, and 1,2-dichlorobenzene to the list of hazardous air pollutants (HAPs) for the state. Contact AQCC, (303) 692-2000.

The Colorado AQCC issued a proposal that provides an exemption for any facility or equipment constructed, reconstructed, or modified after August 30, 1989, that is designed to burn less than 10 metric tons per day of municipal solid waste. The rule removes the continuous monitoring requirement and the recordkeeping and reporting requirements for municipal incinerators meeting the requirements. Contact Joseph Palomba, Jr., AQCC, (303) 692-2000.

A regulation adopted by the Colorado Department of Health (DOH) requires the submittal of state implementation plans (SIPs) for particulate matter of an aerodynamic diameter of 10 microns or less (PM<sub>10</sub>) to the U.S. EPA. The rule specifies that areas which are in nonattainment for PM<sub>10</sub> and which must show attainment and maintenance of the National Ambient Air Quality Standard for PM<sub>10</sub>. The new rule went into effect on January 10, 1995. Contact DOH, (303) 692-2000.

*Hazardous Waste (Regulation)*—A final rule of the Colorado Hazardous Waste Commission (HWC) amends current hazardous waste management regulations to incorporate Federal EPA amendments to the wood

preserving standards. The rule was effective on October 30, 1994. Contact Karen Osthus, HWC, (303) 692-3321.

*Water Quality (Regulation)*—A rule adopted by the Colorado Water Quality Control Commission (WQCC) defines what conditions can be required by the WQCC in connection with certification of federal licenses and permits under Section 401 of the Federal Clean Water Act. The regulation became effective as of December 30. Contact WQCC, (303) 692-3520.

## Connecticut

*Air Quality (Regulation)*—The Connecticut Department of Environmental Protection (DEP) adopted a final regulation to implement the requirements of Title V of the Federal Clean Air Act Amendments of 1990. The rule establishes that fees are based on emissions of nitrogen oxides, volatile organic compounds (VOCs), and any pollutants for which an ambient air quality standard has been listed or any pollutant subject to Section 111, 112, or Title VI. The rule also sets procedures for determining and paying the fees. The rule became effective on September 30, 1994. Contact DEP, Bureau of Air Management, (203) 566-2506.

*Lead*—CT H. 5016 (Godfrey) extends state financial assistance for lead-based paint abatement. The bill was introduced on January 4, 1995, and referred to the Joint Select Committee on Housing.

*Lead (Regulation)*—A proposed regulation of the Connecticut Department of Public Health (DPH) would provide for licensing and certification of lead abatement contractors, consultants, and workers. Contact DPH, (203) 240-9200.

## Delaware

*Air Quality (Regulation)*—The Delaware Department of Natural Resources and Environmental Control (DNREC) issued a final regulation, effective December 29, 1994, which requires statewide compliance with emissions standards for aerospace coatings, motor vehicle refinishing, surface coatings of plastic parts, control of VOC emissions from volatile organic liquid storage vessels, batch processing operations, industrial cleaning solvents, offset lithographic printing, reactor processes, and distillation operations. Contact Lee Randolph, DNREC, (302) 323-4542.

*Solid Waste (Regulation)*—The Delaware DNREC proposed a regulation that would require the certification of contractors working with underground storage tanks. Contact DNREC, (302) 323-4588.

## Florida

*Air Quality (Regulation)*—The Florida Department of Environmental Protection (DEP) proposed amendments to current regulations regarding air emissions monitoring for stationary sources. The changes will modify testing and emissions monitoring requirements. Contact Michael Hewett, DEP, (904) 488-0114.

The Florida DEP revised general requirements for stationary sources of air pollutant emissions by adding lists of hazardous air pollutants and procedures for obtaining federally enforceable non-Title V operating permits. Contact Michael Hewett, DEP, (904) 488-0114.

*Recycling (Regulation)*—A proposed rule of the Florida DEP requires the certification of persons who handle, purchase, receive, recover, sell, or are end-users of recovered materials. Contact John Lowry, DEP, (904) 488-0300.

*Solid Waste (Regulation)*—The Florida DEP announced changes to a proposed rule that would implement a noncompliance fee for violations of underground and aboveground storage tank requirements. Contact Marshall Mott-Smith, DEP, (904) 488-3935.

## Georgia

*Air Quality (Regulation)*—A rule adopted by the Georgia Department of Natural Resources (DNR) details requirements for facilities from which air contaminants are or may be emitted. The provisions, which became effective on November 17, 1994, include emission limitations and standards, sampling requirements, ambient air standards, and open burning and source monitoring. The regulation also specifies the requirements for construction permits; operating permits; the revocation, suspension, modification or amendment of permits; nontransferable permits; combined permits and applications; permit fees; and Title V operating permits. Contact DNR, (404) 656-4713.

*Water Quality (Regulation)*—The Georgia DNR adopted a final rule updating water quality control standards, including revising provi-

sions for waste water treatment and general permits, establishing storm water permitting requirements, and adopting requirements for sewage sludge. Contact Sam Shepard, DNR, (404) 656-4708.

### Idaho

*Air Quality (Regulation)*—A proposed rule of the Idaho Department of Health and Welfare (DHW) would loosen the requirements for reporting deadlines concerning air pollution. Contact Sue Richards, DHW, (208) 334-5898.

### Illinois

*Air Quality (Regulation)*—The Illinois Pollution Control Board (PCB) proposed a regulation which would establish VOC limitations for fountain solutions used in specific lithographic printing, including heatset web offset, nonheatset web offset, and sheet fed. The rule addresses organic emissions from storage and loading operations, organic emissions from miscellaneous equipment, solvent cleaning, coating operations, use of organic material, and printing and publishing. Contact Dorothy Gunn, IPCB, (312) 814-6929.

The Illinois PCB has proposed a rule that would regulate VOC emissions from batch operations in the Chicago and Metro East (St. Louis) areas. Contact Audrey Lozuck-Lawless, IPCB, (312) 814-6923.

*Automotive Refinishing (Regulation)*—The Illinois PCB has issued proposed rules which would amend regulations regarding motor vehicle refinishing. The proposed amendments regulate VOCs from refinishing operations in specific areas, provide equipment specifications, describe work practices, and implement testing, monitoring, registration, reporting, and recordkeeping requirements for those operations. Contact Audrey Lawless, PCB, (312) 814-6923.

*Hazardous Waste (Regulation)*—The Illinois PCB has issued a final rule that incorporates Federal EPA regulations regarding general hazardous waste management provisions, including an update of test methods under RCRA. The rule also incorporates EPA's decision not to regulate wastes from wood surface protection as listed hazardous wastes and addresses other requirements for owners and operators of hazardous

waste treatment, storage, and disposal facilities. Contact Michael McCambridge, PCB, (312) 814-6924.

### Indiana

*Air Quality (Regulation)*—The Indiana Department of Environmental Management (DEM) has announced a hearing on proposed revisions to regulations limiting emissions of VOCs from wood furniture coating operations and liquid storage tanks in Clark, Floyd, Lake, and Porter counties. The hearings are scheduled for March 1, 1995 in Indianapolis. Contact Patricia Toth, DEM, (317) 233-5681.

A proposed regulation of the Indiana DEM would amend the definitions of "nonphotochemically reactive hydrocarbon" and "volatile organic compound." Contact Kiran Verma, DEM, (317) 233-5678.

The Indiana DEM has proposed a rule to amend standards for hazardous air pollutants by incorporating federal NESHAP standards to change the framework for achieving federal air toxic requirements. Contact DEM, (317) 232-5586.

*Hazardous Waste (Regulation)*—A proposal of the Indiana DEM would amend procedures for financial requirements for owners and operators of hazardous waste, treatment, storage, and disposal facilities. Contact Jeff Stevens, DEM, (317) 232-8901.

*Water Quality (Regulation)*—The Indiana Water Pollution Control Board (WPCB) proposed a rule to clarify the procedure for obtaining a permit variance from a water quality standard. Contact WPCB, (317) 232-8476.

### Iowa

*Air Quality (Regulation)*—The Iowa Environmental Protection Commission (EPC) proposed amendments to the list of requirements for obtaining air construction permits and operating permits. Contact Christine Spackman, Department of Natural Resources, (515) 281-8941.

The Iowa EPC adopted a regulation which establishes a voluntary operating permit program and provides an alternative to the Title V operating permit program for small sources able to qualify under these rules. The rule went into effect on December 14, 1994. Contact Christine Spackman, Department of Natural Resources, (515) 281-8941.

### Kansas

*Air Quality (Regulation)*—The Kansas Department of Health and Environment (DHE) adopted a final rule, effective January 23, 1995, establishing an operating permit program required by the Federal Clean Air Act. The rule increases emission fees imposed on air pollution sources, revises the construction permit program, and implements new source performance standards, national emission standards for hazardous air pollutants, and maximum achievable control technology (MACT) for hazardous air pollutants. Contact Chuck Layman, DHE, (913) 926-1579.

*Transportation (Regulation)*—The Kansas State Corporation Commission (SCC) issued a final rule that adopts federal motor carrier safety and hazardous materials transportation requirements. Contact Judith McConnell, SCC, (913) 271-3159.

### Kentucky

*Air Quality (Regulation)*—A proposed rule of the Kentucky Department of Environmental Protection (DEP) would modify the definition of VOCs to make it consistent with the federal definition. Contact John Hornback, DEP, (502) 573-3382.

The Kentucky DEP has proposed a rule which would establish standards of performance for emissions that are at least as stringent as current federal standards. Operations affected include (1) automobile/light-duty truck surface coatings; (2) pressure sensitive tape/label surface coating; (3) large appliance industrial surface coatings; (4) metal coil surface coatings; (5) beverage can surface coatings; and (6) flexible vinyl/urethane coatings. Contact John Hornback, DEP, (502) 573-3382.

The Kentucky DEP issued a proposal which would incorporate, by reference, federal MACT standard compliance extension for sources of hazardous air pollutants that comply with voluntary provisions. Contact John Hornback, DEP, (502) 573-3382.

### Louisiana

*Air Quality (Regulation)*—A proposed regulation of the Louisiana Department of Environmental Quality (DEQ) defines architectural and industrial maintenance (AIM) coatings and sets VOC limits for these



coatings. Contact Patsy Deaville, DEQ, (504) 765-0399.

The Louisiana DEQ issued a correction to a final rule adopted on October 10, 1994 governing fugitive emissions of VOCs in the Baton Rouge ozone nonattainment area. The correction specifies a January 1, 1996 compliance date for monitoring requirements affecting existing equipment. Contact David Hughes, DEQ, (504) 765-0399.

The Louisiana DEQ has proposed a regulation which revises increments for PM10 and restricts increases in ambient air concentrations of PM10 to established levels. Contact Patsy Deaville, DEQ, (504) 765-0399.

A proposed rule of the Louisiana DEQ would amend air quality regulations to bring state standards of performance for new stationary sources into compliance with federal requirements. Contact Patsy Deaville, DEQ, (504) 765-0399.

*Lead (Regulation)*—A proposed regulation of the Louisiana DEQ would, among other things, establish licensure and certification provisions for lead hazard reduction personnel. Contact Patsy Deaville, DEQ, (504) 765-0399.

### Maine

*Air Quality (Regulation)*—The Maine Department of Environmental Protection (DEP) proposed revisions to the state implementation plan for ozone, including, among other things, a calculation of the 15% reduction in VOC emissions needed by 1996. Contact Donald Anderson, DEP, (207) 287-2437.

### Maryland

*Air Quality (Regulation)*—A proposed regulation of the Maryland Department of Environment (DOE) would establish requirements for control equipment at organic chemical manufacturing operations that, among other things, would require that VOC emission sources operate only when their control device is functioning properly. Contact Deanna Miles-Brown, (DOE), (410) 631-3173.

The Maryland DOE has proposed rules which would establish reasonably available control technology (RACT) standards for organic chemical manufacturing operations. Contact Deanna Miles-Brown, DOE, (410) 631-3173.

A proposed rule of the Maryland DOE would amend regulations to expand the RACT requirement to include point sources in series

nonattainment areas with VOC emissions of 25 tons or more per year. The proposal also specifies compliance dates for such sources to notify the department of their status. Contact Deanna Miles-Brown, DOE, (401) 631-3173.

*Automotive Refinishing (Regulation)*—The Maryland DOE has proposed a rule setting forth reasonable control measures for vehicle refinishing to include the use of lower VOC content coatings, improved application methods, and improved cleanup methods. Contact Deanna Miles-Brown, DOE, (410) 631-3173.

*Lead (Regulation)*—The Maryland DOE proposed a rule that would establish standards and procedures for the accreditation of lead abatement inspectors, contractors, supervisors, and workers. Contact Deanna Miles-Brown, DOE, (410) 631-3173.

The Maryland DOE issued a proposed rule that would establish requirements for the maintenance and inspection of residential rental properties where lead paint and lead dust is likely to be present. Contact Deanna Miles-Brown, DOE, (410) 631-3173.

*Transportation (Regulation)*—A final rule of the Maryland Public Service Commission (PSC) provides that an owner or operator of a motor vehicle engaged in bulk transport of flammable or combustible liquids who fails to permit inspection of the vehicle or records relating to a permit is subject to civil penalty. The rule went into effect December 19, 1994. Contact Donald Eveleth, PSC, (410) 767-8064.

### Massachusetts

*Air Quality (Regulation)*—The Massachusetts Department of Environmental Protection (DEP) issued a final rule setting VOC content limits for AIM coatings. The rule went into effect November 18, 1994. Contact Paul Reilly, DEP, (617) 556-1097.

The Massachusetts DEP has issued a proposed rule that would revise the state implementation plan for ozone, including the identification of emission reduction measures to reduce VOC emissions by at least 15% between 1990 and 1996. Contact Leah Weiss, DEP, (617) 292-5520.

*Labeling (Regulation)*—The Massachusetts DEP issued a final rule, effective December 16, 1994, that specifies requirements for the labeling of automotive refinishing coating

containers and the dissemination of mixing instructions. Contact Paul Reilly, DEP, (617) 556-1097.

*Lead (Regulation)*—The Massachusetts Department of Health (DPH) is proposing a rule which would amend regulations regarding blood lead screening and residential lead inspection and abatement. Contact Roy Petre, DPH, (617) 522-3700.

*Solid Waste (Regulation)*—A final rule of the Massachusetts Board of Fire Prevention (BFP) amends provisions affecting aboveground and underground storage tanks. The changes apply to design, construction, installation, testing, and maintenance of tanks and containers. Contact Dana Bowers, BFP, (617) 351-6055.

*Toxic Substances (Regulation)*—The Administrative Council of Toxic Use Reduction (ACTUR) adopted a final rule that revises procedures for adding new toxic or hazardous substances for reporting purposes under the Toxic Use Reduction Act. Contact Regina McCarthy, ACTUR, (617) 727-9800.

### Montana

*Air Quality (Regulation)*—The Montana Department of Health and Environmental Sciences (DHES) finalized a rule to increase air quality operation and permit application fees, effective December 23, 1994. Contact Yolanda Fitzsimmons, DHES, (406) 444-2544.

A final rule of the Montana DHES amends current regulations in order to keep state air quality standards at least as stringent as federal requirements, to retain state control over the air quality program, and to implement revised requirements for sampling and data collection. The rule became effective on October 28, 1994. Contact Yolanda Fitzsimmons, DHES, (406) 444-2544.

### Nebraska

*Air Quality (Regulation)*—The Nebraska Department of Environmental Quality (DEQ) proposed a rule which allows the DEQ to approve alternate methods for establishing annual emission rates and to set criteria for the development of insignificant activities for emission reporting requirements. Contact Thomas R. Lamberson, DEQ, (402) 471-2186.

### Nevada

*Hazardous Waste (Regulation)*—The Nevada Department of Conservation and Natural Resources

(DCNR) issued a final rule that applies to any person who generates, transports, treats, stores, disposes, or otherwise manages hazardous waste and which incorporates current federal hazardous waste regulations. Contact DCNR, (702) 687-5872.

### New Hampshire

**Air Quality (Regulation)**—A regulation proposed by the New Hampshire Department of Environmental Services (DES) would amend regulations regarding standards for sources required to obtain Federal Clean Air Act Title V and state operating permits. The proposed rule would establish permitting requirements, permit application procedures, and relevant fees. Contact Craig Wright, DES, (603) 271-6791.

The New Hampshire DES has issued a proposed rule which would make the state operating permit program federally enforceable. Contact Craig Wright, DES, (603) 271-6791.

### New Jersey

**Air Quality (Regulation)**—The New Jersey Department of Environmental Protection (DEP) proposed a rule regarding the control of VOC emissions. Contact Janis Hoagland, DEP, (609) 292-0716.

The New Jersey DEP is proposing regulations that would establish affirmative defense procedures for individuals who have violated an issued air permit. The proposal relates to violations that occur because of equipment malfunction, start-up, or shut-down or during the performance of necessary equipment maintenance. Contact Janis Hoagland, DEP, (609) 292-0716.

**Graffiti**—NJ A. 765 (Catania and DeCrocce) revised the law concerning graffiti by requiring that anyone convicted of an act of graffiti provide restitution for the damage. On December 5, the Assembly approved the legislation; it was then sent to the Senate.

NJ A. 2343 (Pascrel and Solomon) authorizes courts to order community service for a juvenile and parent if the juvenile commits an act of graffiti. Introduced on November 21, the bill was referred to the Assembly Committee on Judiciary, Law and Public Safety.

NJ S. 1374 (Girgenti) requires the suspension of a driver's license and community service for acts of graffiti. On December 1, the bill was released

with amendments from the Senate Committee on Law and Public Safety.

**Toxic Substances (Regulation)**—A final rule of the New Jersey Department of Health (DOH) establishes the program-implementing requirements of the Worker and Community Right-to-Know Act. The readoption of the rule became effective on September 28, 1994 and the amendments became effective on November 7, 1994. The rule expires on September 28, 1999. Contact Richard Willinger, DOH, (609) 984-2202.

The New Jersey DEP has issued a final rule amending state community right-to-know requirements by increasing the reporting threshold from 100 pounds to 500 pounds. In addition, it adds substances that are federally regulated for accidental release prevention to the list subject to reporting requirements. The rule went into effect November 21, 1994, and expires June 16, 1999. Contact Janis Hoagland, DEP, (609) 292-0716.

### New Mexico

**Air Quality (Regulation)**—The New Mexico Environmental Improvement Board (EIB) issued a final rule that requires sources of air pollutant emissions to comply with enhanced monitoring standards. The rule went into effect December 10, 1994. Contact Phyllis Ludi, EIB, (505) 827-2971.

The New Mexico EIB adopted a final rule that requires a permit for construction or modification of major sources of hazardous air pollutants listed under the Federal Clean Air Act amendments in excess of 10 tons per year of any one pollutant or 25 tons per year of any combination of pollutants. The rule was effective December 14, 1994. Contact Jim Nellesen, EIB, (505) 827-2971.

### New York

**Lead**—NY S. 57 (Padavan) increases the penalty for the failure to comply with a notice and demand for the discontinuance of a paint condition conducive to lead poisoning from \$2,500 to \$5,000. Introduced on January 4, 1995, the bill was referred to the Senate Committee on Health.

**Hazardous Waste (Regulation)**—The New York Department of Environmental Conservation (DEC) adopted a rule which provides for the administration and enforcement of the DEC's hazardous waste management in lieu of equivalent federal regulations, including the identifica-

tion and listing of hazardous waste, land disposal restrictions, financial assurance and liability requirements, and new household hazardous waste regulations. The rule went into effect on January 16, 1995. Contact Margaret O'Neil, DHEC, (518) 485-8988.

### North Carolina

**Air Quality (Regulation)**—A proposal issued by the North Carolina Department of Environment, Health and Natural Resources (DEHNR) would adopt RACT standards for nitrogen oxides for ozone nonattainment areas and define potential emissions for various type sources. Contact Thomas C. Allen, DEHNR, (919) 733-1489.

### Ohio

**Air Quality (Regulation)**—The Ohio Environmental Protection Agency issued a final rule regarding *de minimis* air contaminant source exemptions, permits to operate, and federally enforceable limits on potential to emit. Contact Tami Saunders, Ohio EPA, (614) 644-2270.

**Lead (Regulation)**—The Ohio Department of Health (DOH) issued a proposal relating to the state lead program which includes, among other things, lead abatement activity specifications, general application procedures, recordkeeping and reporting requirements, public health lead inspection procedures, levels of lead hazardous to human health, and environmental sampling techniques. Contact Gay Lynn Rice or Jackie Vermillion, (614) 466-4882.

### Oklahoma

**Transportation (Regulation)**—A proposed regulation of the Oklahoma Department of Public Safety (DPS) would provide for the adoption, by reference, of federal regulations pertaining to motor carrier safety and hazardous materials transportation. Contact John Hardridge, DPS, (405) 425-6103.

### Oregon

**Air Quality (Regulation)**—A proposed rule of the Oregon Department of Environmental Quality (DEQ) would revise the particulate matter increments by replacing total suspended particulates with PM10 in order to comply with federal prevention of significant deterioration rules. Contact Chris Rich, DEQ, (503) 229-6775.



**Packaging (Regulation)**—The Oregon Department of Environmental Quality (DEQ) adopted regulations which require product manufacturers who use rigid plastic containers and container manufacturers that produce such containers to comply with recycling, recycled use, or reuse standards. The rule went into effect January 1, 1995. Contact Chris Rich, DEQ, (503) 229-6775.

### Pennsylvania

**Air Quality (Regulation)**—The final rule of the Pennsylvania Department of Environmental Resources (DER) implements permit requirements for major sources under the state Air Pollution Control Act and Title V of the Federal Clean Air Act. Contact Gary Triplett, DER, (717) 787-9702,

### Tennessee

**Air Quality (Regulation)**—The Tennessee Department of Environment and Conservation (DEC) proposed a regulation which would establish emission standards and control technology determinations for hazardous air pollutants for source categories. Contact Ted Wilkinson, DEC, (615) 532-6819.

### Texas

**Air Quality (Regulation)**—The Texas Natural Resources Conservation Commission (NRCC) issued a final rule that gives companies constructing new facilities or modifying old ones a more flexible type of permit option for compliance with air emissions standards. Contact Mary Ruth Holder, TNRCC, (512) 239-1966 or Gary McArthur (512) 239-1917.

**Hazardous Waste (Regulation)**—A proposed regulation of the Texas NRCC would eliminate notification and manifesting requirements for industrial generators that generate less than 100 kilograms per month of hazardous waste and/or 100 kilograms of nonhazardous waste. Contact Hygie Reynolds, NRCC, (512) 239-6087.

The Texas NRCC proposed a rule which would update general provisions for industrial solid waste and municipal hazardous waste management. The rule would also revise standards applicable to generators of hazardous waste and change provisions for industrial and solid waste classification. Contact Grace Faulkner, NRCC, (512) 239-6087.

### Utah

**Air Quality (Regulation)**—The Utah Department of Environmental Quality (DEQ) proposed a rule that would incorporate enhanced monitoring requirements in the state implementation plan. Contact Jan Miller, DEQ, (801) 536-4042.

The Utah DEQ proposed a regulation to add seven insignificant emission activities to a list of activities which are excluded from the permitting application process. Contact Jan Miller, DEQ, (801) 536-4042.

**Solid Waste (Regulation)**—The Utah DEQ issued a proposed rule to clarify situations under which certificates of compliance for underground storage tanks lapse or may be revoked. The proposal would provide for the revocation of a certification of compliance if a tank owner or operator does not satisfy all leaking underground storage tank cleanup requirements. Contact Gary Astin, DEQ, (801) 536-4100.

**Toxic Substances (Regulation)**—A Utah DEQ proposed rule revises regulations to incorporate federal amendments concerning new test methods associated with pentachlorophenol in wood surface protection. Contact Susan Toronto, DEQ, (801) 538-6170.

### Vermont

**Air Quality (Regulation)**—The Vermont Agency of Natural Resources (ANR) has issued a proposed rule that would require air contaminant sources with the potential to generate 10 tons of air pollution annually to apply for and obtain an operating permit. The proposal also specifies that certain source categories would be required to use RACT or other pollution prevention techniques to limit the discharge of air contaminants. Contact Richard Valentineti, ANR, (802) 241-3860.

### Virginia

**Solid Waste (Regulation)**—The Virginia Department of Conservation and Recreation (DCR) has proposed to update existing minimum technical criteria for storm water management programs to reflect current engineering methods. Contact David Nunnally, DCR, (804) 786-3998.

### Washington

**Air Quality (Regulation)**—The Spokane County Air Pollution

Control Authority (SCAPCA) issued a final rule to limit particulate and toxic air pollutant emissions from surface coating operations. The rule establishes requirements for techniques with respect to surface coating applications and the use of spray booths and clean-up materials, adopts disposal procedures, places limitations on certain toxic substances and solvent content of auto refinishing, and establishes recordkeeping procedures. The rule went into effect December 10, 1994. Contact Eric Skelton, SCAPCA, (509) 456-4727.

### West Virginia

**Transportation (Regulation)**—The West Virginia Public Service Commission (PSC) permanently adopted an emergency amendment relating to uniform registration and permitting program for motor carriers transporting hazardous materials. Contact Howard M. Cunningham, PSC, (304) 340-0426.

### Wisconsin

**Air Quality (Regulation)**—A regulation proposed by the Wisconsin Department of Natural Resources (DNR) would regulate VOCs by reducing emissions from architectural and industrial maintenance (AIM) coatings. The rule affects suppliers, sellers, and end users in the moderate or worse ozone nonattainment, counties of Kenosha, Kewaunee, Manitowoc, Milwaukee, Ozaukee, Racine, Sheboygan, Washington, and Waukesha, Contact Josie Pradella, DNR, (608) 266-0815

A proposed rule of the Wisconsin DNR relates to VOC emissions from lithographic printing. The regulation identifies the three types of offset lithographic presses that will be regulated and establishes emission limits for web lithographic presses in the nine county ozone nonattainment areas. Contact Jim Crawford, DNR, (608) 266-1054.

**Automotive Refinishing (Regulation)**—The Wisconsin DNR proposed a rule which would establish controls for VOC emissions from autobody refinishing. The regulation applies to autobody and repair shops, production paint shops, new and used car dealer repair and paint shops, and fleet operator repair and paint shops in the nine southwestern counties which are in the moderate or worse ozone nonattainment area. Contact Ron Dillahunt, DNR, (608) 266-1054.

## Kent State University Releases Spring Schedule for Rheology, Dispersion, and Adhesion Courses

The Professional Development Institute in the Chemistry Department of Kent State University, Kent, OH, will conduct three short courses this spring. The courses are: "Applied Rheology for Industrial Chemists," April 24-28; "Dispersion of Pigments and Resins in Fluid Media," May 8-12; and "Adhesion Principles and Practice for Coatings and Polymer Scientists," May 22-26.



### "APPLIED RHEOLOGY FOR INDUSTRIAL CHEMISTS"

Rheology fundamentals will be introduced followed by instrument selection and their use for meaningful measurements. Lecturers will discuss processing and performance problems. The relevance of rheology to application, flowout, and defects formation of liquid systems and its use in the coatings industry is presented. The following will also be addressed: flow modification, thermal mechanical properties analysis during cure, and the rheology of latexes and thermosets.

#### Monday, April 24

"Rheological Measurements: Controlled Stress or Controlled Strain, Which to Use?"—Abel Gaspar-Roas, Paar Physica USA; "Rheometry"—Irvin M. Krieger, Case Western Reserve University; "Fundamentals of Linear Viscoelasticity"—I.M. Krieger; and "Rheology of Dispersion"—I.M. Krieger.

#### Tuesday, April 25

"Rheological Instruments and Selection"—Edward A. Collins, E.A. Collins, Inc.; "Meaningful Rheological Measurements"—Edward A. Collins; "Application of Rheology to Processing Problems"—Charles L. Rohn, Bohlin Instruments, Inc.; and "Application of Rheology to End-Use Performance Problems"—Charles L. Rohn.

#### Wednesday, April 26

"Thermal Mechanical Properties of Coatings"—Daniel J. Skrovanek, Miles, Inc.; "Rheology in the Protective and Decorative Coatings Industry, Part I"—Richard R. Eley, The Glidden Co.; "Rheology in the Protective and Decorative Coatings Industry, Part II"—Richard R. Eley; and "Rheology of Liquid Coatings with Emphasis on Defect Formation"—Clifford K. Schoff, PPG Industries, Inc.

#### Thursday, April 27

"Control and Rheological Measurement of Crosslink Density in Films"—Loren W. Hill, Monsanto Chemical Co.; "Rheological Additives for Flow Modification"—Sharad Thakkar, Scitex Digital Printing,

Inc.; "Application of Rheology to Engineering Problems"—Donald Bigg, R.G. Barry Corp.; and "Chemorheology of Thermosetting Coatings"—Richard R. Eley.

#### Friday, April 28

"Cure Characterization for Product Research and Development, Part I"—Theodore Provder, The Glidden Co.; and "Cure Characterization for Product Research and Development, Part II"—Theodore Provder.



### "DISPERSION OF PIGMENTS AND RESINS IN FLUID MEDIA"

Relevant surface chemistry fundamentals will be presented in the dispersion course,

followed by lectures on dispersing agent selection; dispersion of inorganic, organic, silica and carbon black pigments, and the evaluation of dispersion quality by rheological means. Experts will discuss dispersion of resins and latexes in water and organic solvent systems. Discussion of dispersion equipment and selection of mixers; ball, sand, and other media mills; high-viscosity dispersion equipment; and particle size analysis for evaluating processing results will be included.

#### Monday, May 8

"Fundamentals of Surface and Colloid Chemistry I"—Richard J. Ruch, Kent State University; "Dispersant Selection Based on Pigment Functionality"—Robert F. Conley,

(continued on next page.)

## Piedmont Society's Show and Symposium Focuses on Technology and Regulatory Issues

The Piedmont Society for Coatings Technology will again be holding their Mini-Trade Show and Symposium, concentrating on clean air and technology, on March 15, 1995, at Showcase in the Park, Corner of Elm and Commerce, High Point, NC.

The Show and Symposium will include some 50 or 60 tabletop displays of raw material suppliers' product lines. An on-site catered buffet luncheon, sponsored by the Piedmont Paint and Coatings Association, will feature a speaker from Reichhold.

Presentations are scheduled on "Reactive Additives and Their Application in Radiation Curing Systems," from Tego Chemie; "Replacing HAPS Solvents in Conventional Systems," from Eastman; "Benefits of RC Technology for Wood Coatings," from SC Johnson; and "Designing Polymers to Meet Emerging Market Needs—Glycol Ether Free Graphic Arts Polymers," from BFGoodrich.

This forum is intended for all coatings manufacturers and suppliers, as well as anyone interested

or involved in the coatings industry in general. About 200 attendees are expected.

For further information, please contact Nellie Moretz, Guardsman Products, Inc., 2147 Brevard Rd., High Point, NC, 27263; (910) 889-6344.

### "Process Safety"

Sponsored by  
The Chicago Society For Coatings  
Technology

March 8, 1995  
Oak Brook Marriott Hotel  
Oak Brook, IL

Topics will include:

- Water/Solvent Waste Classification and handling
- Regulatory affairs OSHA, EPA, third party viewpoint
- Worker Safety Equipment operation and static electricity

Contact Stephen Brauer,  
Mozel, Inc., 1110 Lake Cook Rd.,  
Ste. 250, Buffalo Grove, IL 60089.  
(708) 285-0065



# Spring Schedule Announced by Kent State University

(Continued from previous page.)

Mineral and Resource Technology; "Fundamentals of Surface and Colloid Chemistry II"—Richard J. Ruch; and "Inorganic Pigment Processing Parameters and Their Influence on Polymer Properties"—Robert F. Conley.

## Tuesday, May 9

"White Pigments—Processing and Rheology"—Robert F. Conley; "Dispersion of Organic Pigments"—Theodore Vernardakis, Sun Chemical Corp.; "Dispersion of Fumed Silica and Carbon Black for Optimum Performance"—Maria Nargiello, Degussa Corp.; and "Wetting and Dispersing in Aqueous and Nonaqueous Coatings Systems"—Robert W. Vash, Iron Chemicals, Inc.

## Wednesday, May 10

"Introduction to Dispersion Rheology"—Richard R. Eley; "Determining Dispersion Quality by Rheological and Related Methods"—Richard R. Eley; "Overview of Dispersion Equipment for Inks and Coatings"—Ramon Pineiro, Daniel Products Co.; and "Optimizing Your Dispersion Process with Premilling"—David Ulrich, Kady International.

## Thursday, May 11

"Primary Mixing and Blending"—Leo Dombrowski, Gibraltar Chemical; "High Viscosity Dispersion and New Dispersion Techniques"—James White, White Chemical Equipment Co.; "Small Media Milling and Dispersion Technology"—Leo Dombrowski; "Principles of Ball and Pebble Milling"—Warren Fuller, Paul O. Abbe, Inc.; and "Attritor Grinding and Dispensing Equipment"—Arno Szegvari, Union Process, Inc.

## Friday, May 12

"Assessing Pigment Dispersion through Fineness, Tint Strength, and Transparency"—Leo Dombrowski; "Particle Size Analysis of Coating Systems, Part I"—Theodore Provder; and "Particle Size Analysis of Coating Systems, Part II"—Theodore Provder.

## NYSCT/MNYPCA Coatings Course to Begin in March

Are you interested in learning more about paint formulation and calculations, dispersion techniques, application equipment and test methods? If so, "Understanding the Basics of Coatings II" may be the course for you.

Sponsored by the Joint Educational Committee of the New York Society for Coatings Technology and Metropolitan New York Paint and Coatings Association, this course is designed for individuals just entering the coatings industry as well as those who wish to gain a broader background in coatings fundamentals. No previous courses in chemistry are required.

"Understanding the Basics of Coatings II" will be conducted on the campus of Fairleigh Dickinson University, Elizabeth, NJ, beginning on March 29, 1995. The course will run for 15 consecutive Wednesday evenings.

For more information, contact Mildred Leonard, NYSCT Office, Room 208, 520 Westfield Ave., Elizabeth, NJ 07208; (908) 354-3200.



## "ADHESION PRINCIPLES FOR COATINGS AND POLYMER SCIENTISTS"

The adhesion course will outline surface chemistry and rheology relevant to adhesion, deformation, and fracture of elastomeric and glassy adhesives, surface preparation, plasma treatment, and adhesion promoters. Speakers will lecture on plastic to plastic adhesion, tack, surface analysis, release coatings, structural adhesives, and bond durability. Other subjects include adhesives in the web form, practical aspects of pressure sensitive adhesives, caulks and sealants, and adhesive application methods.

## Monday, May 22

"Interfacial Energies and Their Role in Adhesion"—Richard J. Ruch; "General Rheology and its Application to Adhesion"—Donald L. Hunston, National Institute of Standards and Technology; "Fracture Behavior of Glassy Adhesives"—Donald L. Hunston; and "Deformation and Fracture of Elastomeric Adhesives"—Alan N. Gent, The University of Akron.

## Tuesday, May 23

"Fracture Mechanics and Bond Durability Considerations"—James Koutsky, University of Wisconsin; "Etching, Cleaning, and Surface Modification of Polymer and Other Surfaces"—James Koutsky; "Plasma Surface Treatment and the Effect on Adhesion"—Eric Finson, Airco Coatings Technology; and "Organofunctional Silanes as Adhesion Promoters and Crosslinkers"—Bruce A. Waldman, OSi Specialties, Inc.

## Wednesday, May 24

"Plastic to Plastic Adhesion"—Rose Ryntz, Ford Motor Co; "Principles of Elas-

tomers Tack"—Gary R. Hamed, Institute of Polymer Science, The University of Akron; "Microscopic/Spectroscopic Studies in Adhesion Related to Durability of Adhesively Bonded Metals and Composites"—John G. Dillard, Virginia Polytechnic Institute; "Application of Surface Analysis to Adhesion of Coatings"—Kenneth D. Bomben, The Perkin-Elmer Physical Electronics Laboratory.

## Thursday, May 25

"Corrosion Aspects of Paint Adhesion and Adhesive Bond Durability"—Ray A. Dickie, Ford Motor Co.; "Surface Chemistry of Release"—Michael J. Owen, Dow Corning Corp.; "Fundamental and Practical Aspects of Pressure Sensitive Adhesives, Caulks, and Sealants"—Krishan C. Sehgal, Union Carbide Corp.; "Formulation, Application, and Evaluation of Adhesives in Web Form"—James A. Miller, Chemsultants International Network; and "Bonding and Chemistry of Structural Adhesives"—Kurt C. Frisch, Jr., 3M Co.

## Friday, May 26

"Bonding Plastics and Elastomers" and "Adhesive Application Methods"—Edward M. Petrie, ABB Transmission Technology Institute.



These programs are designed for research and development personnel who have interest in coatings, adhesives, elastomers, inks, and composites. Information may be obtained from Carl J. Knauss, Professional Development Institute, Kent State University, Chemistry Dept., P.O. Box 1792, Kent, OH 44240, (216) 672-2327.

## Coatings Newcomers Training Course Planned

All technical and nontechnical newcomers to the coatings industry are invited to attend the Philadelphia Society for Coatings Technology's Eastern Training Conference

and Show on May 8-9, 1996. Planned for the Valley Forge Convention Center, Valley Forge, PA, this course is designed to help attendees understand the challenges and opportunities in the coatings industry.

Participants will learn the basic principles of coatings technology during a two-day series of lectures. The following subjects will be covered: composition of coatings/resins; composition of coatings/pigments; composition of coatings/additives; trade sales paints; industrial finishes; laboratory testing; coatings formulation concepts; handling customer complaints; environmental considerations—MSDS; and coatings specification.

For more information, contact Wayne Kraus, Hercules, Inc., Research Center, 500 Hercules Rd., Wilmington, DE 19808; (302) 995-3435. For booth reservations, contact Larry Kelly, Eastech Chemical, Inc., 5700 Tacony St., Philadelphia, PA 19135.



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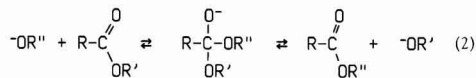
# Epoxy Nucleophile Catalyzed Transesterification

Gary P. Craun—The Glidden Company

## INTRODUCTION

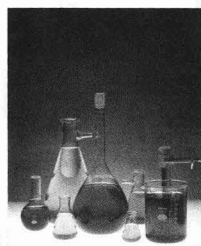
Transesterification as a cure chemistry for coatings is well-documented in the literature.<sup>1-4</sup> Fairly weak transesterification catalysts, such as zinc octoate, are generally used in transesterification cure with activated ester or hydroxyl functional materials. The more effective strong base transesterification catalysts, such as sodium methoxide, are too toxic and difficult to handle to be of practical use in coatings. Epoxy nucleophile catalyzed transesterification (ENCAT) utilizes a strongly basic alkoxide species which is produced in situ by the combination of a nucleophile with an epoxide moiety. Transesterification rates with ENCAT are sufficiently rapid to give good cure with simple ester and hydroxyl functional materials under normal bake conditions.

Transesterification occurs by an alcoholysis reaction in which an ester is cleaved by an alcohol in the presence of a basic catalyst.<sup>5</sup> The alcohol, which is converted to an alkoxide ion by the base, attacks the electron deficient carbonyl carbon of the ester causing cleavage:



The alkoxide in equation (1) is generated from the alcohol by reaction with a hydroxide ion. At higher temperatures (150-250°C), the alkoxide could be generated from an alcohol by reaction with a metal salt or a metal complex, such as butyl stannic acid, zinc octoate, or zinc acetylacetonate.

Since transesterification is an equilibrium reaction, the R' containing ester in equation (2) can only be produced by high yield if  $^-\text{OR}'$  is converted to an alcohol, and is then removed from the system. In transesterification cure the alkoxide can abstract a proton from an alcohol, forming a new catalytic alkoxide, and the new alcohol formed,  $\text{HO-R}'$ , can then escape from the coating as a volatile. (Typical levels of vola-



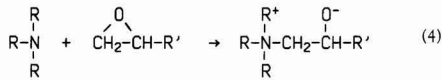
*Epoxy nucleophile catalyzed transesterification (ENCAT) is a new cure technology, which is particularly well-suited for coatings applications. Acrylic and acrylic-polyester blends in high-solids paints self-cure with ENCAT under normal bake*

*conditions to give an excellent balance of hardness, flexibility, and solvent resistance. Powder paints prepared with ester derivatives of epoxy resins self-cure, and likewise give an excellent balance of properties. The ENCAT catalyst is formed during cure by the combination of a nucleophile with an epoxide moiety. A strong base alkoxide, which is produced when the epoxide ring opens by nucleophilic attack, initiates transesterification. This mechanism is supported by <sup>13</sup>C NMR and gas chromatographic studies. Rate of transesterification is related to the structure of the hydroxyl and ester groups undergoing transesterification. Transesterification rate is directly related to the concentrations of the nucleophile and epoxy co-catalysts, as well as to the basicity and nucleophilicity of the nucleophile.*

tile alcohol generated during ENCAT cure range from 2-10% by weight based on binder.)

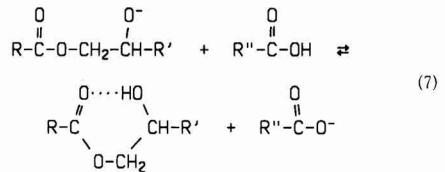
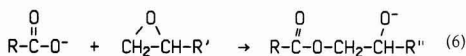
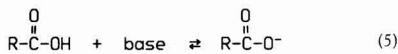
Alkoxide species can be formed when nucleophiles attack epoxides. Ring strain in the three member cyclic ether makes the epoxy C-O bond less stable than the C-O bond in linear or larger cyclic ethers.<sup>6</sup>

Presented at the 72nd Annual Meeting of the Federation of Societies for Coatings Technology, on October 13, 1994, in New Orleans, LA.  
\*16651 Sprague Rd., Strongsville, OH 44136.



Weakly basic nucleophiles, such as the acetate ion (equation (3)), form the potent alkoxide derivative of epoxy at temperatures above about 100°C giving ENCAT cure. This latency can provide one pack paint stability. More strongly basic nucleophiles, such as the tertiary amines (equation (4)), attack the epoxy at lower temperatures. Two pack paints catalyzed with tertiary amines and epoxies have ambient cure potential.<sup>4</sup> Although referred to as catalysts in this paper, the epoxy and the nucleophile function as initiators, not catalysts, for the transesterification reaction, because they are consumed in the process of forming an alkoxide. The alkoxide species produced from hydroxyl functional materials are the transesterification catalysts in ENCAT.

Carboxylic acids react with epoxy resins to form epoxy esters [equations (5)-(7)].



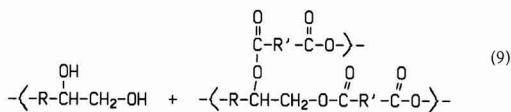
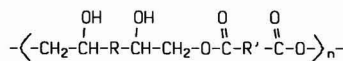
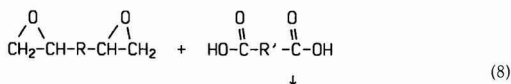
The ester product of (7) contains a secondary hydroxyl, which, under most circumstances, would be less reactive to transesterification than a primary hydroxyl. The carbonyl oxygen of the ester is, however, only six atoms removed from the hydrogen of the hydroxyl, and this close proximity allows hydrogen bonding to occur in a six member ring structure. Since hydrogen bonding can increase reaction rate,<sup>6</sup> it is hypothesized that this hydroxyl is activated by hydrogen bonding, allowing epoxy esters to undergo higher rates of transesterification and provide good cure.

Karel Dusek has published several papers on transesterification as a side reaction to the base (nucleophile) catalyzed reaction between diepoxides and dicarboxylic acids.<sup>7-9</sup> For pure diepoxides (bisphenol A diglycidyl ether) and dicarboxylic acids used in a 1:1 stoichiometric ratio, gel formation is not anticipated in polyaddition esterification. Dusek proposed that the linear epoxy ester polymer rearranges by transesterification as in equations (8) and (9):

#### Products Used in this Study

Company	Products	Company	Products
Air Products and Chemicals	1,4-diazabicyclo (2.2.2) octane (DABCO)	Fisher Chemical	Methanol
Akzo Chemie America	Dodecyl trimethyl ammonium chloride	IMC/Americhem	Trimethylol ethane
Aldrich Chemical	Acetic acid, benzoic acid, benzoin, benzyl dimethyl amine, benzytrimethyl ammonium methoxide, methanol, diethylsuccinate, dimethylisophthalate, N-N-dimethylbenzyl amine, potassium hydroxide, tertiary butyl phenyl glycidyl ether, and triphenyl phosphine	International Resources	T-butyl perbenzoate
Amoco	Isophthalic acid and styrene	M&T Chemicals	Butyl stannic acid
Arco Chemical	Propylene oxide and xylene	Monsanto	Adipic acid
BASF	1,6-hexane diol	Morton International	Ethyltriphenyl phosphonium acetate and tetrabutylphosphonium acid acetate
Ciba-Geigy	GT-7013	Parker Chemical	Bonderite 1000
Eastman Chemical	Dimethyl-1,4-cyclohexane dicarboxylate, isopropanol, n-butanol, and neopentyl glycol	Perstorp Polyols	Ethyl acrylate and trimethylol propane
		Rhône-Poulenc	Succinic anhydride
		Rohm and Haas	Hydroxy propyl methacrylate and methacrylic acid
		SBS Chemical	PL-200
		SCM Pigments	Zopaque RCL-6
		Shell Chemical	Epon 828 and Epon 1004
		Union Carbide	Methylamyl ketone





Not every epoxy ester polymer in the product is tied into the network. The glycol in equation (9) was derived from a high molecular weight linear epoxy ester, which was cleaved to form the low molecular weight glycol terminated segment and a branch. A series of such reactions creates a population of smaller molecules and a network. Dusek confirmed this sequence of reactions with a model system, demonstrating that transesterification occurs with stoichiometric mixtures of epoxy and acid after conversion to the epoxy ester has exceeded 90%. This rearrangement of epoxy ester polymers is consistent with ENCAT cure, but no volatile hydroxyl species are produced by the transesterification reaction, so crosslinking is limited.

Green disclosed a process for the transesterification of carboxylic or carbonic acid esters with alcohols.<sup>10</sup> He claimed a catalyst produced by the combination of an epoxide with a Lewis base which contains an element from the group: nitrogen, phosphorus, arsenic, antimony, and bismuth. Preferred Lewis bases are stated to be the cyclic amidines, and other nitrogen containing fused ring compounds. Green's work was limited to small molecule chemistry. Work summarized in this paper demonstrates the application of ENCAT to polymer systems in coatings.

## EXPERIMENTAL

### Model Compound Studies

Model compounds were prepared for a <sup>13</sup>C NMR study. Dodecyl trimethyl ammonium acetate (DTAA) was prepared by adding one mole of acetic acid to 526 g of dodecyl trimethyl ammonium chloride (50% solids in an isopropanol water mixture), and this mixture was then added to 1.0 equivalent of potassium hydroxide in 300 g methanol. Potassium chloride precipitated and was removed by filtration, leaving a dodecyl trimethyl ammonium acetate (DTAA) solution. Solvent was removed under vacuum.

Secondary hydroxypropyl acetate (HPA) was synthesized from 30 g acetic acid, 110 g propylene oxide, and 0.20 g triphenyl phosphine by enclosing the reactants in a pressure cylinder and heating the contents to 110°C for 1 hr, 120°C for 1 hr, and then to 125°C for 3 hr. The product was distilled, and <sup>1</sup>H NMR confirmed HPA as the distillate. Tertiary butyl phenyl glycidyl ether (TBPGE), which was distilled prior to use, DTAA, and HPA were combined in a 1:1:1 weight ratio. After reacting 1 hr at 150°C, the product mixture was sepa-

rated by preparative liquid chromatography into four fractions. These four fractions, the starting materials, and the product mixture were analyzed by <sup>13</sup>C NMR on a Varian XL-200M super conductive Fourier transform NMR system by the Major Analytical Instruments Facility of Case Western Reserve University.

### Transesterification Rate by Gas Chromatography

Transesterification rate was followed with gas chromatography. In a typical reaction a mixture of 33 g dimethyl-1,4-cyclohexane dicarboxylate, 23.7 g n-butanol, 3.7 g TBPGE, and 2.1 g N,N-dimethylbenzyl amine was added to methylamyl ketone to give a total of 100 ml. This mixture was heated in a single neck flask equipped with a reflux condenser in a 150°C oil bath. Samples were withdrawn through the condenser with a long needle syringe at 0, 15, 30, 60, 120, and 300 min, and placed immediately in dry ice. Varian model 3700 gas chromatograph response peaks were used to follow appearance and disappearance of species. Methylamyl ketone was used as the internal standard to which all peaks were normalized. Gas chromatograph oven conditions included an initial column hold at 60°C for 10 min, a programmed rise of 20°C/min to 240°C, and a 10 min hold at 240°C.

Quaternary ammonium carboxylates were prepared in methanol by combining 0.010 mole of benzyltrimethyl ammonium methoxide with 0.011 mole of an acid (such as benzoic acid). The resulting quaternary product was used directly as an ENCAT catalyst.

### Polymer Synthesis

Hydroxyl functional high-solids acrylic A was prepared in xylene under nitrogen at 145°C. A monomer mixture of 866 g styrene, 758 g ethyl acrylate, 544 g hydroxy propyl methacrylate, and 28 g methacrylic acid was prepared, and then 440 g of the mixture was placed in 1098 g xylene in a 5 liter reaction flask with 87 g t-butyl perbenzoate. After a 15 min polymerization exotherm, the remainder of the monomer mixture was pumped into the flask over 3 hr. The acrylic was held at 145°C for 2 hr, 222 g of additional xylene was added, and the polymer solution was cooled.

Hydroxyl functional high-solids polyester A was prepared with 146 g adipic acid, 166 g isophthalic acid,

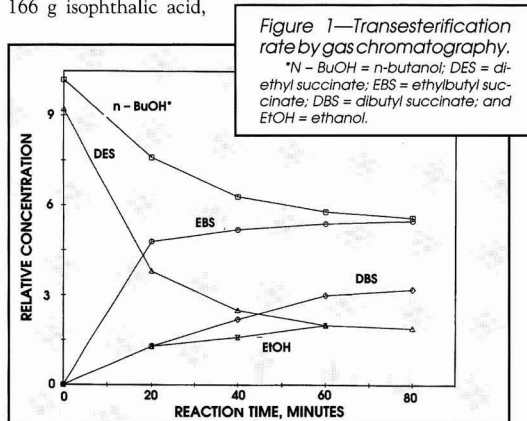


Table 1—Transesterification of Model Compounds

Ester <sup>a</sup>	Alcohol	Relative Temperature	Rate
Diethylsuccinate <sup>b</sup> .....	n-butanol <sup>c</sup>	100°C	0.502
Diethylsuccinate .....	isopropanol <sup>c</sup>	100°C	0.038
Diethylsuccinate .....	isopropanol	140°C	0.090
Dimethyl cyclohexane dicarboxylate <sup>c</sup> .....	n-butanol	140°C	0.041
Dimethyl isophthalate <sup>b</sup> .....	n-butanol	100°C	0.938
Amylacetate .....	neopentylglycol	100°C	0.834

- (a) Methylamyl ketone<sup>d</sup> was used as an internal standard. ENCAT co-catalysts were 5% by weight butylphenyl glycidyl ether<sup>a</sup> and 2% by weight benzyltrimethyl amine.<sup>b</sup>  
 (b) Aldrich Chemical Co.  
 (c) Eastman Chemical Co.  
 (d) Union Carbide Corp.

312 g neopentyl glycol, and 0.20 g butyl stannic acid. Monomers were heated under nitrogen in a reaction flask which was equipped with a glass bead packed column to the point at which water began to reflux through the column (about 170°C), and then the reaction temperature was slowly raised to 220°C over about 3 hr. Reflux temperature at the top of the column was maintained at 99 to 100°C during the reaction. After an acid value of less than 10 mg potassium hydroxide per gram of resin was obtained, the polyester was cooled. Polyester B was prepared by this procedure with 1463 g 1,6-hexane diol, 1933 g neopentyl glycol, 4108 g isophthalic acid, and 766 g trimethylol ethane. Polyester C was prepared by this procedure with 146 g adipic acid, 664.8 g isophthalic acid, and 1342 g trimethylol propane.

An ester functional epoxy resin derivative (epoxy ester) for powder coatings was prepared from 173 g methanol, 494 g succinic anhydride, and 5,300 g of Epon 1004, 1000 epoxy equivalent weight solid epoxy resin. The succinic anhydride and methanol were slowly heated under reflux to 100°C, and held for 2 hr to form the methyl succinate half ester. The solid epoxy resin was added with 15 g benzyltrimethyl amine, and the reaction mixture was held at 120° for 2 hr. The epoxy ester product had a residual acid value of 0.08 meq. acid/g and a residual epoxy content of 0.06 meq. epoxy/g.

### Paint Preparation and Testing

High-solids paints were prepared at a pigment to binder weight ratio of 0.85 to 1.0. Zopaque RCL-6 rutile titanium dioxide was dispersed into high-solids acrylic and polyester resins in a Cowles disperser at 2500 rpm for about 15 minutes. Temperature was maintained at <140°F. Liquid epoxy resin Epon 828 (190 equivalent weight liquid epoxy, 11.5% on resin solids), and nucleophile (2.2% on resin solids, benzyltrimethyl ammonium acetate, or 3% dodecyltrimethyl ammonium acetate, both prepared as previously mentioned) were mixed into the paint just prior to cure. Films were drawn down on Bonderite 1000 panels to give 1.0 mil baked films. Film bakes were 20 min at 300 or 375°F.

Powder paints were prepared by dry mixing 1.0 kg epoxy ester with 100 g of GT-7013, which is a 400 equivalent weight solids epoxy, nucleophile, 760 g titanium dioxide, 9 g benzoin, and 10 g of PL-200 acrylic flow agent. Well-mixed dry blends were extruded at 80°C, and then the melt was passed over a chilled two roll mill to form a flake. This flake was

ground and then sieved with a 140-mesh screen. The powder was electrostatically sprayed onto panels, and baked for 20 min at 300°F to give 1.5 mil films.

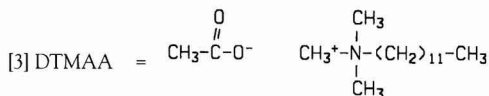
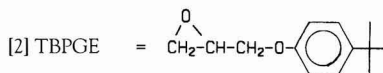
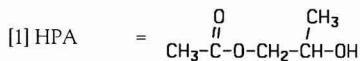
Pencil hardness was measured with Eagle Turquoise pencils (ASTM Method D 3320 (04.04)). Impact resistance was the maximum impact in joules that the film withstood without cracking when impacted with a 5/8 inch dart. Methylene chloride (MEK) double rubs (solvent resistance) was determined by soaking a cotton rag with MEK, and then rubbing the solvent-soaked rag across the film

surface with moderate pressure. The rag was re-soaked with MEK every 20 double rubs. Failure was recorded at the point where the coating was just removed from the panel. Mar was determined after 100 rubs by comparing rubbed to unrubbed film.

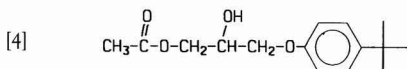
## RESULTS AND DISCUSSION

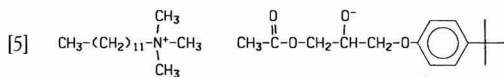
### Model Compound Studies

Model compound studies support the mechanism of ENCAT as proposed in this paper. Hydroxypropyl acetate (HPA), *t*-butylphenylglycidyl ether (TBPGE), and dodecyltrimethyl ammonium acetate (DTMAA) were prepared, reacted, and analyzed by <sup>1</sup>H NMR as outlined in Experimental (structures are presented in the following as compounds [1], [2], and [3]).



Spectra of the reactants were consistent with the structures given. The product mixture had some unreacted starting material in it, but new products were formed. Of the samples which were isolated with preparative liquid chromatography, two most clearly point to the proposed ENCAT mechanism. They had <sup>13</sup>C NMR spectra which were consistent with





Compound [4] could have been formed by the reaction of acetate from DTMAA with the epoxy group in TBPGE, with the subsequent abstraction of a proton from a less basic species. Compound [5] would be formed in similar fashion to [4], but without neutralization of the alkoxide ion. These results are consistent with hypothesis that a nucleophile (acetate from the DTMA) attacks an epoxide (TBPGE) opening the epoxide ring. The strongly basic alkoxide species thus produced (in compound [5]) initiates transesterification.

## TRANSESTERIFICATION RATE STUDIES

### Evidence of Transesterification by Gas Chromatography

Transesterification rate studies were done to further establish that ENCAT is a transesterification cure reaction, and to investigate the effect of catalyst and reactant structure on transesterification rate. Figure 1 is a plot of the reactant and product concentrations as a function of time during the ENCAT reaction of diethylsuccinate with n-butanol. Liquid epoxy (Epon 828) is the epoxide, and a polymeric tertiary amine (proprietary epoxy amine adduct with tertiary amine functionality) is the nucleophile. Values plotted are gas chromatography peak areas relative to a decane internal standard. (These are gas chromatograph response peaks, no correction for actual concentration has been made.) As diethylsuccinate and n-butanol are consumed, three new species appear: ethanol, ethylbutylsuccinate, and dibutylsuccinate. These are the expected products of a transesterification reaction. Since transesterification is a reversible reaction, and this experiment was conducted in a closed environment, concentrations of reactants and products reach equilibrium values. Initial reaction rates were determined by fitting an equation of the form

$$[\text{ester}] = a + b \cdot \text{EXP}(c \cdot x)$$

to the declining peak area of the ester functional reactant. Initial transesterification rate was taken to be the absolute value of the slope as the curve approached time zero.

### Transesterification Rate: Nucleophile and Epoxy Co-Catalyst Effects

Basicity of the nucleophiles used in ENCAT reactions was found to be a key factor in determining transesterification rate. Acids with higher  $\text{pK}_a$ 's, such as phenol ( $\text{pK}_a 10$ )<sup>11</sup> would, upon loss of its proton, produce a stronger base (the phenolate ion), than would acetic acid (the acetate ion), since acetic acid has a  $\text{pK}_a$  of 4.7 (12). This relationship between nucleophile basicity and transesterification rate was established by using a series of quaternary salts as the nucleophile in ENCAT reactions. Initial transesterification rates in Figure 2 are plotted as a function of the  $\text{pK}_a$  of the acid which was used to make the quaternary salt. As conjugate acid  $\text{pK}_a$  goes from 10 (quaternary phenolate), to 4.7 (quaternary acetate), to 3.6 (quaternary formate), reaction rate drops by two orders of

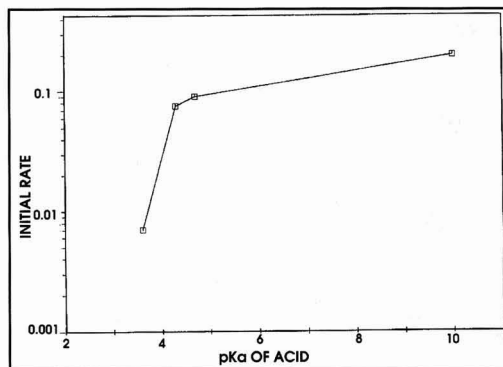


Figure 2—Initial rate versus  $\text{pK}_a$  of acid used to prepare quaternary ammonium nucleophile.  
\*Benzyltrimethyl ammonium carboxylates and phenolate.

magnitude, so transesterification rate is directly related to the  $\text{pK}_a$  of nucleophile's conjugate acid.

Figure 3 contains reaction rates from a series of ENCAT reactions in which the nucleophiles were present as their potassium salts. Transesterification rate is plotted here as a function of nucleophilicity.<sup>12</sup> (Nucleophilicity is a dimensionless quantity which relates to the nucleophilic strength of the species.) Rate increases with nucleophilicity in both plots, but the nonbasic halogen nucleophiles, chloride, bromide, and iodide, have much lower rates of transesterification at equivalent nucleophilicity than the basic nucleophiles, acetate, and phenolate. Transesterification rate increases with both the basicity and the nucleophilicity of the nucleophile.

Transesterification rate data plotted in Figure 4 was generated with an excess of liquid epoxy at three concentrations of ethyltriphenyl phosphonium acetate (ETPPA). Note that rate is directly related to ETPPA concentration. Rate in Figure 5, however, is not a linear function of liquid epoxy concentration. This nonlinearity may be caused by acidic species (acetic acid is an impurity in the ETPPA), which produce a loss of

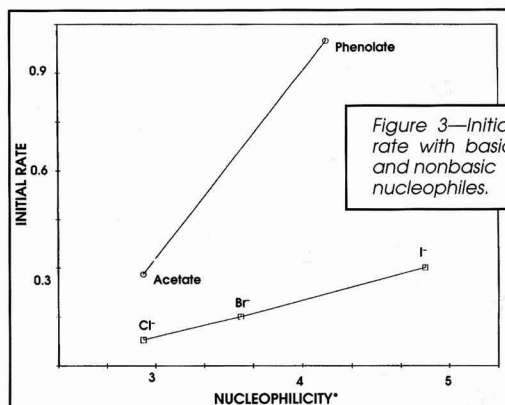


Figure 3—Initial rate with basic and nonbasic nucleophiles.



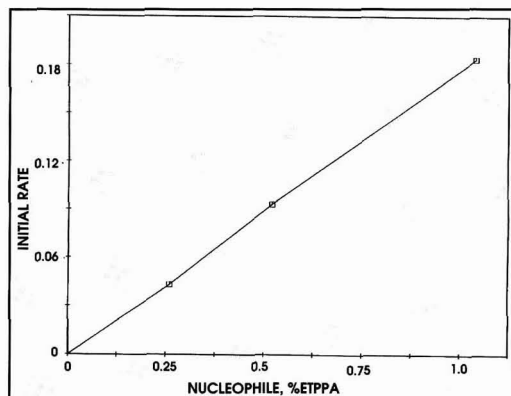


Figure 4—Initial rate versus nucleophile concentration.

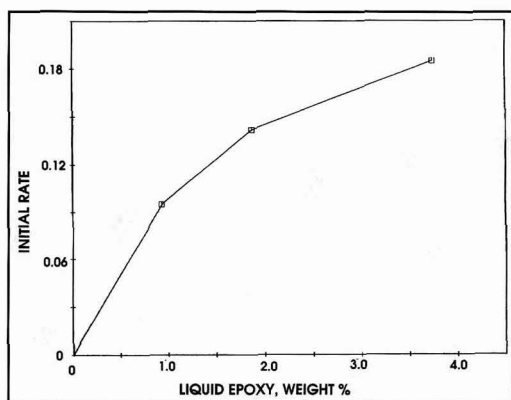


Figure 5—Initial rate versus epoxy concentration.

Table 2—High-Solids Paints

Sample <sup>a</sup>	Polyester	Film Bake	Pencil Hardness	Solvent Resistance	Impact Resistance
HS-1	none	150°C	3H	100	<40 joules
HS-2	16% A	150°C	4H	100	>217 joules
HS-3	13.6% B	150°C	4H	100	>217 joules
HS-4	6% C	150°C	4H	100	>217 joules
HS-5	16% A	190°C	N/A	<10	N/A
HS-6	16% A	190°C	N/A	100	>217 joules

(a) Sample HS-6 was catalyzed with 11.5% liquid epoxy (Epon 828) and 3.0% dodecyltrimethyl ammonium acetate. All other samples had 11.5% liquid epoxy and 2.2% benzyltrimethyl ammonium acetate (see Experimental). Solvent resistance in MEK rubs were stopped at 100. Most samples were only slightly marred at 100 rubs. Polyester levels were adjusted to give 0.61 eq. hydroxyl per kg total resin solids. Impact resistance values are forward and reverse direction.

epoxy functionality. The presence of acidic species like acetic acid keep the pH of the reaction mixture low until they are consumed by the epoxy.<sup>13</sup> Since transesterification is a base catalyzed reaction, it cannot occur until the acid is consumed. This epoxy ester reaction occurs during the induction period at the beginning of the ENCAT reaction. At a constant ETPPA concentration, the length of this induction period decreases as epoxy concentration rises. When liquid epoxy is used at a concentration which is significantly larger than the acid concentration, this induction period approaches zero. Transesterification rate after the induction period should be proportional to the concentration of the remaining liquid epoxy.

Although the range of nucleophiles which can be used for ENCAT is quite broad, heterocyclic compounds other than simple epoxides do not seem to work. Figure 6 contains the structures of a variety of heterocyclic compounds which were tested in a series of rate experiments. Each heterocyclic compound was tested with Aldrich Chemical Company's sodium acetate and potassium iodide as the nucleophile. With the exception of hydroxyethyl ethylene imine (HEEI) none of the compounds gave any evidence of transesterification. Since HEEI is stabilized with potassium hydroxide, and it is not stable under acidic conditions, it was difficult to determine whether the potassium hydroxide of the aziridine was acting as the catalytic species.

#### Transesterification Rate: Ester and Hydroxyl Structure Effects

Initial transesterification rates by gas chromatography were determined for a series of esters and alcohols (see Table 1).

Diethylsuccinate and n-butanol have an initial relative transesterification rate of 0.502 at 100°C. When the linear n-butanol is changed to a secondary alcohol, isopropanol, the rate decreases by more than an order of magnitude to 0.038. By raising the reaction temperature to 140°, rate increases with diethylsuccinate and isopropanol about twofold to 0.090. Branching in a primary alcohol, neopentyl glycol, does not prevent a high rate of transesterification with the simple linear ester, amylacetate.

A hindered ester, dimethyl cyclohexane dicarboxylate, when transesterified with n-butanol, has a very low rate of 0.041, even at 140°C. The aromatic ester, dimethyl isophthalate, has a more compact ring structure, and is thus less hindered than dimethyl cyclohexane dicarboxylate. At 100°C the transesterification rate of dimethyl isophthalate with n-butanol is 0.923, higher even than that of diethylsuccinate and n-butanol.

Transesterification rate in ENCAT reactions is related to alcohol and ester structure. Rate with linear aliphatic esters is higher with primary than with secondary alcohols. Cycloaliphatic esters react slowly with primary alcohols, while aromatic esters have a fairly high transesterification rate. These results suggest that many of the monomers which are commonly used to prepare polyesters and acryl-

ics could be used in making ENCAT cured paints.

### Paint Properties

High-solids acrylic A, which contains 24% hydroxypropyl methacrylate at 63% solids in xylene, self-cured in the presence of liquid epoxy and benzyltrimethyl ammonium acetate (HS-1 in Table 2). The hydroxypropyl methacrylate (HPMA) in the acrylic acts as both a hydroxyl and an ester functional monomer. Propylene glycol would be liberated when two HPMA units condense to form an ester crosslink. This paint has good solvent resistance and hardness, but poor impact resistance.

High-solids acrylic A was also evaluated at a 150°C, 20 min bake (see Experimental Section) with polyesters A, B, and C (see Table 2). Levels of the polyesters were adjusted to give 0.61 eq. polyester hydroxyl functionality per kg total resin solids. With as little as six percent polyester, acrylic A produced paint films with >217 joules forward and reverse impact resistance, while maintaining 100 MEK rubs of solvent resistance and 4H pencil hardness. If terminal polyester hydroxyl groups reacted with HPMA units in the acrylic (again liberating propylene glycol), then the polyester could function as a crosslinker for the acrylic. A flexible ester crosslink provided by the polyester could provide toughness that gave good impact resistance. These acrylic-polyester blends gave an exceptional combination of hardness, flexibility, and solvent resistance.

At a higher temperature bake, 180°C, dodecyltrimethyl ammonium acetate gave excellent cure, while benzyltrimethyl ammonium acetate at the same molar concentration gave poor solvent resistance and impact resistance. Poor cure with the benzyltrimethyl quaternary ammonium salt is likely due to thermal degradation of the nucleophile.

An epoxy ester powder resin was evaluated with 1,4-diazabiscyclo (2.2.2) octane (DABCO) and tetrabutylphosphonium acid acetate (TBPA) as the nucleophile (see Table 3). Since the epoxy has a very low level of active epoxy functionality (see Experimental Section), solid epoxy resin was added to provide catalytic epoxy functionality.

Although DABCO is a strongly basic nucleophile, cure, as measured by solvent resistance, impact resistance, and pencil hardness, was significantly better with TBPA [(Table 3), sample P-1 versus P-2, P-3]. Ammonium acetates, such as were used above in high-solids coatings, and phosphonium acetates, such as TBPA, have proven to be especially effective nucleophiles for ENCAT cure. Lower molecular weight nucleophiles like DABCO may be volatile enough to leave the film during bake before good properties are produced. In addition to being low in volatility, ammonium and phosphonium acetates have good compatibility with the polymers used in these high-solids and powder paints.

This epoxy ester appears to self-cure with ENCAT. The terminal methyl succinate ester transesterifies with hydroxyl groups on the epoxy ester backbone, liberating methanol. As stated in the Introduction, these secondary hydroxyls can be

Table 3—Powder Paints

Sample	Nucleophile	Solvent Resistance	Impact Resistance	Pencil Hardness
P-1	3.0% 1,4-diazabiscyclo-(2.2.2)octane <sup>b</sup>	40	<30 joules	H
P-2	3.0% tetrabutylphosphonium acid acetate <sup>c</sup>	100	>217 joules	5H
P-3	1.5% tetrabutylphosphonium acid acetate	100	>217 joules	4H

(a) Solvent resistance in MEK rubs were stopped at 100 rubs. Forward impact resistance in joules.  
 (b) Air Products and Chemicals, Inc.  
 (c) Morton International, Inc.

activated by their close proximity to the ester carbonyls of the epoxy ester. This activation would explain the excellent cure obtained in these powder paints.

### CONCLUSIONS

Model compound studies with <sup>13</sup>C NMR support the hypothesis that nucleophiles and epoxies act as co-catalysts in transesterification cure. The basic alkoxide ion is produced by

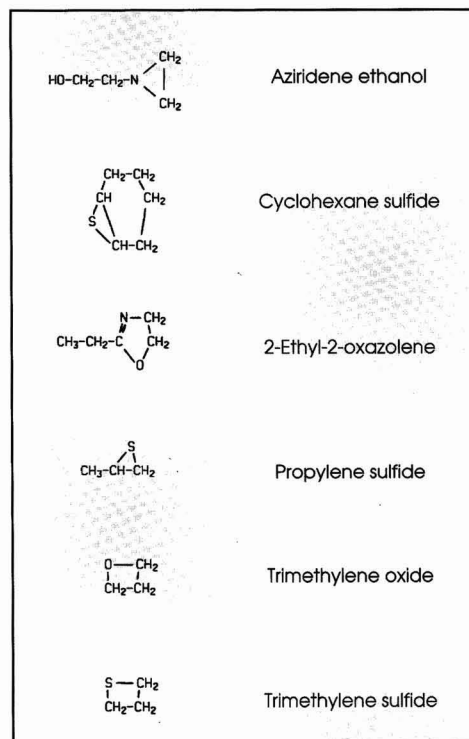


Figure 6—Heterocyclic compounds.

nucleophilic attack on the epoxide group in a manner which is consistent with known epoxy chemistry. The alkoxide initiates transesterification in a manner which is similar to other bases, such as sodium methoxide. Transesterification rate in ENCAT reactions can be followed by gas chromatography. Rate is directly proportional to nucleophile and liquid epoxy concentrations. Epoxy reactions with carboxylic acids give an induction period prior to the transesterification reaction, especially at low liquid epoxy concentrations. ENCAT rate varies as would be expected with the structure of ester and hydroxyl functional materials (rate is slower with more hindered structures).

Paints produced with mixtures of hydroxyl functional polyesters and acrylics give films with an exceptional combination of solvent resistance, hardness, and impact resistance, with as little as six percent polyester. Hydroxyl functional acrylics will self-cure to give good solvent resistance and hardness, but impact resistance is lacking. Powder paints can be produced with methylsuccinate ester derivatives of epoxy resins. When cured with active epoxy resin and a phosphonium acetate, the powder paint films had good pencil hardness, impact resistance, and solvent resistance.

Epoxy nucleophile catalyzed transesterification is a significant new cure chemistry for coatings, which utilizes low cost and readily available raw materials. Good cure with a balance of hardness, solvent resistance, and impact resistance can be obtained under normal bake conditions.

## ACKNOWLEDGMENTS

The author is grateful to David Trumbo and Kirk Abbey for their insight and stimulating conversation about transesteri-

fication mechanisms, and to Susan Sobek for her help in preparing and testing high-solids paints.

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# Initiators for High-Solids Polyacrylates Resins: Selection Guidelines and Performance Review

Ginger G. Myers—Akzo Nobel Chemicals, Inc.\*

## INTRODUCTION

Over the past several years, increasingly stringent environmental regulations have exerted pressure to reduce levels of organic solvents in coatings applications. One solution is higher-solids acrylate resins. These resins are most commonly used in automotive refinishing and coating applications. Resins are typically high in polymer content with the lowest possible solvent content to give reasonable flow properties. Initiator properties contribute to the final resin molecular weight, viscosity, and polydispersity.

Relatively little information has been published on the effects of specific initiators (i.e., organic peroxides) on acrylate resin characteristics. Most literature on free-radical polymerization of acrylic monomers gives minimal information on the selection of initiator for the system. However, several papers have been published which compare tert-butyl and tert-amyl peroxides.<sup>1</sup> These papers stress the advantages tert-amyl peroxides offer over their tert-butyl equivalents. However, our work has shown that tert-butyl peroxides can give polyacrylates with similar properties as those polymers made using tert-amyl peroxides.

Since initiators can be a significant expense in resin manufacture, and can affect the batch cycle times and conversion, we have evaluated a large number of organic peroxides for high-solids resins. We have chosen two procedures for evaluating organic peroxides: a high-solids model system and the standard ampoule technique.

## EXPERIMENTAL

### Model System

Our model system consists of a typical original equipment manufacturer (OEM) monomer blend, an appropriate solvent (usually aromatic) for the polymerization temperature and the initiator (6 mmol/100g monomer). A standard monomer blend is shown in Table 1. Monomers were selected to give a broad range of reactivity and functionality in the system. MAA gives the -COOH function; HEMA gives the -OH function. Styrene is the least reactive monomer in the system, while BA is the most reactive.

*This paper covers the selection criteria for choosing an effective free-radical initiator for the polymerization of acrylic monomers. Two basic systems have been used to evaluate a large number of organic peroxides, including an original equipment manufacturer (OEM) model system. The systems and resin characterization procedures are described. Results of approximately 100 polymerizations using different initiators and conditions are presented. Table 6 summarizes all of the results obtained and can be used for initiator selection screening. Resin characterization, cost performance, and conversion are used as guidelines in all of the initiator evaluations.*

Standard solvents used are Exxate<sup>®</sup> 700 (isoheptyl acetate mixture) and Solvesso<sup>®</sup> 100 (aromatic mixture C<sub>8</sub>-C<sub>11</sub>, containing 75% mesitylene isomers). Theoretical solids content is 71%, to allow for ease of handling and adequate agitation during polymerization. The apparatus used in our model system consists of a 500-mL jacketed glass reactor fitted with a reflux condenser, temperature probe, and nitrogen bypass. The reaction temperature is maintained by a recirculating bath equipped with a temperature controller. Monomers and initiators are dosed at constant rates via a metering pump.

Exxate and Solvesso are trademarks of Exxon Chemical.

**Table 1—Standard OEM Monomer Mix**

40% .....	Butyl acrylate (BA)
28% .....	2-Hydroxyethyl methacrylate (HEMA)
20% .....	Styrene
10% .....	Methyl methacrylate (MMA)
2% .....	Methacrylic acid (MAA)

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Table 2—Initiators for High-Solids Resins

Akzo Nobel Trade Name	Chemical Name	Abbreviation	1 Hr Half-Life T, °C	Active Oxygen, %
Cadef® BPO-78W	Dibenzoyl peroxide	BPO	92	5.15
Perkadox® AIBN	2,2'-Azobis (isobutyronitrile)	AIBN	79	8.36 <sup>a</sup>
Perkadox AMBN	2,2'-Azobis (2-methylbutyronitrile)	AMBN	84	7.14 <sup>a</sup>
Perkadox BC	Dicumyl peroxide	DCP	128	5.80
TAPIN	tert-Amyl peroxy-3,5,5-trimethylhexanoate	TAPIN	105	6.35
Trigonox® 121	tert-Amyl peroxy-2-ethylhexanoate	TAPEH	85	6.60
Trigonox 127	tert-Amyl peroxybenzoate	TAPB	116	7.22
Trigonox 133-C60	tert-Amyl peroxyacetate	TAPA	108	6.56
Trigonox 141	2,5-Dimethyl-2,5-(2-ethyl-hexanoylperoxy) hexane	DEHH	—	—
Trigonox 201	Di-tert-amyl peroxide	DTAP	124	8.70
Trigonox 21	tert-Butyl peroxy-2-ethylhexanoate	TBPEH	90	7.18
Trigonox 29	1,1-Di-(tert-butylperoxy)-3,3,5-trimethylcyclohexane	DTBTC	104	10.05
Trigonox 42S	tert-Butyl peroxy-3,5,5-trimethylhexanoate	TBPIN	110	6.73
Trigonox A-W70	tert-Butyl hydroperoxide	TBHP	199	12.43
Trigonox B	Di-tert-butyl peroxide	DTBP	136	10.83
Trigonox BPIC	tert-Butylperoxy isopropyl carbonate	BPIC	114	6.81
Trigonox C	tert-Butyl peroxybenzoate	TBPB	122	8.07
Trigonox D-E50	2,2-Di-(tert-butyl peroxy) butane	DTBB	114	6.83
Trigonox F-C50	tert-Butylperoxyacetate	TBPA	117	6.05

(a) Active nitrogen, %.

Cadef, Perkadox, and Trigonox are registered trademarks of Akzo Chemicals Inc.

### Ampoule Technique

For evaluating higher conversion initiators, the ampoule technique is employed. This consists of MMA solvent polymerization in a 3-mL amber ampoule. We typically use MMA, at 65% wt in toluene and initiator concentration of 6-10 mmol/L MMA. The polymerization takes place in an oil bath held at constant temperature. Samples are taken at fixed intervals in order to determine the degree of polymerization. Intermediate samples are quenched with an antioxidant (i.e., Topanol® OC) in ethylbenzene or another appropriate solvent and set aside for later determination of residual monomer content.

### Resins Characterization

Final high-solids resins are characterized by molecular weight, polydispersity, and viscosity determinations. Molecu-

Topanol is a registered trademark of ICI Specialty Chemicals.

Table 3—Initiator Cost Comparison

Product	Assay, %	Act. Ox, %	\$/lb <sup>a</sup>	\$/%A.O.
TBPIN	97	6.94	8.43	1.25
TBPA	50	12.10	3.38	0.56
TAPA	60	10.93	6.25	0.95
TAPEH	95	6.95	5.50	0.83
TBPEH	97	7.40	3.83	0.53
DTBTC	95	10.58	6.69	0.66
BPO	78	6.61	2.88	0.56
DTBP	99	10.94	2.37	0.22
DTAP	95	9.18	5.76	0.66
AIBN	99	8.53 <sup>b</sup>	6.30	0.75
AMBN	99	7.29 <sup>b</sup>	7.20	1.00
TBPB	98	8.24	3.05	0.38
BPIC	75	9.08	8.26	1.21
TAPB	94	7.68	4.27	0.59
DCP	99	5.92	5.18	0.89

(a) Published list prices as of Jan. 1994.

(b) Active nitrogen, %.

lar weight and molecular weight distribution are determined by gel permeation chromatography using polystyrene standards. Viscosity is measured using the falling ball method at 25°C. Solids contents are determined by percent nonvolatile matter. For the ampoule technique, residual monomer is determined by gas chromatography.

## RESULTS AND DISCUSSION

A large number of initiators have been screened in our model system. Table 2 lists the initiators that are included in this report. These initiators were selected on the basis of half-life temperatures, which should fall into the polymerization temperature range used in the industry. The products are all liquids except BPO, DCP, and the azo initiators (AIBN and AMBN), which are free-flowing solids. Table 3 shows the cost comparisons on an active oxygen basis (or nitrogen basis in the case of the azos) of the initiators tested. On this basis alone, it appears that the most effective initiators are DTBP, TBPB, BPO, TBPA, and TBPEH. However, there are other considerations in selecting the most efficient initiator. These will be covered in detail later.

### Results

**Test Series I. Tert-Amyl Versus Tert-Butyl Peroxides:** We selected the following three pairs of organic peroxides to compare in this test series: (1) TBPEH and TAPEH (peroxy-2-ethyl hexanoates); (2) DTBP and DTAP (di-tert-butyl and di-tert-amyl peroxides); and (3) TBPIN and TAPIN (peroxy isononanoates).

TBPEH and TAPEH were evaluated at several temperatures and concentrations in the ampoule technique. (See results in Appendix 1a). Results show that very similar polymerization rates can be achieved by adjusting these two parameters. Specifically, when TBPEH is used at 8 mmol/L

Table 4—Initiator Half-Life Temperature

Product	40-min Half-Life Temperature, °C
AMBN .....	90
TBPEH .....	94
BPO .....	96
TBPIN .....	115
TAPA .....	116
BPIC .....	118
DTBTC .....	119
TBPA .....	121
DTAP .....	137
DTBP .....	142

MMA at a polymerization temperature of 80°C, it gives identical polymerization curves to TAPEH at 6 mmol/L run at 76°C. This higher usage rate is offset by the lower price of TBPEH versus TAPEH (\$0.53/%a.o. and \$0.83/%a.o., respectively).

DTBP and DTAP were selected for high-solids resins in the higher temperature range (150-180°C). Results are shown in Appendix 1b. Three parameters were modified to compare these two peroxides: temperature, initiator concentration, and metering time. Temperature and initiator concentration have a significant effect on the final polymer viscosity and molecular weight. Metering time did not appear to affect the resin properties in our experiments. Again we observed that comparable resins can be obtained from DTBP and DTAP, if an increase in temperature and/or initiator concentration can be tolerated. In this case, the cost per active oxygen of the DTAP is three times the cost of DTBP, so there is considerable incentive to use the tert-butyl derivative.

In the third comparison, TBPIN and TAPIN (tert-butyl and tert-amyl peroxyisobutyrate) were used. These trials were similar to the DTBP and DTAP comparisons, but at a

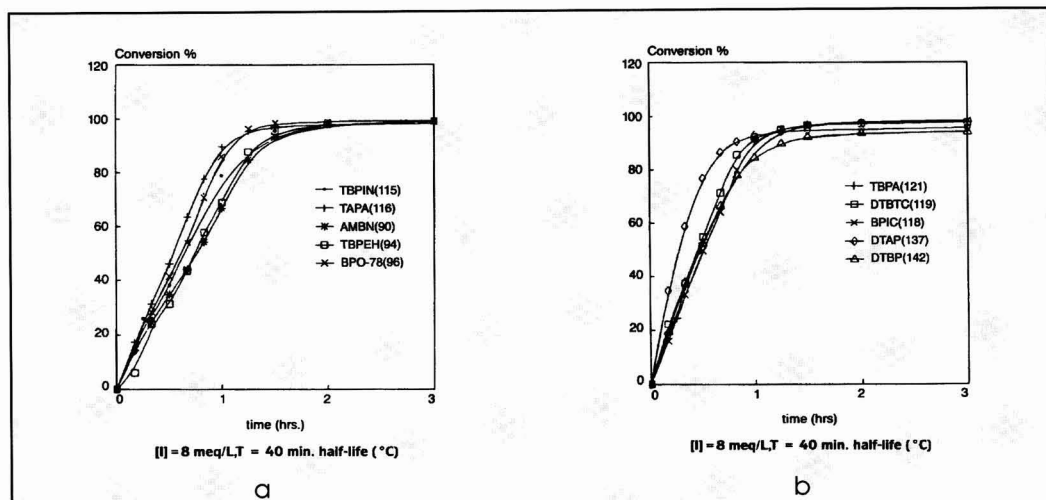
much lower temperature range (124-157°C). For these products, we saw effects on the final resin properties from all three parameter changes, particularly for TBPIN. See results in Appendix 1c. Metering time had less influence than initiator concentration and temperature. Temperature had the largest influence: resin made with TBPIN at 157°C had identical properties to resin made at 148°C using TAPIN.

From this data, it can be concluded that significant cost savings can be obtained through the use of tert-butyl peroxides whenever possible. A careful review of the system temperature range, initiator concentration, and dosing rates could allow for direct substitution without detrimental effects to the final resin properties.

**Test Series II: High Conversion Initiators:** Ten initiators were selected for conversion evaluations using the ampoule technique. Polymerizations were conducted at the respective 40 min half-life temperature of each initiator (see Table 4). Cycle time was three to four hours. For this series, we used Exxate 700 (initial b.p. approximately 176°C) as the solvent to accommodate the wide temperature range of initiators used.

All initiators were tested at 8 mmol/L. Some were also tested at different concentrations and temperatures to evaluate the effects on conversion rates. Figures 1a and 1b show all of the initiators tested at 8 meq/L. TBPEH, BPO, and AMBN gave the highest conversion after three hours (all about 99%).

Figures 2 and 3 show conversion versus polydispersity (D) and average molecular weight, respectively. At higher temperatures we observed a decrease in conversion for MMA. This might be due to the increased potential for polymer "unzipping" at elevated (>110°C) temperatures. We also observed a relationship between molecular weight distribution and conversion. For example,  $M_w$  and D generally decreased with an increase in polymerization temperature. (This was



Figures 1a & 1b—PMMA solvent conversion initiator comparison.



Table 5—Initiators Tested in High-Solids System

Initiator	T, °C	% Solids	Visc, P	M <sub>w</sub> (10 <sup>3</sup> )	M <sub>n</sub> (10 <sup>3</sup> )	Solvent
AIBN .....	93	69.8	993	17.8	8.8	Exxate 700
	93	70.0	127	13.2	7.1	Butace
BPO .....	107	72.1	846	12.1	5.6	Exxate 700
	107	71.9	160	11.7	5.4	Butace
DCP .....	142	70.9	128	8.6	4.2	Exxate 700
TAPEH .....	98	70.1	520	14.0	7.6	Exxate 700
	98	70.0	127	11.2	5.3	Butace
TAPB .....	129	72.4	432	13.3	5.7	Exxate 700
TBPEH .....	103	70.2	965	24.9	9.4	Exxate 700
	103	70.7	24	19.5	6.7	Butace
DTBTC .....	119	71.7	3320	47.6	12.2	Exxate 700
TBPIN .....	124	71.5	745	18.7	7.3	Exxate 700
	135	71.6	273	11.7	5.4	Exxate 700
* .....	135	71.1	138	8.6	4.1	Exxate 700
	126	71.6	181	16.2	6.7	Butace
	124	72.0	845	12.3	5.5	Solvesso 100
BPIC .....	126	71.2	577	18.0	6.3	Exxate 700
	126	71.8	259	21.4	7.2	Butace
TBPB .....	135	72.9	640	14.1	5.3	Exxate 700
TBPA .....	131	69.5	240	12.1	5.6	Exxate 700
DTBP .....	151	72.6	231	12.3	4.4	Exxate 700
TAPA .....	126	71.7	200	9.3	5.0	Exxate 700

Note: All polymerizations done at 30 meq active oxygen, except where noted.

\* = 45 meq active oxygen.

Table 6—Initiator Performance Summary

Initiator	HS Resin Properties			Cost	Conversion Rate (MMA)	Overall Rating
	Low V	High M <sub>w</sub>	Low D			
TBPIN .....	+	+	+	-	+	4+
TBPA .....	+	+	+	+	-	4+
TAPA .....	+	-	+	-	+	3+
TAPEH .....	+	-	+	-	+	3+
TBPEH .....	-	+	-	+	+	3+
DTBTC .....	-	+	-	+	-	2+
BPO-78 .....	-	-	-	+	+	2+
DTBP .....	+	-	-	-	+	2+
DTAP .....	-	+	-	-	+	2+ <sup>a</sup>
AIBN .....	-	+	+	+	-	3+
AMBN .....	+	+	-	+	-	2+ <sup>a</sup>
TBPB .....	+	-	+	+	-	3+ <sup>a</sup>
BPIC .....	+	+	-	+	-	3+
TAPB .....	+	-	+	+	-	3+ <sup>a</sup>
DCP .....	+	-	-	-	-	1+ <sup>a</sup>

(a) Blank cells indicate data not available.

**G**inger G. Myers is currently employed by Akzo Chemicals as the Research Manager for organic peroxides at their Research Center in Dobbs Ferry, NY. She previously held a position in research as Technical Development Manager, and positions in manufacturing as Process and Quality Control Manager and Analytical Chemist at Akzo's production facility in Pasadena, TX. Ms. Myers has Master of Science and Bachelor of Science Degrees in Chemistry from the University of Houston in Texas.

She is a member of New York Society for Coatings Technology, Society of Plastics Engineers, American Chemical Society, and the American Society for Quality Control.

also observed in the high-solids resins synthesis). Exceptions to this trend were TBPEH and TBPIN, which are alpha-branched peroxyesters. These both produced the highest molecular weight polymers (450,000) at polymerization temperatures of 94 and 115°C, respectively.

Two peroxides were tested at different concentrations: DTBTC (a difunctional perketal) and BPIC (a peroxy carbonate). Although the rates differed slightly at different concentrations, the overall conversions at two hours were the same. The molecular weight of DTBTC at the higher concentration was much lower than that at 8 meq/L (220,000 versus 350,000). This is due to a higher ratio of initiation/propagation at the higher initiator concentration, leading to a lower molecular weight polymer. The same phenomenon was observed with BPIC, to a lesser extent, in the comparison of 6 versus 8 meq/L concentrations.

Finally, Appendix 2 contains graphs comparing five initiators at 80°C. We selected three high conversion initiators (TBPEH, BPO, and AMBN), and two low conversion initiators, (DTBTC and TAPA), to compare performance at a lower temperature. The three high conversion products still gave the highest conversion (all >99% after four hours). DTBTC and TAPA produced slightly higher conversion at this temperature. However, M<sub>w</sub> for these two initiators was extremely high, 2.5 million. This is due to crosslinking at the lower temperature for these peroxides. Initiator selection should be carefully matched with the polymerization temperature to avoid undesired crosslinking of the polymer.

TBPEH, BPO, and DTBTC were also evaluated to compare conversion at different temperatures. Again, it was observed that identical conversions are obtained at the lower (80°C) temperature after four hours. Molecular weights were significantly higher (2X) for all three peroxides at the lower temperature, due to higher crosslinking potential. From these results, it can be concluded that final conversion is not affected by temperature. However, conversion rate and M<sub>w</sub> are dramatically different at different temperatures.

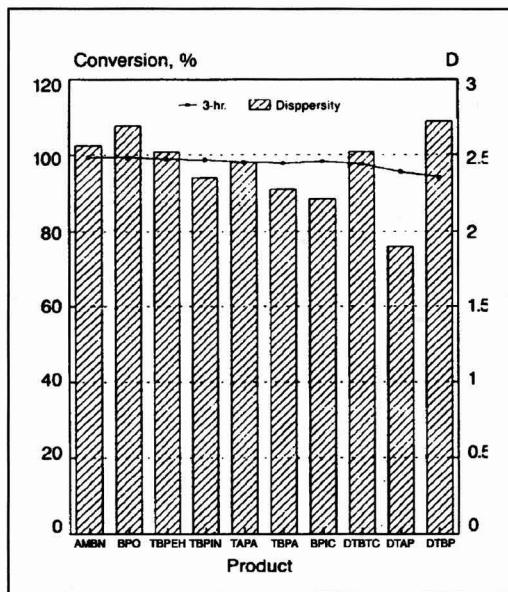


Figure 2—PMMA solvent conversion polydispersity vs conversion.

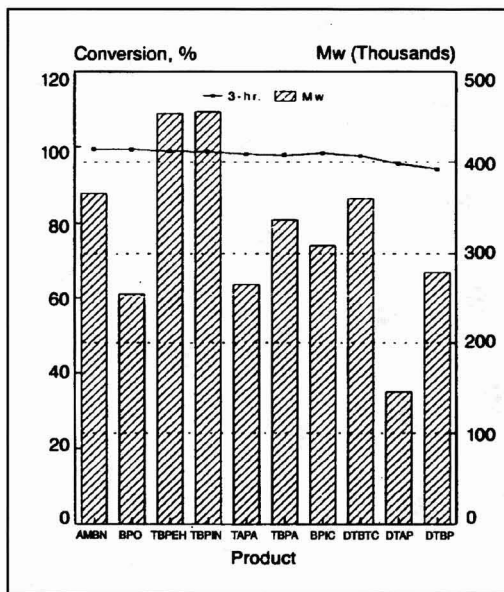


Figure 3—PMMA solvent conversion molecular weight vs conversion.

**Test Series III: Initiator Performance in a High-Solids Model System:** As a final aide in selecting an initiator for high-solids resins, we have tested a large number of initiators in our high-solids model system. The system is set up as previously described. Dose time is four hours followed by a reaction time of one hour. Table 5 lists all of the initiators tested and the corresponding polymer characterization and solids contents. (Theoretical solids = 71%.)

Appendix 3 and Table 5 show results on the initiators tested. Conversions are based on solids content measured in the final resin. No significant deviations were observed for conversion. The initiators were tested using two different solvents: Exxate 700 and butyl acetate. The resins made in butyl acetate have much lower viscosity (see Appendix 3). We also observed that DTBTC gave much higher  $M_w$  and viscosity results than all of the other initiators tested. This again is indicative of higher crosslinking efficiency for this perketal.

TBPA gave favorable results. Resins produced using this initiator had high  $M_w$  (12,100) with low viscosity (240 Poise). TBPEH and TBPIN gave very similar results in  $M_w$  (approx. 20,000) and viscosity (750-950 Poise). Tert-amyls tested gave slightly better results than the tert-butyl analogues; however, not enough to offset the price differential (see Table 3).

## CONCLUSIONS

In addition to conclusions already given in the body of this paper, we have summarized our results on the three test series performed on various initiators. Table 6 shows the rating of the various resin characteristics and cost efficiencies of all initiators tested. The following rating scheme is used:

Ratings	+	-
$M_w$ .....	>10,000	≤10,000
D .....	≤2	>2
V .....	≤800P	>800P
Cost .....	≤\$.70/%a.o	>\$.70/%a.o
Conversion .....	≥98%	≤98%

This table can be used as an initial screening method in developing or optimizing a high-solids resin system. The results discussed in this paper should be used on a relative basis, since there are many other contributing factors to the final resin quality (i.e., solvents, additive package, monomer reactivities, etc.).

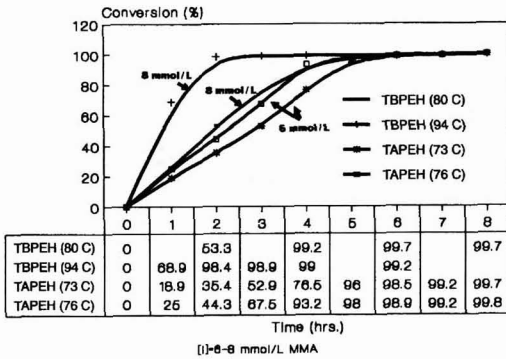
## ACKNOWLEDGMENTS

I would like to thank Kai-Yun Wang and Ruth Gallagher of Akzo Nobel Chemicals Inc. for their contributions to the experimental portion of this paper.

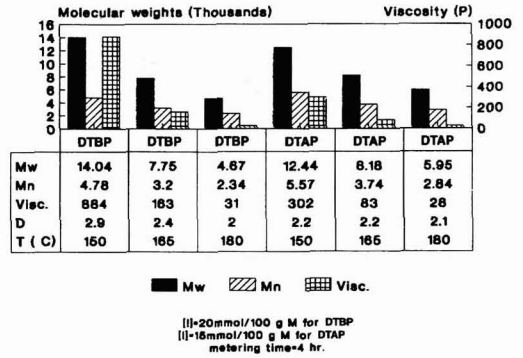
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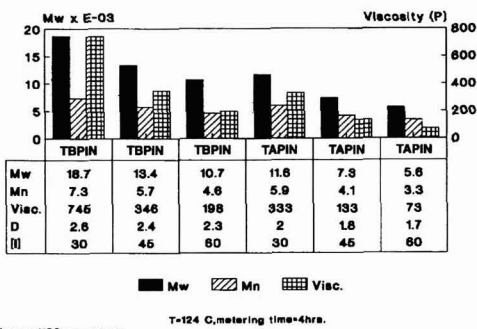
## Appendix 1



Appendix 1a—MMA solvent polymerization  
Solvent: toluene, MMA conc.: 65% wt.

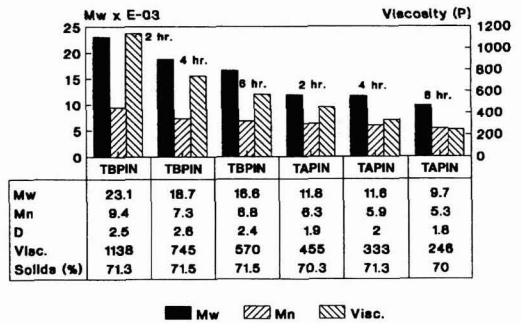


Appendix 1b—Influence of T on MWD and viscosity.

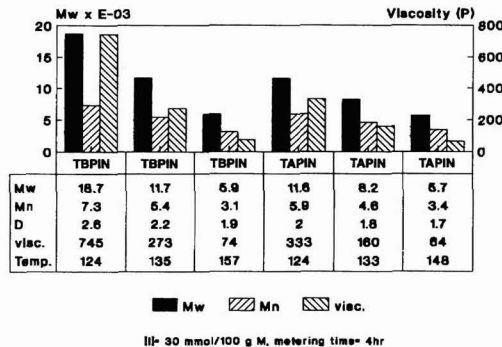


III in meq/100g monomer

Appendix 1c, Graph 1—Influence of (I) on MWD and viscosity.



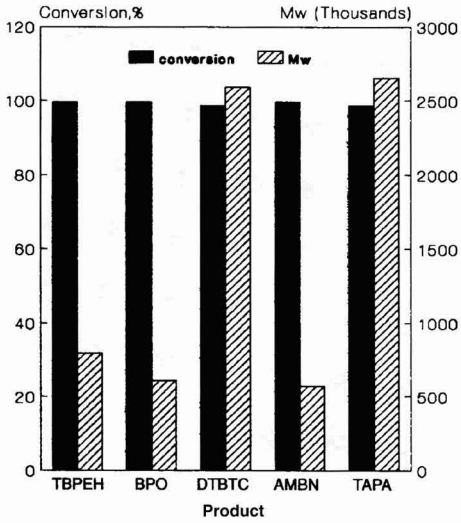
Appendix 1c, Graph 2—Influence of metering time on MWD and viscosity.



Appendix 1c, Graph 3—Influence of T on MWD and viscosity.



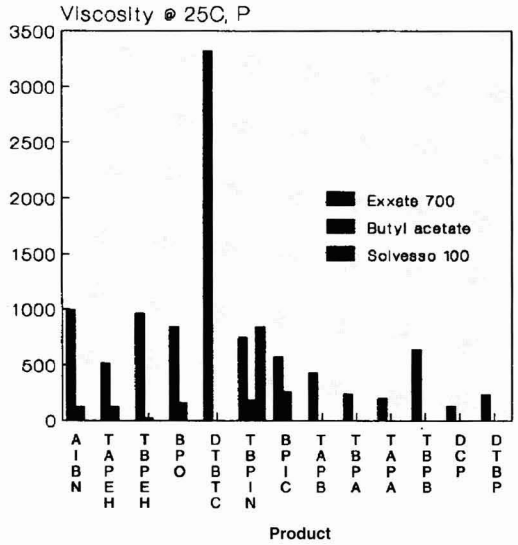
Appendix 2



[I] = 8meq/L MMA, 65% solids

Appendix 2—Initiator comparison. PMMA solvent polymerization, T = 80°C.

Appendix 3



Refer to Table 5 for more results.

Appendix 3—High-solids initiators. Viscosity comparison.

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# Novel Organosilane Crosslinking Agents for Powder Coatings

S.F. Thames, K.G. Panjani, S.D. Pace, M.D. Blanton, and B.R. Cumberland —  
University of Southern Mississippi\*

## INTRODUCTION

**P**owder coatings have traditionally lacked ideal flow and leveling, and it was our intent to investigate leveling as a function of silane content. Since powder coatings are deposited as a solid, the factors affecting flow during bake are very different from conventional coatings. According to De Lange, unbaked powders are not a continuous layer, but instead consist of particles whose major dimensions are on the order of the final coating thickness. Further, De Lange postulates that film formation in powder coatings consists of two phases: (1) coalescence of the molten powder on baking, followed by (2) flow of the film to a smooth coating, the efficiency of which is controlled by surface tension and its effects on flow, leveling, and viscosity.<sup>1</sup>

Silicon-containing polymers have been used widely in industry to achieve proper flow, leveling, exterior durability, electrical resistance,<sup>2</sup> corrosion resistance, and weather resistance.<sup>3</sup> Structurally, most silicon-containing materials have one or more Si-O bonds and are therefore classified as siloxanes.

Organosilane polymers, on the other hand, contain a hydrolytically stable Si-C bond which imparts property enhancements normally associated with silicone modification, i.e., increased thermal stability, improved solubility, enhanced processibility, and better weatherability.<sup>4-7</sup> Thames and Patel reported the synthesis, characterization, and evaluation of powder coatings containing organosilane diacids and anhydrides as crosslinking agents. These coatings exhibited improved gloss, gloss retention, flow, and leveling.<sup>8</sup> Later, Thames et al. demonstrated that silane incorporation lowered glass transition temperatures, enhanced thermal stability, and improved solubility in organic solvents.<sup>9,10</sup> Their work focused on Si-C bonds as a component of the polymer backbone structure. This investigation considers the influence of pendent organosilane substituents on powder properties.

## EXPERIMENTAL

### Materials

All resins, reagents, and additives, with the exception of the organosilane crosslinking agents, were used as received. The

*Organosilane dicarboxylic acids have been substituted for a portion of the carboxyl-terminated polyesters in conventional epoxy-polyester powder coatings and provide improvements in a number of performance characteristics. For instance, environmental scanning electron microscopy (ESEM) studies confirm improvements in flow and leveling; the modified powders permit lower cure temperatures; and chemical and weather resistance properties are improved as is their thermal stability. Adhesion and pencil hardness are essentially unchanged, but minor improvements are noted in some formulations. However, impact strength and flexibility are lowered for most silane-containing formulations, probably a consequence of higher aromatic content in the silane diacids.*

epoxy resins, Epon® 2002 and Epon® 1001F, were provided by Shell Chemical Co. The carboxyl terminated polyester, Cargill 30-3051, was supplied by Cargill, Inc. Modarez MFP and Actiron CC-6 were obtained from Synthron, Inc., while benzoin was obtained from Generichem. Titanium dioxide, Ti Pure® R-960 and sparmitte barytes are products of E.I. duPont de Nemours & Co. and Pfizer, Inc., respectively.

The monosubstituted pendent organosilane dicarboxylic acids, 2-trimethylsilyl terephthalic acid, 2-dimethylphenylsilyl terephthalic acid, 2-diphenylmethylsilyl terephthalic acid, and 2-triphenylsilyl terephthalic acid were synthesized according to Scheme 1 (see page 44). The disubstituted pendent organosilane dicarboxylic acids, 2,5-di (trimethylsilyl) terephthalic acid, 2,5-di (dimethylphenylsilyl) terephthalic acid, 2,5-di (diphenyl-methylsilyl) terephthalic acid, and 2,5-di (triphenylsilyl) terephthalic acid were synthesized according

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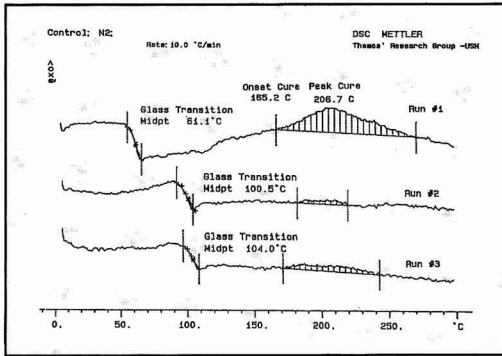


Figure 1a—DSC thermograms of control formulation.

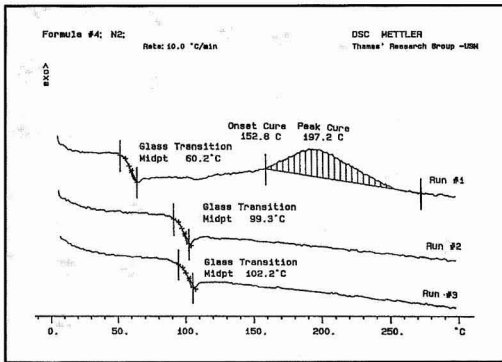


Figure 1b—DSC thermograms of formulation 4.

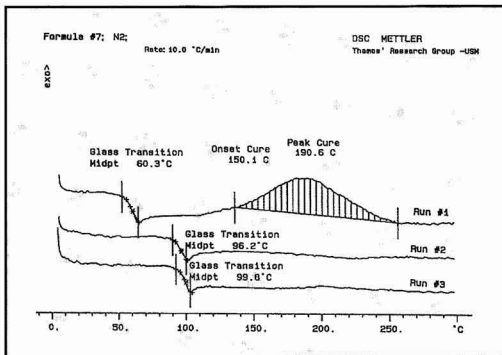


Figure 1c—DSC thermograms of formulation 7.

Table 1—Typical Powder Formulation

Resins selected:	Epon® 2002 and Epon® 1001F
Crosslinking agents selected:	a) Cargill 30-3051 b) Cargill 30-3051 and organosilane diacids (90:10 ratio by equivalent)
Pigment:	Ti Pure® R-960 ..... 60.0 phr
Extender:	Sparmite barytes ..... 35.8 phr
Flow additive:	Modarez MFP ..... 2.4 phr
Degasser:	Benzoin ..... 1.6 phr
Catalyst:	Actiron CC-6 ..... 0.2 phr
Stoichiometry: carboxyl/epoxy equivalent ratio = 1.00.	

to Scheme 2. All organosilane intermediates and dicarboxylic acids were characterized via Fourier transform infrared spectroscopy (FTIR), nuclear magnetic resonance (NMR) spectroscopy, and elemental analysis.<sup>11</sup>

### Synthesis of Monosubstituted Organosilane Dicarboxylic Acids — Scheme 1

10 M *n*-butyl lithium in hexanes (10% molar excess) was added to a solution of 2-bromo-*p*-xylene in anhydrous ether at 0°C under nitrogen. After one hour, the desired chlorosilane (10% molar excess) was added dropwise, lithium chloride was subsequently removed by filtration, and the solvents were removed *in vacuo*. The intermediates formed were purified by vacuum distillation or recrystallization followed by oxidation with KMnO<sub>4</sub> in a water/pyridine mixture to give the respective monosubstituted organosilane dicarboxylic acids.

### Synthesis of Disubstituted Organosilane Dicarboxylic Acids—Scheme 2

10 M *n*-butyl lithium in hexanes (10% molar excess) was added to a solution of 2,5-dibromo-*p*-xylene in anhydrous ether at 15–20°C under nitrogen. After one hour, the desired chlorosilane (10% molar excess) was added dropwise. The process of metalation and addition of chlorosilane was completed one more time to give the disubstituted product. The intermediates formed were purified by recrystallization and subsequently oxidized with KMnO<sub>4</sub> in a water/pyridine mixture to the respective disubstituted organosilane dicarboxylic acids.

### Powder Coating Formulation

A control powder coating was developed from epoxy resins, Epon 2002 (equivalent weight 675–760 g), Epon 1001F (equivalent weight 525–550 g), and Cargill 30-3051 (equivalent weight 561–660 g), a carboxyl-terminated polyester (Table 1). The structures of monosubstituted and disubstituted silane diacids used for the preparation of silane containing powders are included in Tables 2 and 3, respectively. The carboxyl to epoxy equivalent ratio for all formulations was maintained at 1.00 and all other formulation parameters were held constant.

Individual powder batches were prepared by premixing the ingredients in a Henschel FM-10 mixer followed by melt compounding in a Werner-Pfleiderer ZSK-30 twin-screw ex-

**Table 2—Monosubstituted Organosilane Crosslinking Agents Used in Powder Coatings Formulations**

Formulation	Organosilane Monomer	Structure
Control	—	—
2	2-trimethylsilyl terephthalic acid	
3	2-dimethylphenylsilyl terephthalic acid	
4	2-diphenylmethylsilyl terephthalic acid	
5	2-triphenylsilyl terephthalic acid	

**Table 3—Disubstituted Organosilane Crosslinking Agents Used in Powder Coatings Formulations**

Formulation	Organosilane Monomer	Structure
Control	—	—
6	2,5-di (trimethylsilyl) terephthalic acid	
7	2,5-di (dimethylphenylsilyl) terephthalic acid	
8	2,5-di (diphenylmethylsilyl) terephthalic acid	
9	2,5-di (triphenylsilyl) terephthalic acid	

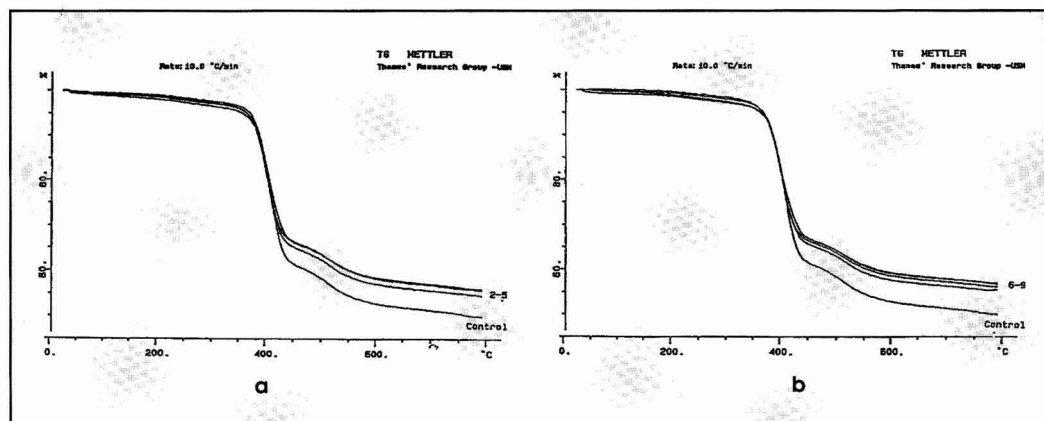


Figure 2—Comparative thermogravimetric analysis data of (a) control and formulations 2-5; and (b) control and formulations 6-9.

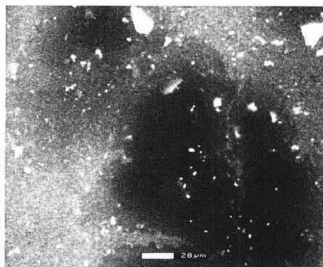


Figure 3a—ESEM photomicrograph of control formulation.

Figure 3b—ESEM photomicrograph of formulation 4.

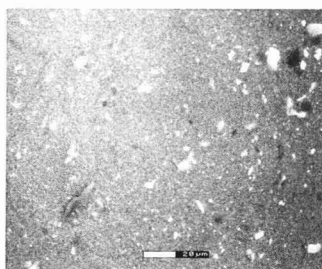
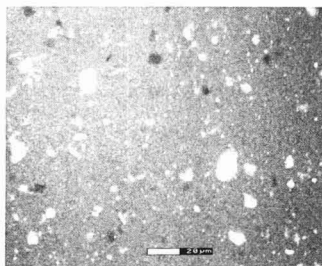


Figure 3c—ESEM photomicrograph of formulation 7.

truder with the extruded mass being cooled via a chill roll. The cooled chips were pulverized in a micro-pulverizer hammer mill and sieved through a 105  $\mu\text{m}$  screen with a Kem-U-Tec centrifugal sifter. All powder coatings were applied by corona electrostatic spray technique onto 3 in. X 6 in. X 0.02 in. cold rolled, low-carbon, mild steel (QD-36) panels which were previously wiped with acetone, MEK, and methylene chloride to remove oil, grease, and other impurities. The coatings were cured at 150°C for 30 min.

### Testing Equipment

Particle size analysis was performed with a Leeds & Northrup Microtrac Model 9200-2-00-1 standard range particle size analyzer. Electron microscopy studies were conducted with an electro scan type II environmental scanning electron microscope (ESEM).

Differential scanning calorimetry was performed with a Mettler DSC 30 measuring cell where the heating and/or cooling sequence between 0-300°C was set at 10°C/min. Each powder was subjected to three heating and cooling cycles. Thermogravimetric analysis was performed on a Mettler TG 50 analyzer via heating the samples to 800°C at 10°C/min in  $\text{Al}_2\text{O}_3$  crucibles.

Corrosion resistance was evaluated by placing coated panels in a Q-fog prohesion chamber, Model SF450 at 35°C for 500 hr. Ultraviolet stability was measured using an Atlas Model UC-1 ultraviolet-condensation screening device. Panels were exposed to alternating ultraviolet/condensation cycles at 60°C for 410 hr. After 410 hr, the extent of yellowing was measured with an ACS colorimeter.

Impact resistance was determined with a BYK-Gardner impact tester, using a 1.82 kg, 1.27 cm 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 cross-hatch constructed by eleven cutting blades. Flexibility was measured by bending coated panels on a Paul N. Gardner Model MN-CM conical mandrel. Stain resistance was performed via an in-house test in which lines were drawn with red, blue, and black solvent-based ink markers. Stain resistance was measured 10 days after marking by visual examination

of the amount of stain retained when the marks were wiped with xylene soaked gauze.

## RESULTS AND DISCUSSION

The average particle size fell in the range of  $49 \pm 3 \mu\text{m}$  with the exception of formulations 8 and 9, which were found to be 34.9  $\mu\text{m}$  and 41.1  $\mu\text{m}$ , respectively. The particle size distribution for all powders ranged between 0-105  $\mu\text{m}$ .

Differential scanning calorimetry data are noted in Table 4 and Figures 1a-c. Increases in glass transition temperatures ( $T_g$ ) were observed for all samples after the second heating cycle. This is attributed to the polymer forming reaction via the epoxide ring opening with carboxyl terminated polyester/organosilane diacids. Further increases in  $T_g$  in the third heating cycle are attributed to annealing effects, and cur-



Table 4—Powder Analysis

Formulation/ Property	Control	2	3	4	5	6	7	8	9
Average particle size ( $\mu\text{m}$ ) <sup>a</sup>	46.8	50.1	46.7	52.0	46.5	46.0	47.6	34.9	41.1
Glass transition temperature ( $^{\circ}\text{C}$ )									
Run 1	61.1	60.0	58.3	60.2	59.6	59.8	60.3	59.6	58.6
Run 2	100.5	99.4	99.7	99.3	99.2	98.6	95.8	96.1	98.3
Run 3	104.0	104.1	102.2	102.2	102.1	102.9	99.8	100.6	100.7
Cure temperature ( $^{\circ}\text{C}$ )									
Onset	165.2	162.4	159.8	152.8	163.5	160.3	150.1	159.1	163.5
Peak	206.7	199.9	202.6	197.2	203.9	205.2	190.6	199.9	205.2
Thermogravimetric analysis % weight retained at $650^{\circ}\text{C}$	52.0	57.5	57.9	57.9	58.4	57.9	58.6	58.8	58.4

(a) Particle size analysis by ASTM Standard: D 3451.

Table 5—Weathering and Chemical Resistance of Cured Films

Formulation/ Property	Control	2	3	4	5	6	7	8	9
Initial Gloss									
$20^{\circ}$	41	48	47	47	44	53	30	31	47
$60^{\circ}$	81	85	86	83	85	88	70	77	82
$85^{\circ}$	79	85	87	87	87	90	74	83	90
UV resistance (410 hrs) <sup>a</sup>									
$\Delta E$	7.74	5.20	5.14	5.01	5.16	5.05	5.31	5.06	4.91
$\Delta L$	-1.12	-0.99	-1.03	-0.94	-0.85	-0.82	-0.96	-1.08	-0.93
$\Delta a$	-0.93	-0.48	-0.51	-0.51	-0.55	-0.53	-0.56	-0.52	-0.52
$\Delta b$	7.60	5.08	5.01	4.90	5.06	4.96	5.19	4.92	4.79
MEK double rubs	30	76	100+	100	100	100+	100+	100+	100+
Salt fog (500 hr) <sup>b</sup>	pitting	none	none	none	none	none	none	none	none
Stain resistance	stains	none	none	slight staining	none	none	slight staining	none	none

(a) UV resistance by ASTM Standard: D 2244.

(b) Corrosion resistance by ASTM Standard: B 117.

Table 6—Mechanical Properties of Cured Films

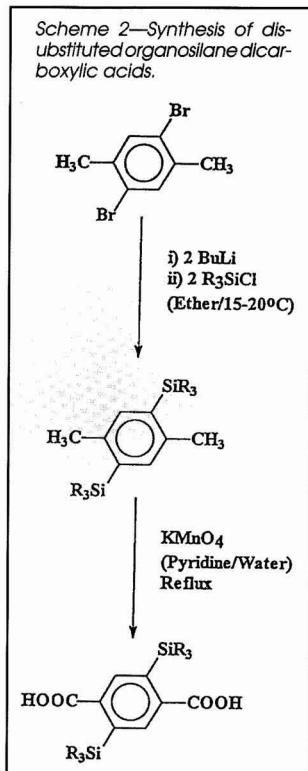
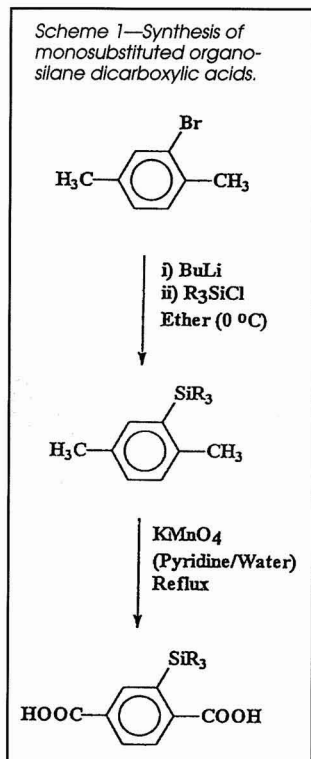
Formulation/ Property	Control	2	3	4	5	6	7	8	9
Impact resistance <sup>a</sup>									
Direct (joules)	13.6	4.5	4.5	13.6	2.3	3.4	15.9	2.3	<1.1
Indirect (joules)	5.7	<1.1	2.3	5.7	<1.1	<1.1	9.1	<1.1	<1.1
Flexibility ( $\text{cm}$ ) <sup>b</sup>	0.64	1.27	1.91	1.91	2.54	1.91	1.27	1.27	2.54
Pencil hardness <sup>c</sup>	3H	5H	3H	5H	3H	5H	3H	5H	3H
Adhesion <sup>d</sup>	2B	2B	3B	3B	2B	B	5B	2B	4B

(a) Impact resistance by ASTM Standard: D 2794.

(b) Flexibility by ASTM Standard: D 522.

(c) Pencil hardness by ASTM Standard: D 3363.

(d) Adhesion by ASTM Standard: D 3359 (0B = less than 35%, 5B = 100% adhesion).



The improvement in catalysis by (p-d)  $\pi$  bonding provides for an efficient ring opening reaction and thus a more effective and complete curing profile. For instance, unlike the nonsilicon counterpart, no curing exotherms were present during the second and third heating cycles of powders formulated with organosilane crosslinking agents (Figures 1a vs 1b-c).

Weight loss profiles are shown in Figure 2, while Table 4 includes a compilation of the weight loss data, including the percent weight of sample retained at 650°C. The thermograms in Figure 2 confirm the onset of thermal degradation of all powders to be in the range of 375°C although the organosilane based coatings retained 5-7% more weight than the control, a percentage which approximates the amount of organosilane diacids present at a 10% equivalent replacement.

Weathering resistance data (Table 5) indicate significant improvement in ultraviolet stability and a slight improvement in initial gloss in most of the silane-modified coatings. The ultraviolet stability data were obtained from  $\Delta E$  values determined via the ACS colorimeter. The color difference is attributed primarily to yellowing as indicated by the  $\Delta b$  values.

The xylene:MEK (90:10) double-rub solvent resistance and stain resistance (Table 5) were measured to determine the degree of crosslinking. The organosilane modified powder coatings showed solvent

resistance superior to the control, reinforcing the concept of more efficient cure when organosilane diacid crosslinking agents are part of the formulation.

rently is under investigation. Although  $T_g$  difference between most organosilane modified powder coatings and the control in heating cycles 2 and 3 is small, the  $T_g$  lowering is significant given the concentration levels of the organosilane diacids, and is consistent with prior published results.<sup>10</sup>

The onset cure temperature(s) of the organosilane-based powder coatings are lower than the control, and this feature holds potential for curing temperature reductions (Table 4). The acidity of the organosilane diacids (Tables 2 and 3) are enhanced via (p-d)  $\pi$  bonding and, as a result, increase the catalytic efficacy of epoxide ring opening.<sup>12</sup>

However, catalysis is influenced by steric hindrance of the ring substituents. For example, in case of the monosubstituted organosilane powder coatings, the onset curing temperature decreases in formulations 2-4. The triphenylsilyl moiety of formulation 5, however, hinders carboxyl attack of the epoxide ring, thereby diminishing the effect of (p-d)  $\pi$  bonding. Similar results are noted for the disubstituted organosilane powder coatings as (p-d)  $\pi$  lowers the onset curing temperature of formulations 6 and 7, while steric effects diminish the influence of (p-d)  $\pi$  bonding in formulations 7-9. However, the curing temperatures for all organosilane based formulations are lower than control, confirming that (p-d)  $\pi$  bonding is the predominating influence with regard to epoxide ring-opening reactions.

resistance superior to the control, reinforcing the concept of more efficient cure when organosilane diacid crosslinking agents are part of the formulation.

Salt spray analysis data (Table 5) confirm the significant contribution of silane addition, in that no corrosion was observed in the silane modified coatings, while the control panel was extensively corroded. Environmental scanning electron microscopy photomicrographs (Figure 3) clearly identify corrosion (Figure 3a) being initiated at sites of pinholes, or thin film areas, approaching 100  $\mu$ m in diameter. However, no such defects were observed in the organosilane modified powder coatings via ESEM photomicroscopy examination (Figures 3b-c).

Mechanical property evaluations are included in Table 6. Adhesion and pencil hardness were similar for all coatings with slight improvements noted for select organosilane modified coatings. Flexibility and impact resistance were reduced with the existing silane modifications, a feature which can be attributed to more extensive and shorter crosslinking segments when the lower molecular weight organosilane curing agents are used. Therefore, current efforts are focused to optimize organosilane monomers/polyester ratio, and synthesize flexible, carboxyl-terminated pendent organosilane polyesters to improve mechanical properties.

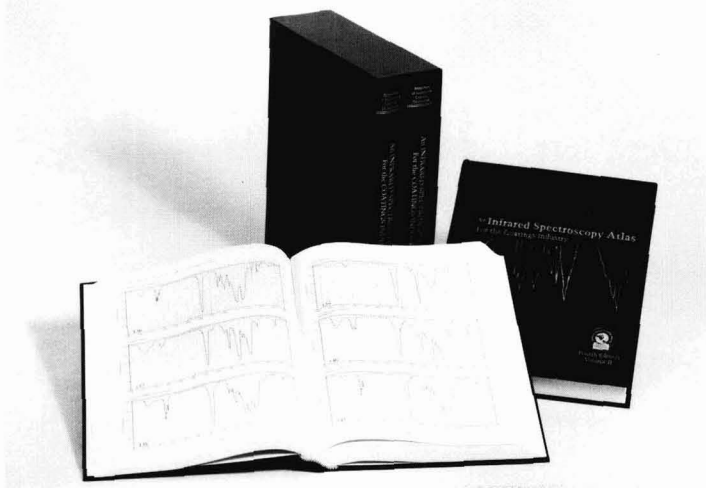
## CONCLUSIONS

Novel organosilane curing agents have been synthesized and formulated into several epoxy-polyester powder coatings. In most instances, the organosilane modified powder coatings were superior to the control. Specifically, organosilane modification provided improved flow, leveling, thermal stability, chemical resistance, weather resistance, and faster and more efficient cure. Adhesion and pencil hardness were similar; yet, impact strength and flexibility were lower in most formulations. Optimization of organosilane dicarboxylic acid/polyester ratio and synthesis of flexible, carboxyl-terminated organosilane based polyester are currently in progress to improve the mechanical properties of these powder coatings.

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# Styrenation of Oils Based on Secondary Esters of Castor Oil

O.S. Kabasakal, F.S. Güner, A.T. Erciyes, and Y. Yagci\*—Istanbul Technical University†

## INTRODUCTION

Drying and semidrying oils are used extensively in the manufacture of oil-based binders. Since these natural products are somewhat deficient in their film properties, various methods have been used for their modification. The well-known method is the copolymerization of oils with vinyl monomers, such as styrene. Recently, styrene-oil copolymerization has been treated in a review article by Saxena et al., who emphasized the reaction mechanism, preparation techniques, raw materials, analysis of the products, and their use.<sup>1,2</sup> Oils used in the styrenation process include linseed, tung, soybean, oticica, sunflower, olive, sardine, rubberseed, tobacco seed, and nigerseed oils.<sup>2-19</sup> Another widely used oil is the dehydrated castor oil (DCO) which is synthetic oil obtained by dehydration of castor oil.<sup>4-6,10,11,20-24</sup> DCO can be prepared by splitting secondary esters of castor oil.<sup>25-29</sup> In our previous study,<sup>30</sup> we reported on the synthesis of triglycerides with drying oil properties from the splitting mixture of secondary esters of castor oil. The splitting mixture was also used in the preparation of oil modified alkyd resin.<sup>31</sup> As a part of our continued interest, the preparation of styrenated oils based on secondary esters of castor oil is described.

This study shows the results, specifically, the outline of the synthetic strategy and the results of a representative series of experiments on the film properties of oil-styrene copolymer.

## EXPERIMENTAL

**Materials:** Commercially purchased linseed oil, sunflower oil, and dehydrated castor oil (DCO) were used. Table 1 presents the main characteristics of these oils. Mixed acids of linseed and sunflower oils were prepared by refluxing the corresponding triglyceride with ethanolic potassium hydroxide followed by acidification and extraction with ether. 4,4'-azobis(4-cyanopentanoic acid) (ACPA), analytical grade from Fluka, was used as received in the preparation of the corresponding acid chlorides (ACPC) by means of phosphorus pentachloride as described previously.<sup>36</sup> Styrene and solvents were purified according to conventional drying and distillation procedures.<sup>37</sup>

**Preparation of Castor Oil Esters and Their Splitting:** Castor oil and mixed fatty acids of given oil, in equivalent proportion, and p-toluenesulfonic acid (0.1% wt. of fatty

*The synthesis of styrenated oils based on secondary esters of castor oil is described. The steps of the process are as follows: (1) esterification of castor oil with fatty acids of drying and semidrying oils; (2) splitting of castor oil esters; and (3) conversion of the splitting mixture to either triglyceride or partial glyceride. While the triglyceride samples were styrenated by a conventional process, the partial glycerides' thermally labile azo groups were initially inserted into the oil molecule, and then this intermediate was reacted with styrene. The obtained styrenated oils showed good film properties.*

acids) were placed in a four-necked reaction flask equipped with a thermometer, an inert gas (nitrogen) inlet tube, a mechanical stirrer, and an inclined air condenser. The mixture was heated to 225°C under nitrogen flow and heating was continued for 2.5 hr at this temperature. The acid value of the ester product was determined.<sup>32</sup> Castor oil esters thus obtained were split at 280°C for 2 hr under inert gas flow as described previously.<sup>30</sup> Since the esterification catalyst, i.e., p-toluenesulfonic acid, was not removed, it served as a splitting catalyst as well. Conjugated diene and free fatty acid contents of the splitting mixture were determined. To determine the content of conjugated diene, ultraviolet absorption analysis was applied with a Beckman (München, Germany) UV spectrophotometer Model DB-GT.<sup>35</sup>

**Preparation of Tri- and Partial Glycerides:** In the case of triglyceride manufacture, glycerol, in an equivalent amount to the free fatty acids, was added to the splitting mixture and the content was heated. When the temperature reached 218°C, 0.1% wt. of calcium hydroxide was added and the temperature raised to 232°C. Reaction was conducted at this temperature for 45 min. To obtain partial glycerides, the same procedure

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**Table 1—Some Properties of Oil Specimens Used in the Study<sup>a</sup>**

Oil	Refractive Index, n <sub>D</sub> <sup>20</sup>	Acid value	Saponification value	Iodine Value (Hanus) <sup>b</sup>	Hydroxyl Value	Conjugated di-enoic acid <sup>d</sup> %
Sunflower oil .....	1.4750	0.7	186	120	—	—
Linseed oil .....	1.4811	0.9	191	174.6	—	—
Castor oil .....	1.4780	3.3	176.7	83.7	165	—
DCO .....	1.4839	3.2	189.3	156 <sup>c</sup>	19.1	26.1

- (a) Analytical methods were taken from reference (32) unless otherwise stated.  
 (b) Reference (33).  
 (c) Reference (34).  
 (d) Reference (35).

was applied by using 8.5% of glycerol based on the splitting mixture. Acid and hydroxyl values of the resulting partial glycerides were determined.<sup>32</sup>

**Styrenation of Tri- and Partial Glycerides:** Synthetic triglycerides as well as linseed oil and DCO were styrenated by a conventional process using benzoyl peroxide as an initiator.<sup>38</sup> Partial glycerides were initially reacted with ACPC. Thus, 30 g of ACPC in 60 ml of dichloromethane was added dropwise to a mixture of the equivalent amount of partial

(10% in volume with respect to styrene), when stated. After eight hours, the unreacted monomer was removed by vacuum distillation until a clear solid was obtained. The degree of conversion of styrene was calculated by weight increase after precipitation of the mixture into methanol. The viscosities of styrenated oil samples were determined.

**Determination of Film Properties:** Film properties, such as drying time,<sup>39</sup> flexibility,<sup>40</sup> adhesion,<sup>41</sup> water resistance,<sup>42</sup> alkali resistance,<sup>42</sup> and acid resistance,<sup>42</sup> were determined by using the samples thinned with xylene. The solid contents were 60% and 40% for the samples based on linseed and sunflower oils' mixed acids, respectively. Lead naphthenate (0.5%) and cobalt naphthenate (0.05%) as metal based on solid content were added. Dryers were added 24 hr before film application. In drying time determinations, a Bird film applicator with 40 μm aperture was used for film application on glass plate; dry film thickness was found to be 16-18.2 μm. For the flexibility, adhesion, and water resistance tests, tin plate panels were used as a substrate, while glass tubes were used for alkali and acid resistance tests, as explained in the related standard methods. A water resistance test was performed 48 hr after the film application. However, this aging period was 72 hr for other tests.

**Table 2—Some Properties of Secondary Esters and Corresponding Splitting Mixtures**

Property	Secondary Ester Based on:		Splitting Mixture of Secondary Ester Based on:	
	LOFA	SOFA	LOFA	SOFA
Acid value .....	18.6	31.4	79.4	75.7
Dienoic acid, % .....	—	—	17.2	23

- Legend: LOFA = Linseed oil fatty acids.  
 SOFA = Sunflower oil fatty acids.

glycerides, as determined by hydroxyl value and pyridine at 0°C. After 30 min the temperature was raised to 35°C and kept constant while stirring for 80 hr. The reaction mixture was then dissolved in diethyl ether and washed successively with 0.1% aqueous sodium hydroxide solution and water and finally dried over sodium sulfate. After removing the solvent, the product (ACPC-PG) was characterized by IR spectroscopy. For this purpose, nitrile absorption was observed at 2200 cm<sup>-1</sup> wavenumber. Additionally, the hydroxyl value was determined.

Prior to heating using a constant temperature bath at 70°C, nitrogen was bubbled through the mixture of oil having azo groups (30 g) and styrene (30 g) and allyl diethyl malonate

## RESULTS AND DISCUSSION

Figure 1 outlines the strategy used for the styrenation process. The process starts by esterification of secondary hydroxyl groups followed by thermal splitting. Details of these reactions have been discussed extensively elsewhere.<sup>27</sup> Acids used in this dehydration include acetic acid, 18-carbon fatty acids, phthalic anhydride, and rosin. Since the acetic acid is a volatile material, it is removed easily by distillation, leaving a residue of DCO.<sup>28</sup> However, when less volatile acids are used, they remain in the splitting mixture; therefore, vacuum distillation is used to remove the free acids.<sup>26</sup>

Since the main objective is to prepare styrenated oil, the next step is the conversion of the splitting mixture directly to triglyceride or partial glyceride by using an equivalent or excess amount of glycerol, respectively. Table 2 presents the acid values and diene contents of the

**Table 3—Acid and Hydroxyl Values of Synthetic Tri- and Partial Glycerides and ACPC-PG intermediates**

Property	Triglyceride from splitting mixture of:		Partial glyceride from splitting mixture of:		ACPC-PG based on:	
	LOFAE	SOFAE	LOFAE	SOFAE	LOFAE	SOFAE
Acid value .....	25.82	24.5	7.2	10.1	7.8	9.5
Hydroxyl value .....	—	—	103	115	39	23

- Legend: LOFAE= Linseed oil fatty acids esters.  
 SOFAE= Sunflower oil fatty acids esters.

castor oil esters and corresponding splitting mixtures. The diene content of splitting mixture based on sunflower oil fatty acids esters is higher than the linseed oil fatty acids case. This is probably due to relatively favorable reactions of conjugated double bonds by thermal polymerization in the presence of high conjugation.

The subsequent event is the attachment of thermolabile azo groups to partial glycerides in a manner similar to that described for drying and semidrying oils.<sup>36</sup> In Table 3, acid and hydroxyl values of tri- and partial glycerides are given together with those of (ACPC-PG) intermediates. As shown, free acid contents of the condensation products fell to a reasonable level under the applied reaction conditions. Notably, hydroxyl values of partial glycerides decreased from about 100 to 30, indicating successful incorporation of ACPC.

The oil-styrene copolymer structure was assigned by means of IR measurements. The spectra of the copolymer samples show the characteristic bands of both styrene and initial oil segments (Figure 2). Viscosities and film properties of the samples are given in Tables 4 and 5, respectively. Sunflower oil fatty acids esters-based samples have higher viscosities than those based on linseed oil acids esters. This is because of the higher content of the inserted azo groups due to higher hydroxyl content of the corresponding partial glycerides mixture. Moreover, the chain length of the polystyrene segment was regulated in the former case by performing styrenation experiments in the presence of diethyl allyl malonate as a degradative chain transfer agent. On the contrary, a signifi-

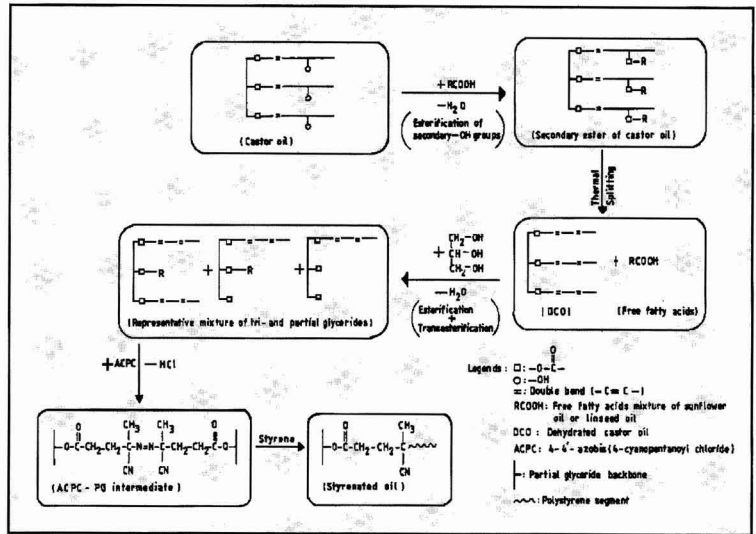


Figure 1—Preparation stages of the styrenation process.

cant viscosity increase was observed in the latter case under the same reaction conditions. Again, this increase seems due to the difference in the azo content. Only some of the propagating radical sites were terminated by allyl malonate, while the others give rise to the formation of longer chains.

As shown in Table 5, film properties of the styrenated oils via the proposed method show much better alkali resistance

Table 4—Viscosities (St, 25°C) of Styrenated Oil Samples<sup>a</sup>

Styrenated oil samples based on:	Samples from the Reaction:	
	Without Allyl Malonate	With Allyl Malonate
Synthetic triglyceride from splitting mixture of		
LOFAE .....	1.65	—
SOFAE .....	2.5	—
ACPC-PG intermediate based on splitting mixture of		
LOFAE .....	63.4	54.85
SOFAE .....	123.3 <sup>b</sup>	148 <sup>b</sup>
Linseed oil by conventional process ...	0.85	—
DCO by conventional process .....	0.6	—

(a) Samples were thinned with xylene to 60% solid content.  
 (b) Samples were thinned with xylene to 55% solid content.

Legend: LOFAE = Linseed oil fatty acids esters.  
 SOFAE = Sunflower oil fatty acids esters.

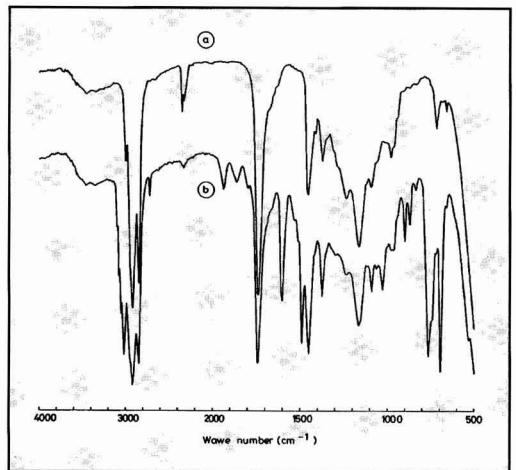


Figure 2—Infrared spectra of ACPC-PG intermediate based on sunflower oil fatty acids esters (a) and the corresponding styrenated oil (b).

Table 5—Film Properties of Styrenated Oils Prepared by the Proposed Process<sup>a</sup>

Styrenated Sample Based on	Drying time <sup>b</sup> (Set-to-Touch)	Adhesion <sup>c</sup>	Flexibility <sup>d</sup>	Water Resistance	Alkali Resistance <sup>e</sup>	Acid Resistance <sup>f</sup>
Synthetic triglyceride from splitting mixture of						
LOFAE .....	2 m	5B	2 mm	10 m	420 m	nc
SOFAE .....	1.5 m	5B	2 mm	75 m	1950 m	nc
ACPC-PG intermediate from spitting mixture of						
LOFAE .....	1 m (1.5 m)	5B (5B)	2 mm (2 mm)	61 m (59 m)	310 m (1950 m)	nc (nc)
SOFAE .....	1.5 m (2 m)	5B (5B)	2 mm (2 mm)	6 m (6 m)	nc:2820 m (nc:2820 m)	nc (nc)
Linseed oil by conventional method .....	2.5 m	5B	2 mm	5 m	60 m	nc
DCO by conventional method .....	3 m	5B	2 mm	nc	nc	nc

(a) Values in parentheses belong to samples prepared in the presence of allyl malonate.

(b) Test was carried out at 25°C and 60% relative humidity.

(c) Test Method B was applied.

(d) Diameter of cylinder which caused no crack on the film.

(e) Test was carried out at 25°C and 5% NaOH solution.

(f) Test was carried out at 25°C and 9% NaOH solution.

Legend: m = minutes, mm = millimeter, nc = no change.

LOFAE = linseed oil fatty acids esters.

SOFAE = sunflower oil fatty acids esters.

and dry as fast as the styrenated linseed oil and DCO samples obtained by the classical method. It is well-known that in conventional styrenation, polystyrene segments are attached to the fatty acid chains and are expected to be far from the ester groups of glycerides. However, in the proposed process shown in Figure 1, polystyrene segments are combined with the oil molecule through the radicals formed by decomposition of azo groups. Therefore, polystyrene molecules linked this way are closer to glyceryl moiety and more likely to prevent the diffusion of alkali to hydrolyze the ester groups of glycerides.

It is well-known that sunflower oil is a semi-drying oil and is consumed largely for edible purposes. In alkali refining of this oil, a large amount of soapstock is obtained. This by-product, as well as other semi-drying oils, may be the source of the mixed fatty acids to be evaluated in the process. Additionally, the proposed process eliminates the problem of removing the catalyst and liberated fatty acids encountered in catalytic and splitting methods of DCO manufacture. No doubt that these features reduce processing costs. In some cases, the mixture of DCO and a semi-drying oil is recommended as the oil component for the alkyd resin to be used for special purposes. For instance, dilution of DCO with soybean oil was proposed for the phthalic alkyds suitable for post styrenation.<sup>43</sup> Since the free acids of the splitting mixture were not removed in the proposed process, the oil component of the styrenated product can be considered as a mixture of DCO and sunflower or other oils used as a fatty acids source.

## CONCLUSION

This study demonstrated that styrenation of oils based on secondary esters of castor oil, which directly incorporates azo initiator, results in a considerable advantage over classical styrenation regarding film properties.

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# VOC Testing Comparison: EPA Method 24 Versus the Cal Poly Method

Los Angeles Society for Coatings Technology Technical Committee<sup>1</sup>—  
V.C. Bud Jenkins, Joseph C. Reilly, Bob Sypowicz, and Max T. Wills\*

## Introduction

Presently, the experimental determination of the volatile organic compound (VOC) content of water-based paints is of major concern to the coatings industry. The South Coast Air Quality Management District (SCAQMD), in California, have caused the concern since their regulatory efforts require paint manufacturers to continue lowering the levels of these substances. The existing methods for carrying out the VOC determination may no longer be adequate to carry out the determination with sufficient precision on the newer coatings systems that are being developed. These methods are contained in ASTM Practice D 3960<sup>2</sup> (EPA Reference Method 24<sup>3</sup>) and require, as a minimum, the measurement of paint density, total volatile content, and water content. The ASTM D01.21 committee's Task Group 56 is involved with revision efforts of D 3960 and has discussed the constant effort required to bring the practice up-to-date.<sup>4</sup>

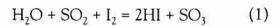
The Technical Committee of the Philadelphia Society for Coatings Technology recently conducted a "round-robin" study of ASTM D 2369, the method used to determine the total volatile content in coatings on single-component and two-component high-solids, solvent-based coatings to test the suitability of the method for these systems. They found that single-component coatings gave reliable results and that two-component coatings yielded higher results than expected with large standard deviations. The Philadelphia Society points out that water-based coatings also introduce the additional problem of water determination.<sup>5</sup>

The ASTM D01.21 Committee Task Group 24 is actively working to ascertain the suitability of ASTM D 2369 and is planning a round-robin study of high water/low VOC content coatings to answer criticisms the method has received with regard to these

coatings types. Task Group D01.21.24 has elected to address the problems associated with multi-component systems at a later date.<sup>4</sup>

Water is determined by gas chromatography (GC) using ASTM Method D 3792<sup>6</sup> or by a Karl Fischer titration (KF) using ASTM Method D 4017.<sup>7</sup> Round-robin data collected in 1986<sup>8</sup> indicate that the GC method gives better precision than the KF method, but it is generally acknowledged that it is technically more difficult to perform. Accordingly, the Karl Fischer method is often the method of choice for water determination despite its lower precision. ASTM's D01.21 Task Group 54 is responsible for revisions of the Karl Fischer method and plans to add a methanol extraction

either visually or electrometrically. The reagent combines rather slowly with water and has an unpleasant odor. New pyridine-free Karl Fischer reagents substitute various proprietary amines for pyridine and glycol ethers for methanol, which has led to increases in speed and accuracy and elimination of the objectionable odor of pyridine. The basic reaction of the KF reagent with water is given in equation (1):



When water in paint and related paint materials is determined by ASTM Method D 4017, the neat sample is weighed, by difference, directly into pyridine which has been dried with Karl Fischer reagent. Pyridine is used to minimize interferences from

**Table 1—Comparison of Experimental VOC Values with Calculated VOC Values of Several Commercial Waterborne Coatings Using the Cal Poly Water Method**

Waterborne Coating	Experimental Coating VOC	Theoretical Coating VOC	Percent Error
JCR 518-4 (roof coatings) <sup>a</sup>	3.7	0	— <sup>d</sup>
HG74-M (latex emulsion) <sup>b</sup>	4.4	3 to 5 (est.)	— <sup>d</sup>
XW-200-6 (exterior flat) <sup>b</sup>	185	189	2.1
W-200-2 (exterior flat) <sup>b</sup>	188	196	4.1
WL-91-1 (pigmented acrylic lacquer) <sup>b</sup>	264	251	6.6
P-46-1 (HG-56 based red primer) <sup>b</sup>	154	141	9.7
P-46-4 (2-component acrylic/epoxy) <sup>b</sup>	139	134	3.7
EWC-38-AZ (elastomeric wall coating) <sup>b</sup>	82	69	18.8
Masterformula (flat interior) <sup>c</sup>	99	102	2.9
PVA-sealer (wallboard sealer) <sup>c</sup>	46	43	7.0
Velvasheen (semigloss enamel) <sup>c</sup>	208	219	5.0

(a) Proprietary latex roof coating.  
 (b) Rohm and Haas latex and starting point formulations.  
 (c) San Luis Point Company, San Luis Obispo, CA.  
 (d) Cannot be determined.

method as an alternate method to D 4017. This alternate method uses a nonpyridine-containing KF reagent and is reported to give better precision than the existing KF method.<sup>4</sup>

The traditional Karl Fischer reagent is a solution of iodine and sulfur dioxide in methanol and pyridine. As long as any water is present, the iodine is reduced to colorless hydrogen iodide. The end point is the first appearance of free iodine, determined

ketones and because it tends to be a good solvent for a wide variety of the resins normally found in paint products. To increase the rate of reaction in pyridine, 1-ethylpiperidine is added at 5% and is reported to function as a buffer or "catalyst." Despite the unique and specific procedures of ASTM D 4017, the determination of water in paints represents a special challenge. This can be explained in part by examining the process taking place during the titra-

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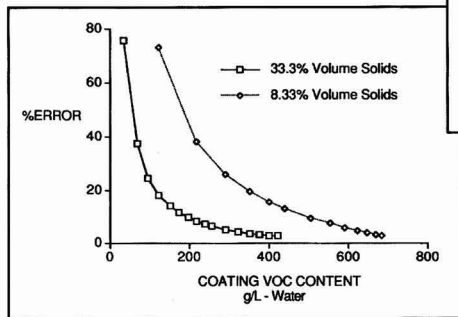


Figure 1—Predicted % error at various VOC contents with nonvolatile content fixed at 33.3 and 8.33%.

GC method at the 95% confidence level. The derived 1986 precision limits for the individual methods of ASTM D 3960 (VOC content) have been used to assess the effect of the combination of these methods on the precision of the calculation of VOC content.

In several such exercises, it was shown that the maximum between laboratory differences in VOC content can become unacceptably large, and as the water content approaches the total volatile content, it may become totally unreliable.<sup>8</sup>

Because ASTM Method D 4017 is not applicable for use with the newer and faster pyridine-free Karl Fischer reagents and because of the limitations of the method, this study investigated alternate ways of determining water in paint using Karl Fischer reagents. Initially, Dr. Max Wills and student researchers in the polymers and coatings program of California Polytechnic State University (Cal Poly), investigated the possibility of dissolving/extracting coatings with tetrahydrofuran (THF). This solvent was chosen because it tends to dissolve a large number of polymers and resins, therefore extracting the water into a homogeneous THF phase. Aliquots of one percent paint in THF solutions/dispersions were prepared and titrated with a commercially available pyridine-free KF reagent. A similar extraction/dissolution method is being investigated by Golton<sup>9</sup> using sonication of a one percent paint in methanol dispersion followed by titration with pyridine-free KF reagent. Cal

tion. Typically, a viscous, pigmented, high molecular weight polymer dispersion is injected dropwise into a pyridine/methanol/sulfur dioxide/iodine mixture forming (in many instances) a dispersion of paint in this mixture. Water is then extracted into the solvent phase for titration. Several problems are routinely encountered with this process, namely: (1) poor dispersibility; large droplets from which water is not completely extracted and/or is extracted in a rate dependent manner; (2) fouling of equipment; deposition of paint or resin on the KF electrodes; and (3) presence of interferences to the KF reagent; certain active metals, metal oxides, metal hydroxides, chromates, melamines, and others.

Published 1986 precision data for water using the ASTM D 4017 KF method indicate that two results, each the mean of duplicate determination, obtained by operators in different laboratories should be considered suspect at the 95% confidence level when the relative difference is greater than 15.0%. For GC water values, the same reproducibility criterion at the 95% confidence level was determined to be 7.5% relative. A more recent study established a precision for reproducibility of 5.0% relative for the Karl Fischer method and 7.8% relative for the

Poly has obtained reasonably good results using both the THF and Golton extraction methods on a limited number of coating samples.

Parallel to our studies with THF, it was found that water could be azeotropically distilled from coatings samples with the solvent 1-methoxy-2-propanol and recovered quantitatively as a solution in this solvent free from pigments, resin, and other non-volatile paint ingredients. Since this distillation method seemed to address and solve most of the problems encountered with the other methods, it was further studied and evaluated for use as an alternate water method with ASTM Practice D 3960 (EPA Reference Method 24). This distillation method appears to be applicable to many different coatings types, and gives superior accuracy and precision as judged by VOC determination of numerous coatings of known composition. A modified "mini" round-robin study also concluded that the Cal Poly method has merit in terms of improved accuracy and precision.

### Cal Poly Water Method

The solvent 1-methoxy-2-propanol (MPA) has a boiling point of 118.5°C and is completely miscible with water. MPA is a relatively good solvent for organic compounds and many polymers, especially those used for making latex paints. It forms an azeotrope with water, which has a composition of 49% water/51% MPA and boils at 97.5°C. When a coatings sample is mixed with MPA and then distilled, water is completely removed and recovered quantitatively as a solution in MPA. Aliquots of this solution are titrated with commercially available Karl Fischer reagent. This procedure eliminates many of the other substances which are sometimes found in coatings that are known to react with Karl Fischer reagent. Additionally, a homogeneous solution of water containing no pigments or polymer is obtained which, unlike the existing ASTM D 4017 Karl Fischer method, does not foul the Karl Fischer electrodes. The complete experimental procedure is given in the following.

#### PROCEDURE

Dry all glassware in an oven and, if possible, cool in a desiccator.

#### SAMPLE

1. Weigh 1.5 to 2.5 g of coating to four places into a 250 mL round bottom one-neck ST 14/20, or 19/22 flask containing a Teflon-coated stir bar. Add 95 mL of dry 1-methoxy-2-propanol (MPA), stopper the flask and stir or sonicate the mixture for 15 min or until the mixture becomes homogeneous. If the MPA is reasonably dry, it may

Table 2—Precision for Within-Laboratory VOC (ASTM E180) Analysis of HG-74M

Precision Measurement	Density g/L	% Water	% Volatile	Coating VOC, g/L - Water
	1035	57.97	57.95	-1
		57.68	57.99	7
		57.51	57.89	11
		57.65	57.95	8
		58.04	57.89	-2
		57.82	58.01	3
		57.86	58.04	2
		57.53	57.89	11
		58.06	57.99	-3
		57.65	57.88	8
Mean		57.78	57.95	4.38
Standard Deviation		0.19	0.06	4.93
Coefficient of Variation		0.33%	0.095%	113%
Repeatability, 95% CL		0.75%	0.21%	255%



be used directly or it may be dried further by storing over 4A molecular sieves.

2. Replace the stopper with a three-way adapter, add a 300 mm West condenser, and a thermometer. The exit end of the condenser should have an adapter which fits part-way down into a 100 mL volumetric flask or is connected to the volumetric flask by means of a ground glass joint vacuum adapter. Heat with a mantle while continu-

obtained by direct weighing of water into the Karl Fischer titration vessel, this is not recommended for precision reasons. Using the same 5 mL pipette for both the sample and the standard cancels volume uncertainties and makes it unnecessary to adjust the temperature of the sample solution to a particular value. In fact, any volume of sample or standard may be used as long as both the sample volume and standard volume are the

individual coatings. The results are presented in Table 1 along with the percent error based on the assumption that the theoretical VOC value is the actual VOC value. The differences between experimental and theoretical VOC values range from 1 to 13 g/L for all samples tested. The differences measured as a percent of the theoretical VOC value have much broader ranges at low VOC content values and become infinite for zero VOC content coatings. This apparent anomaly is a function of how VOC content is expressed and at low VOC content it would appear that an error expressed as a percent of theoretical is simply not valid. We have addressed this anomaly by mathematical modeling (Figure 1 and Table 10).

To demonstrate precision for the Cal Poly water method, a sample of the commercial latex emulsion HG74-M (Rohm and Haas Co.) was distilled with MPA 10 separate times and aliquots of the resulting solutions were titrated in duplicate with a commercial pyridine-free Karl Fischer reagent to determine water content. The density (one trial) and the total volatile content (10 trials) were determined using ASTM Method D 1475 and ASTM Method D 2369, respectively, and the experimental coating VOC content value, measured as g/L-water, was determined. The results of water, total volatile, and VOC content were analyzed for within-laboratory precision using the ASTM Practice for Determining Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals (E 180).<sup>12</sup> The results of this analysis are presented in Table 2.

For HG74-M the within-laboratory repeatability for water content was found to be 0.75%, relative, at 9° of freedom and for total volatile content was found to be 0.21%, relative, at 9° of freedom. When these relatively precise water and total volatile content values are used to calculate the within-laboratory repeatability for VOC content, the apparently absurd value of 255%, relative, is obtained. On an absolute scale the standard deviation for VOC content for HG74-M was found to be 4.93 g/L which we consider to be quite good. Since the actual VOC content of this latex is very close to

**Table 3—Calculated Coating VOC of an 8.33 lb/gal, Zero VOC Coating, Incorporating 1% and 5% Relative Precision Errors for Water and for Volatile Content**

Percent Water	Percent Volatile	Coating VOC Content, with Relative Water Error of		Coating VOC Content, with Relative Volatile Error of	
		1%	5%	1%	5%
40	40	7	32	10	50
50	50	10	48	10	50
60	60	15	70	10	50
70	70	23	104	10	50
80	80	38	166	10	50

ing to stir magnetically through the mantle and collect the distillate in the volumetric flask over a 10-15 min period. When 70 to 80 mL of distillate has been collected, turn off and drain the condenser water and continue the distillation until the adapter leading into the volumetric flask gets hot to the touch and then turn on the condenser water again (this is done to flush any water that might adhere to the condenser into the volumetric flask). When 80 to 90 mL of distillate has been collected, discontinue heating and remove the heating mantle. (At this point, all the water from the sample is in the volumetric flask and is mixed with MPA. The distillation set-up can be used for doing a second sample, which has been prepared in a separate 250 mL flask without cleaning the set-up). Stopper the volumetric flask and bring the temperature to 20-25°C by placing the volumetric flask into a room-temperature water bath.

3. Add 5 mL of 10% salicylic acid in MPA to the distillate. Dilute to volume with MPA and mix the contents of the volumetric flask thoroughly. Salicylic acid is added to neutralize any amines or ammonia that might have codistilled and if left in the free amine form, would react with Karl Fischer reagent. The solid 100% salicylic acid should be dried at 110°C prior to making a 10% solution.

**STANDARD**

4. Weight approximately 1 g of water (to four places) into a 100 mL volumetric flask and add 90 mL of dry MPA. Add 5 mL of 10% salicylic acid in MPA, adjust the temperature to that of the sample, dilute to volume with MPA, and mix thoroughly. (Note: While the Karl Fischer titer may be

same, can be measured with good precision, and are at the same temperature.)

**ANALYSIS AND CALCULATION**

Titrate 5 mL aliquots of sample, standard, and blank MPA with any commercial pyridine-free Karl Fischer reagent.<sup>10</sup> (Conventional pyridine-containing Karl Fischer reagent may also be used).

$$g \text{ H}_2\text{O sample} = \frac{[(\text{mL KF}_{\text{sample}} - \text{blank}) / (\text{mL KF}_{\text{standard}} - \text{blank})] \times (g \text{ H}_2\text{O}_{\text{standard}})]}{1}$$

**VOC Values Obtained with Known Samples**

A latex emulsion, a latex roof coating, several waterborne coatings representing Rohm and Haas starting formulations, and three latex paints from a local paint company, all of known calculated VOC content, were analyzed in at least duplicate for water using the Cal Poly distillation method. In addition to the water analysis, the total volatile content using ASTM Method D 2369 and the density using ASTM Method D 1475<sup>11</sup> were determined for each coating. The coating VOC content (g/L-water) was calculated from these experimental values and compared with the theoretical coating VOC content values provided by manufacturers of the

**Table 4—White Base Formula RS-052**

Raw Material	Weight (lb) <sup>a</sup>	Volume (gal)
Acrylic emulsion (50% NVM).....	20.155	2.316
Titanium dioxide.....	7.200	0.216
Anti-settling/thickening agent.....	0.205	0.013
Flattening pigment.....	14.400	0.637
Wetting agent (21% NVM).....	0.325	0.038
Defoamer.....	0.375	0.053
Texanol.....	0.515	0.066
Ethylene glycol.....	0.515	0.056
Water.....	5.040	0.605
Totals.....	48.730	4.000

(a) All weights were measured to the nearest 0.005 lb.

zero, the precision parameter of repeatability begins to approach infinity because division by a value approaching zero is a part of the repeatability calculation. The latter result illustrates that good precision in the individual methods used for determining VOC content cannot give good precision expressed as repeatability (or reproducibility) as defined by ASTM E 180 for VOC content at low VOC values because a linear relationship does not exist for precision in VOC content to precision in water content. This point is further illustrated in Table 3 where a theoretical zero VOC coating (density = 8.33 lb/gal) is compared to the calculated coating VOC content when relative repeatability precision limits of 1% and 5%, respectively, for water and volatile content are incorporated into the calculation. Table 3 shows that the coating VOC content and the total volatile content are directly and linearly related and are therefore also related in precision. The water content and coating VOC content, however, are not linearly related and the respective precisions, therefore, are also not related. As the density of a coating increases, the relationship between the VOC content's lack of linearity with the precision of water is magnified even further. As the water content becomes less than the total volatile content, the relationship between the precision of water content and precision of coating VOC content improves but continues to be nonlinearly related.

### Preparation of Samples for Round-Robin Analysis

Three laboratories, Cal Poly, United States Testing Company (a commercial laboratory), and the SCAQMD (a California governmental regulatory laboratory), participated in a modified round-robin study in which the VOC content of four water-based paints was determined using the Cal Poly distillation method for water, ASTM Method D 1475 for density, and ASTM Method D 2369 for total volatile content. The four water-based paints were prepared by Cardinal Industrial Finishes, South El Monte, CA. The emphasis for formula selection was placed on critical levels of water and

**Table 5—Additional Formulations Prepared from White Base Formula RS-052**

Raw Material	Formulation <sup>a</sup>		
	RS-053	RS-054	RS-055
White base formula (RS-052) .....	5.060	10.005	6.885
2% Thickener solution .....	6.590	3.970	6.885
Water .....	3.540	2.060	3.440
Butyl carbitol .....	1.010	2.000	0.000
Totals (lb) .....	16.200	18.035	17.210
Totals (gal) .....	1.753	1.793	1.798

(a) All weights are measured to the nearest 0.005 lb.

cosolvent content such that either the water level was high, the VOC content was low or, as in one case, such that a single coating was both high in water content and low in VOC content. The high water/low organic solvent content coating was of interest because both of these conditions tend to magnify the error in VOC content determination. A four-gallon white base formulation, RS-052, was prepared (Table 4). This coating was then reformulated further by adding varying amounts of butyl carbitol, a two percent thickener solution, and/or water to give three additional paints, RS-053, RS-054, and RS-055 (Table 5). The theoretical VOC content of these formulations was calculated from data provided by raw materials manufacturers.

### Round-Robin Discussion

The samples were evaluated by each of the three laboratories by performing a minimum of two distillations on each, followed by at least duplicate titration of the distillate from each sample using any commercially available Karl Fischer reagent to obtain water values. The density and total volatile content was determined for each sample in duplicate. No attempt was made in this study to perform different day or different operator analyses. Data in this paper are identified as originating from Lab A, Lab B, and Lab C.

Lab A distilled each sample in duplicate and titrated each sample in triplicate using a single water standard. Both Lab A and Lab B titrated the individual distillates using conventional pyridine containing Karl Fischer

reagent. Lab B deviated significantly from the Cal Poly procedure because it was not equipped with a standard Karl Fischer titration apparatus. Lab B used a coulometric KF titrator and used 100 microliter aliquots for titration rather than the specified 5 mL aliquots and obtained lower repeatability precision than the other labs. Lab C carried out three distillations on each sample, titrated each distillate in duplicate and used the pyridine free one-component Karl Fischer titrant "Aquistar Comp-5."<sup>12</sup> Because the titration methods of the three laboratories were not the same, precision for the 95% confidence level within-laboratory repeatability is presented separately for each of the participants (Table 6).

One of the participating laboratories has extensive experience in determination of water in coatings by both the Karl Fischer method (ASTM D 4017) and the gas chromatographic method (ASTM D 3792). This lab also analyzed two of the round-robin samples using the conventional ASTM KF method and experienced many of the difficulties presented in the introduction of this paper.

Duplicate water results of each of the four coatings obtained by the three participating laboratories were analyzed statistically in accordance with Practice E 180.<sup>12</sup> The between-laboratory coefficient of variation was 0.98% relative, at 27° of freedom. Based on this coefficient, the reproducibility at the 95% confidence level is considered suspect if they differ by more than that relative amount. The data for duplicate water determinations by the three participants are presented in Table 7. A summary of the combined density, total volatile content, water content, and VOC content is presented in Table 8.

### Calculation of the VOC Content

Precision data for ASTM Method D 1465 (density), obtained in a 1990 round-robin study of three water-reducible topcoats and one electrocoat primer<sup>8</sup> gave a 95% confidence level value for relative repeatability of 0.4% and a relative reproducibility value of 3.1%. For RS-052, RS-053, and RS-054, the three laboratories in this study agreed within 0.43%. The variance from theoretical values were typically less than 1.0%. Of all density values, only one fell outside this norm at 1.5% of the theoretical value. The density values of all three laboratories fell within the reported 1990 relative reproduc-

**Table 6—Percent Relative Repeatability at the 95% Confidence Level Using the Cal Poly Water Method by Different Laboratories**

Participant	Percent Repeatability for Sample <sup>a</sup>				
	RS-052	RS-053	RS-054	RS-055	Average
Lab A .....	1.97	1.59	1.36	3.55	1.72
Lab B <sup>b</sup> .....	2.03	2.72	2.83	2.28	2.47
Lab C .....	1.36	1.47	1.15	1.04	1.21

(a) Repeatability of 0 is perfect; 1.0 is considered excellent.  
 (b) Used 100 microliter aliquots for titration.

**Table 7—Comparative Round Robin Water Results of Three Laboratories Using the Cal Poly Water Method**

Precision Summary/Lab	Percent Water in Sample			
	RS-052	RS-053	RS-054	RS-055
Lab A .....	32.48	71.24	50.23	68.62
	32.71	71.81	49.89	69.84
Lab A mean .....	32.60	71.53	50.06	69.23
Lab B .....	32.10	71.67	50.12	71.83
	32.85	71.15	51.04	71.16
Lab B mean .....	32.48	71.41	50.58	71.50
Lab C .....	32.19	72.13	49.97	71.04
	31.86	72.05	50.28	70.79
	32.14	71.27	50.31	71.08
Lab C mean .....	32.06	71.82	50.19	70.97
Theoretical value .....	31.57	71.38	50.40	71.62
Total mean .....	32.38	71.59	50.28	70.57
Standard deviation .....	0.36	0.40	0.38	1.06
Coefficient of variation .....	1.11%	0.56%	0.76%	1.50%

ibility precision. In the same 1990 study, a relative repeatability precision of 1.4% and relative reproducibility precision of 3.4% at the 95% confidence level was obtained for ASTM Method D 2369 (total volatile content). In the present round-robin study, the mean values for total volatile content of all three laboratories were within 1.3% of the

calculated total volatile content and well within the reported reproducibility precision.

The ASTM D 4017 Karl Fischer Method for water content determination gave a relative repeatability of 2.4% and relative reproducibility of 5.0% (95% confidence level) in the 1990 study. In a 1986 study<sup>2</sup> these

numbers were 4.7% for relative repeatability and 15% for relative reproducibility. When the 1986 precision limits for density, total solids, and water (by KF titration) were used to calculate the 95% confidence level precision limits for coating VOC content (i.e., repeatability and reproducibility of ASTM Practice D 3960) for the two water-reducible automotive topcoats which were studied, values of 5.3% and 9.8% for repeatability and reproducibility, respectively, were obtained. Using the same procedure to calculate repeatability and reproducibility precision for ASTM D 3960 with the 1990 density, total volatile content, and water (by KF titration) precision limits, values of 10.7% for repeatability and 54.4% for reproducibility are obtained at the 95% confidence level for the four automotive coatings tested.<sup>6</sup> At first glance, it appears that *better precision in the methods* being used to calculate VOC content gives *poorer precision in the numerical VOC content*. This anomaly is explained by the fact that the precision for the methods used in calculating VOC content is not directly related to the precision of the numerical value of the VOC content in coatings of varying VOC content. Particular precision limits can be related to numerical VOC content only when the numerical VOC content values of different

**Table 8—Summary of Density, Water Content, Solids, and VOC Values for Round Robin Samples Tested by Three Laboratories**

Sample	Participant	Precision	Density, lb/gal	Water Fraction	Solids Fraction	Material VOC <sup>a</sup>	Coating VOC <sup>b</sup>
RS-052	(Theory) .....		12.19	0.3157	0.6627	32	59
	Lab A .....		12.26	0.3260	0.6609	19	37
	Lab B .....		12.28	0.3248	0.6517	35	66
	Lab C .....		12.29	0.3206	0.6637	23	44
	Mean .....		12.28	0.3238	0.6588	26	49
	Std. Dev. ....		0.01	0.0028	0.0063	8	15
	Coeff. Var. ....		0.11%	0.86%	0.95%	31%	31%
RS-053	(Theory) .....		9.24	0.7138	0.2171	77	365
	Lab A .....		9.28	0.7153	0.2149	78	379
	Lab B .....		9.29	0.7141	0.2118	83	403
	Lab C .....		9.22	0.7182	0.2163	72	350
	Mean .....		9.26	0.7159	0.2143	77	377
	Std. Dev. ....		0.04	0.0021	0.0023	5	27
	Coeff. Var. ....		0.46%	0.29%	1.07%	7%	7%
RS-054	(Theory) .....		10.06	0.5040	0.3731	148	378
	Lab A .....		10.11	0.5006	0.3717	155	393
	Lab B .....		10.16	0.5058	0.3782	141	368
	Lab C .....		10.15	0.5019	0.3723	153	393
	Mean .....		10.14	0.5028	0.3741	150	385
	Std. Dev. ....		0.02	0.0027	0.0036	7	15
	Coeff. Var. ....		0.24%	0.54%	0.95%	5%	4%
RS-055	(Theory) .....		9.57	0.7162	0.2752	10	55
	Lab A .....		9.56	0.6923	0.2748	38	182
	Lab B .....		9.56	0.7150	0.2757	11	59
	Lab C .....		9.44	0.7097	0.2746	18	90
	Mean .....		9.52	0.7057	0.2750	22	110
	Std. Dev. ....		0.07	0.0119	0.0006	14	64
	Coeff. Var. ....		0.76%	1.68%	0.21%	64%	58%

(a) Material VOC = g/L.  
(b) Coating VOC = g/(l-water).

coatings are approximately the same. VOC contents of approximately 120 g/L and water values of 60 to 70% were reported for the coatings tested in the 1990 study. No numerical VOC values were reported for the two coatings tested in the 1986 study (we estimate that these were probably near 300g/L-water).

The data in Table 9 summarize the experimental VOC content values obtained

$$dV = (dV/dN)_{W,D} \times dN + (dV/dW)_{N,D} \times dW + (dV/dD)_{W,N} \times dD \quad (3)$$

This derivative was used to construct several graphs that predict error. Figure 1 illustrates a case where the coating VOC content is examined between 0 and 480 g/L and at a fixed nonvolatile content of 4.0 lb/gal or 33% by volume. Water and organic solvent content is varied to create VOC content change. The resolution of measurement

These predicted errors are not caused by poor measurement of water, volatile content, or density, but are a function, or mathematical quirk, of the formula used for calculating the VOC content from what are, in most cases, very precise measurements.

The Cal Poly distillation method gave good interlab agreement and results well within predicted error ranges as indicated in Table 10. By controlling the determination of the water content to 0.5%, the volatile content to 0.3% and the density to 0.1 lb/gal the VOC content is determinable for most waterborne coatings. This study not only pointed to the excellent results in the water content analysis, but also the limits of VOC determination by test methods in ASTM D 3960. Further study of waterborne coatings with low VOC contents and/or high water contents along with mathematical modeling should be used to set standards for accuracy in VOC content determination. The 10% allowance by air quality regulators may be practical at water contents less than 60% or VOC contents above 160 g/L. As the water content increases or the VOC content decreases from those levels, the allowance by air quality regulators should certainly be widened to reflect the large changes in VOC content that occur with small measurement errors of water content.

**Table 9—Comparison of Theoretical VOC Content with Experimental VOC Content for the Modified Round Robin and Precision Data**

Sample	RS-052	RS-053	RS-054	RS-055
VOC content, theory .....	59	365	378	55
Mean VOC content, actual ....	49	377	385	110
Standard deviation .....	15	27	15	64
Coefficient of variation .....	31%	7%	4%	58%

in the modified round-robin study of samples RS-052 to RS-055 using the Cal Poly distillation method followed by Karl Fischer titration of the distillate for water determination and compares it with theoretical values calculated from raw material ingredient data.

The results for the VOC content of RS-053 and RS-054 are good. The theoretical and experimental VOC content values agree within 10% of the allowance that the SCAQMD and other regulatory districts permit. RS-052 gave results within 15 g/L VOC content of the calculated value. RS-055 was expected to be the most difficult to analyze since it combines a low coating VOC content (0.86% total by weight) and a high water content (71.62% by weight). Test results of this study confirm this conclusion for sample RS-055.

### Formula for Calculation of Coating VOC Content

Using a mathematical modeling approach,<sup>13</sup> equations (2) and (3), it is possible to assign an expected absolute precision range value to the experimental determination of density, total volatile content (or nonvolatile content), and water content. The error that may be expected in calculating VOC values from the experimental results of these methods can then be calculated as follows:

Let

- V = VOC content, less water
- W = % Water by weight
- N = % Nonvolatile by weight
- D = Density of coating (lb/gal)
- D<sub>w</sub> = Density of water

Then the VOC content is

$$V = (100 - W \cdot N) \times D / (100 - W \times D/r_w) \quad (2)$$

By taking the derivative dV with respect to the variables W, N and D, an estimate of error can be derived, equation (3)

of each variable was held at the following raw numbers:

$$dN = 0.30 \% \quad dW = 0.50 \% \quad dD = 0.10 \text{ lb/gal}$$

(% Nonvolatile)    (% Water)    (Density)

At these parameters, a 10% error would be expected at about 168 g/L coating VOC content. At 360 g/L VOC content, the determination was anticipated to yield results within a three percent error margin. At 60 g/L VOC content, the determination was anticipated to yield results with a 30% error margin. At 10 g/L VOC content and/or as the total water content approaches the total volatile content, errors approaching infinity are possible.

A second graph was constructed to illustrate the predicted error of a waterborne coating with a lower nonvolatile by volume

### Conclusion and Recommendations

The Cal Poly distillation modification of the Karl Fischer water determination of ASTM D 4017 gives accurate and precise water content of coatings and consequently better VOC values on waterborne paints. The precision appears to be substantially

**Table 10—Error Comparison to Predictions from Error Analysis**

Sample	RS-052	RS-053	RS-054	RS-055
VOC content, theory .....	59	365	378	55
Mean VOC content, actual ....	49	377	385	110
Deviation from calculated .....	10	12	7	55
Coefficient of variation .....	31%	7%	4%	58%
Predicted range of error .....	37.2%	14.1%	7.5%	96%

content and was superimposed on the previous graph. Figure 1 illustrates this case where the coating VOC content is examined between 0 and 480 g/L at a fixed nonvolatile content of 1.0 lb/gal or 8.33% by volume. Water and organic solvent content are varied to create VOC content change. The resolution of measurement of each variable was held at the same value as those used in the previous example. The graph shows that, at these parameters, a 10% error would be expected at about 500 g/L of VOC content. Errors approaching infinity would be possible at VOC contents of 100 g/L and lower.

better than that obtained with previous methods. A relative reproducibility of 2.85% at the 95% confidence level was obtained on four coatings with water contents ranging from 32 to 72%. Azeotropic distillation of water with 1-methoxy-2-propanol followed by Karl Fischer titration of the distillate appears to be applicable to many different coatings types. The distillation offers several advantages, including the elimination of numerous potential interfering materials which are known to react with Karl Fischer reagent, the prevention of electrode fouling by polymer, and allows the use of the



more general and extensively documented ASTM E 203 Karl Fischer titration method<sup>14</sup> for water determination.

Mathematical modeling shows that waterborne coatings with greater than 60% water content or VOC contents lower than about 160 g/L are extremely difficult to analyze using the existing ASTM methods for VOC analysis from a precision standpoint in which 10% or better reproducibility is desired. The 10% allowance presently in use by many air quality agencies should be replaced by a sliding tolerance scale which reflects the specific parameters of coating formulations.

We suggest that the EPA and ASTM consider round-robin testing of the Cal Poly distillation method to confirm the results obtained in this study and to test the potential broader applicability of the method. We also suggest that Cal Poly evaluate extraction-dissolution water analysis (e.g., the Golton Method) and compare it against the distillation method in terms of accuracy, precision, and ease of use. The LASCT intends to initiate further studies to determine if the Cal Poly water method will reduce inaccuracies potentially caused by substances such as ammonia, ionic compounds, and additives, such as melamines and certain pigments.

## Acknowledgments

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# A Report on the Paint/Coatings Industry in the Russian State and Forecasts for the Future

Dr. Boris B. Kudryavtsev—International Association of Paint and Pigment Manufacturers\*

The Russian paint industry, reviewed here by Dr. Kudryavtsev, is in a process of evolving from the 1989 democratic revolution in the USSR. Presented are statistics on coatings production, which indicate the effect of economic upheaval being experienced in the former USSR. The size of the Russian market and its coatings requirements are shown by Dr. Kudryavtsev to be outstripping the production levels, causing shortages and high costs in manufacture.

Dr. Kudryavtsev, Chairman of the Executive Committee of the International Association of Paint and Pigment Manufacturers (Commonwealth of Independent States (CIS)), and Editor of the Russian Coatings Journal, has led several Russian industry groups which visited the FSCT Annual Meeting and Paint Industries' Show in recent years.

\* \* \* \* \*

According to information provided by Goskomstat (the State Committee on Statistics) of the Russian Federation, 1,356 Russian enterprises produced 851.1 kilotons (kt—thousand tons) of paint in 1993.

\*LACMA-IMAX, 31, Gilliarovskii St., 129832, Moscow, Russia

This amount was 30% below the level of the previous year and almost one-third that produced in 1990—a year before the democratic revolution (Table 1). In 1994, this downswing continued: in the first quarter of 1994, with a decrease of 30.3% and, in the second quarter, 26%. Volumes of output were 129.2 and 280.2 kt, respectively.

Comparison with the United States paint/coatings industry shows that, in 1990, Russian output of paint accounted for 50% of the U.S. level (4600 kt) but, in 1993, this figure totaled only 17% (4900 kt).

In 1990, Russian need for paint/coatings was determined to be 3500 kt, but only 64.5% of the production demand was satisfied. Import share was 160 kt or 7.3% of the production output. Among the products primarily imported were automotive enamels and primers (25%), marine coatings (20%), coil coatings (2.5%), and consumer goods supplied under cooperative terms with CMEA (the Council of Mutual Economic Aids) member countries (25%).

As could be expected, paint-consuming industries (such as construction, automobiles, and furniture, etc.) were damaged by the total economic recession. Output of these industries decreased 70-200% during the

period of 1990-1993, according to various statistics. (For example, the recession index for the automotive industry was 30%, while it was rather small for the construction industry.)

At the same time, the paint shortage increased greatly and, in 1993, reached 250-280 kt. The production structure changed significantly as a result of the unsteadiness of production caused by the recession in the consumer industries. On one hand, there were changes in the assortment of commercial paints available as a result of the recession. However, this also forced an increase in imports of other coating products. According to the official statistics, in 1993 the increase in imports made up 170 kt, or 20%, of the 1990 level of output.

Tables 2 and 3 show the changes which occurred in the Russian paint/coatings market. From these data it can be noted that there was an increase in production for paints using advanced technology in 1993 (57.2%) as opposed to 1990 (47%). Traditional production methods (such as with oil-based paints, drying oils, and epoxy cellulose based materials) decreased from 53 to 42.8% over the same years. Characteristically, powder coatings manufacture, as well as that for

Table 1—Structure of Paint/Coatings Production

Assortment of Paint Coatings by Main Groups	Production						Production recession during 1990-1993, %
	1990		1992		1993		
	Thous. †	%	Thous. †	%	Thous. †	%	
Varnishes based on condensation resins .....	483.8	20.8	286.9	23.1	204.9	24.1	2.4
including semi-products .....	327.0	—	212.0	—	168.8	—	—
Enamels based on condensation resins .....	417.0	17.9	270.3	21.8	215.2	25.3	1.9
Polymers based varnishes .....	67.8	2.9	40.8	3.3	25.1	2.9	2.7
Epoxy cellulose lacquers .....	300.6	12.9	173.9	14.0	122.4	14.4	2.5
Diluents .....	142.1	6.1	83.7	6.7	55.8	6.6	2.5
Water paints .....	59.7	2.6	37.1	3.0	27.4	3.2	2.2
Oil paints .....	458.9	19.7	194.4	15.7	113.0	13.2	4.1
Drying oils .....	378.2	16.2	147.9	11.9	80.2	9.4	4.7
including semi-products .....	162.0	—	69.8	—	40.9	—	—
Powder coatings .....	2.8	0.1	1.2	0.1	1.4	0.2	2.0
Other products (thermoplastics, alcohol based lacquers, etc.) ....	19.2	0.8	5.1	0.4	5.7	0.7	3.4
<b>Total .....</b>	<b>2330.1</b>	<b>100</b>	<b>1241.3</b>	<b>100</b>	<b>851.1</b>	<b>100</b>	<b>2.7</b>
<b>Total with the exception of semi-products .....</b>	<b>1841.1</b>		<b>959.5</b>		<b>641.4</b>		

**Table 2—Dynamics of Production Recession**

Parameter	1990	1991	1992	1993
Paint coatings output, kt .....	2330.1	1909.5	1241.2	851.1
Production recession, % on 1990 .....	—	81.9	53.3	36.5
on the year preceding .....	—	81.9	65.0	68.6

thermoplastics for traffic paints, increased in 1993 by 16.7 and 13.7%, as compared with 1992; autofinishes output remained at the same level despite the production recession.

There are several groups consisting of paint/coatings enterprises represented in Table 4. Almost half (46.7%) of the total paint output is produced by the eight largest paint plants and amalgamations such as JSC (joint stock company) Cherkessk KhPO, JSC Lakokrasna (Yaroslavl), Liability Limited Partnership Yaroslav Plant Pobeda Rabochikh, as well as Zagorsk, Kotovsk and Chelyabinsk paint coatings plants, SPF (scientific product firm) Pigment (S. Petersburg), JSC Empils (Rostov/Don). In addition, 35.8% of paint production was manufactured by 11 smaller, specialized paint/coatings plants, 14% by domestic chemical plants which also produce paints, and 17% by the defense industry plants which produce paint under conversion terms.

The remaining 88 paint/coatings manufacturers are represented by oil-mills (30% of boiled oils), chemical plants (manufacture of paint-coatings based on the by-products), and small enterprises of local industry (17.5% of paint coatings).

Specialized amalgamations and plants (19) contributed 14% of the number of enterprises producing paint/coatings; their share in the total production output was 52.6%. Concurrently, 70% of advanced paint/coatings grades are produced by the eight largest paint plants (Table 4).

It is typical of the last several years that many smaller cooperative enterprises producing between 100 and 500 kt of paint per year have been established. Generally, considering the shortage of advanced products, these firms turn out high quality products. Thus, JSC Gepard manufactures about 300 tons per year of air-drying automotive enamels and the firm Paraugava annually produces about 800 tons of up-to-date architectural coatings (parquet varnishes, wood glazing paints containing antiseptics, water acrylic paints). Due to their few number and

**Table 3—Dynamics of Paint/Coatings Assortment Progress**

	Production						Volume decrease during 1990-1993, %
	1990		1992		1993		
	Thous. t	%	Thous. t	%	Thous. t	%	
Advanced .....	865.4	47.0	513.0	53.5	366.7	57.2	2.36
Traditional .....	975.7	53.0	446.4	46.5	274.7	42.8	3.55
including							
oil paint and drying oils .....	675.1	36.6	272.5	28.4	152.3	23.7	4.4
epoxy cellulose paints .....	300.6	16.4	173.9	18.1	122.4	19.1	2.5
<b>Total .....</b>	<b>1841.1</b>	<b>100</b>	<b>959.4</b>	<b>100</b>	<b>641.4</b>	<b>100</b>	<b>2.87</b>

small-size but practically new fixed capital stocks, as well as low overhead costs, these firms can successfully compete with the large plants both in product quality and price.

To analyze the state of the industry and to predict its development, information on the geographical situation of paint coatings manufacturers is needed. From Table 5 it can be seen that 74.1% of paint manufacturers are located in the Central and Southern

territories of Russia, 7.1% in the East, 12.2% in the Ural, 6.6% in the Northwestern region of the country.

Also, it must be noted that most raw materials, and especially petrochemical products (except perhaps vegetable oils), are delivered to the Central and South territories of the country from the Ural and Eastern regions. Moreover, the Ural and Eastern regions will remain the primary sources of energy for a long time.

With large plants with a diversified infrastructure (powerful boiler-houses, refrigerators, railway system, own fleet of automotive transport and tank-trucks, repair shops, etc.), a share of their overhead expenses in product cost will be permanently increased, with another production recession predicted to take place within the next two years. Today, transportation costs have already approached the 20% level of the raw material cost. Energy costs, including those for water, gas, and electric energy, total 5-7% of the

cost structure, with those for power-intensive manufacturers (such as those for epoxy resins, pentaerythritol, phthalic anhydride) reaching 15-20%. Thus, finished product cost, especially for enterprises located in the Central and Southern territories, is not defined by raw materials costs and man-hours but depends, first of all, on the energy and transportation expenses as well as overhead costs. As the production output is diminished, a share of these cost substituents will be increased.

These factors have a much lower impact on the price of products manufactured by small enterprises located near sources of raw materials and energy.

It should be noted that such enterprises, whether in existence for a long time or just newly established, are highly mobile and capable of developing new types of paints commercially, are able to afford to produce and market them at a lower cost, and to be able to compete with foreign paint producers. For the immediate future, large enterprises will not be able to really compete against the high-quality and relatively inexpensive products manufactured by western firms. The domestic cost for

**Table 4—Paint Coatings Produced by Other Industries' Plants**

Plant Groups	Number of Plants	Share %		
		Advanced Paints	Traditional Paints	
			Oil Paints and Drying Oils	Epoxy Cellulose Paints
Paint coating plants				
large .....	8	88.6	2.7	8.7
small .....	11	24.1	62.4	13.5
Domestic chemistry plants .....	14	40.0	60.0	—
Chemical plants .....	18	50.5	49.5	—
Chemical concerns on conversion .....	17	33.3	15.4	51.3
Chemical amalgamations and petrochemical plants .....	15	27.7	47.7	24.6



Table 5—Geography of Paint Coatings Manufacturers. Production Output (Thousand Tons)

Central		Southern		Eastern		Ural		Northwestern			
Belgorod r. ....	6.3	Nizhyn Novgorod r. ...	23.7	Astrakhan r. ....	4.2	Altai ter. ....	32.8	Bashkiry .....	21.0	St. Petersburg ....	59.0
Bryansk r. ....	13.5	Orel R. ....	6.2	Degestan .....	2.0	Irkutsk r. ....	6.3	Mary-El .....	5.4	Karley .....	0.6
Volgograd r. ...	7.0	Ryazan r. ....	12.0	Kabardino-		Kemerovo r. ...	18.6	Orenburg r. ....	5.2	Kirov r. ....	6.2
Voronesh r. .	18.3	Kuibyshev r. ....	24.0	Balkary .....	5.0	Kranoyarsk r. .	14.0	Prm r. ....	32.0	Pskov r. ....	0.2
Ivanov r. ....	3.2	Saratov r. ....	14.0	Karacheavo-		Novosibirsk r. ...	8.8	Sverdlovsk r ....	13.0	Novgordo r. ....	2.7
Kaluga r. ....	0.6	Smolensk r. ....	6.3	Cherkessk ....	163.0	Omsk r. ....	2.8	Tatarstan .....	30.4	Arkhangelsk r. ....	6.0
Kurgan r. ....	1.2	Tambov r. ....	54.5	Kranoda ter ....	31.3	Primorsk ter. ....	7.6	Chelyabinsk . r.	44.0		
Kursk r. ....	9.5	Tver r. ....	0.9	Rostov r. ....	176.0	Tyumen r. ....	0.1				
Lipetsk r. ....	4.3	Tula r. ....	18.8	Stavropol ter. ...	36.0	Chuvashy .....	11.4				
Moscow .....	12.5	Yaroslavi r. ....	122.1	South Ossety ....	5.1	Khabrovsk .....	0.7				
Moscow r. .	126.5			Checheno-							
				Ingushety .....	3.1						
<b>Total .....</b>	<b>485.4</b>				<b>425.7</b>		<b>103.1</b>		<b>151.0</b>		<b>74.7</b>

epoxy resins is \$3,000-5,500 (rate 2200 rbl/dol), while Great Britain offers corresponding resins of much higher quality at a price of \$2,000-2,500.

Pentaerythritol prices in the domestic market account for \$1,700-1,750 USD, whereas firms such as Degussa and Perstorp are offering the same product of higher quality at a price of \$1,400-1,450 USD. Cost of waterborne paints made by Helios is lower than similar products of Zagorsk Paint Coating Plant and SPF Pigment. The cost of car finishes produced by Pobeda Rabochikh are comparable to those produced by Tikkurila and Helios, while greatly ranking below them in quality.

There are many examples which could be produced to show the real threat to our enterprises originating from foreign manufacturers. However, it is necessary to take into account another fact: both in Europe

and in the U.S. there are no large paint coatings firms whose personnel number more than 1000 people; yet this number is typical for our enterprises today. Analysis of the current situation in the paint coatings industry makes it possible to forecast the main trends of its development in near future:

- Intensive development and establishment of small firms producing a limited range of high-quality paint coatings, including foreign partner participation. The growth in production and assortment will result from the business activity of these firms;

- The breakup of large enterprises, with the self-dependent and most viable subdivisions becoming independent enterprises;

- Initially, close economic contacts with the conversion plants in the Eastern regions of the country. Transfer of equipment to these plants on the rights to mutually use, joint venture and filial establishments, utilization of releasing facilities of

conversion manufacturers being situated in immediate proximity to the raw materials and energy resources, while establishing joint ventures for paint coating production;

- Further reductions in production at the large plants including conservation or shutdown of manufacturers who are not profitable;

- The greatest possible encouragement of foreign investments for capital funding, in order to increase the technology level and the economic characteristics of the products.

Finally, reported statistics and their analysis confirm the reality of the danger of bankruptcy to which large plants could be exposed by the end of 1994-95. These indicate the necessity for non-standard survival strategies under the conditions of growing competition with the western firms and private capital to be worked out.

## CDIC—DECEMBER

### "Polyurethane Dispersions"

Ohio Paint Council Representative, Hugh Lowery, of Perry & Derrick Co., updated the membership on recent developments from the Ohio Paint Council's (OPC) December meeting.

The OPC elected Mark Reichart, of Maaron, Inc., Cleveland, OH, as Chairman. The council also chose a new operating committee and new committee chairmen.

During the meeting, NPCA staff member David Lloyd listed three primary government-related concerns of the industry for 1995. They are: the Clean Air Act, lead abatement laws, and disposal of household hazardous waste.

NPCA declared the EPA's announcement that a national AIM VOC regulation would be delayed two years was erroneous. According to NPCA, the sequence of events will be as follows: (1) U.S. EPA will publish the congressionally mandated study of VOC control in consumer goods in March. Reportedly it is already complete; (2) U.S. EPA will then publish a proposed "command and control" type national AIM VOC regulation in April or May. It will contain a table of VOC limits that is essentially the same one EPA was proposing during most of this past year, at the reg-neg sessions; (3) The final regulation will be promulgated on January 1, 1996 and will become effective in August, or no later than November.

✓ Mr. Lowery stated that a new national regulation covering automotive re-

finishing paint is being developed. It will be very similar to the present regulation in the South Bay area of California and will be effective at the same time as the AIM VOC regulation.

✓ A delay by U.S. EPA has left numerous states in the position of having relied on VOC reductions from a nonexistent federal paint regulation, to meet their ozone control obligations under the Clean Air Act Amendments (CAA). The CAA does not permit them to wait for EPA, so the threat of proliferation of conflicting state regulations on architectural paint becomes increasingly serious. However, State Paint Councils in various places have succeeded in heading off state regulations that could have contained provisions more stringent than the coming federal regulation.

✓ The Ohio Public Health Council has released a draft regulation implementing Ohio's new lead law. It concentrates inappropriately on lead based paint and omits necessary provisions concerning other sources of lead.

✓ The state's Advisory Committee on Lead Abatement must recommend future steps to be taken in lead abatement, if any, to the legislature within a year. It has divided into four groups to study the various aspects of its job.

✓ Ohio has received seven HUD grants totaling over \$32 million, for lead abatement.

✓ The market share lead bill was soundly defeated in the Massachusetts House. If the bill would have been passed, Mr. Lowery stated that it could have sparked a nationwide disaster for the industry.

✓ The British Columbia paint recycling legislation is being promoted in both Ontario and allegedly in Washington state, Oregon, California, and Massachusetts. NPCA is working on an alternate approach that the industry can promote as a defense.

✓ The Ohio governor and the new Republican leadership in the Ohio legislature have said they would revise environmental regulations in an effort to improve the business climate.

✓ During 1995, OEPA expects to issue regulations for the use of hazardous waste by cement kilns. This issue is important to the paint industry since much of the industry's waste goes into these kilns.

At the conclusion of Mr. Lowery's presentation, he discussed the following proposed action:

The OPC Operating Committee voted to have attorney John McGough prepare a draft of papers establishing an OPC Political Action Committee and describing its operation for Operating Committee consideration. This would be a grassroots organization because only individuals and not corporations may contribute, under Ohio law. A \$3000-5000 PAC is envisioned. It would contribute very selectively to fund raisers for members of legislative committees whose actions were critical to the paint industry and also contribute to appropriate legislative caucuses. Its purposes would be to permit more grassroots involvement and to elevate the OPC in the estimate of legislators, as an organization with important views. One goal would be to achieve a status such that legislators would consult us before introducing legislation.

Mr. Lowery would like feedback from the Society before he votes.

Technical and Environmental Committee Chairman Kenneth Pendleton, of K.A. Pendleton Co., Inc., revealed that the St. Louis Society has requested the assistance of the CDIC Society on a VOC project. A report on this matter will be presented after the Technical and Executive Committees take action.

The Manufacturing Committee is sponsoring an ISO 9000 presentation at the March meeting. In the fall of 1995, the committee will sponsor a plant tour of Reynolds Metals, a coil coater in Ashville, OH.

Nominating Chairman James E. Flanagan, of Flanagan Associates, Inc., announced that Treasurer Steven Prodomo, of Hilton-Davis Co., is unable to complete his term of office due to a change in work



Dallas Society Officers for 1994-95 (from left) — Treasurer—Donald McBride, Publicity Chairman—H. Edward Spradlin, Secretary—Chip Newcomb, President—Benny Puckett, Vice President—Paul D. Kaplan, and Membership Chairman—Barry Clinger.



Serving as Northwestern Society Officers for 1994-95 are (from left) — Society Representative—Larry Brandenburger, President—Mike Grivna, Vice President—Hal Christhilf, and Secretary—Joe Mills.

responsibility. Teresa L. Case, of Fiberglass Evercoat Co., has accepted the nomination.

According to Scholarship Committee Chairman Andrew Nogueira, of Hunting Industrial Coatings, no applications for the Lew Larson Educational Grant have been received.

The general meeting concluded with a slide presentation/report of the 1994 FSCT Annual Meeting and Paint Industries' Show in New Orleans, LA, by Jim Flanagan.

Valentino Tramontino, of King Industries, Inc., delivered the evening's presentation "CROSSLINKING OF WATERBORNE POLYURETHANE DISPERSIONS."

Dr. Tramontino began by stating that many coatings applications are comprised of waterborne polyurethanes, due to their mechanical properties. Conventional polyurethane dispersions (PUD) are high molecular weight ionic polymers that contain both polyurethane and polyurea molecular species. The speaker then explained that film formation in PUD is by coalescence; however, the typical structure of the dispersion is a hydrophobic core with a hydrophilic shell. Also, the molecular diffusion rate is low, therefore, coalescence is slow.

According to Dr. Tramontino, crosslinking improves film formation, but the typical dispersion is carboxyl functional. This moiety has a lower rate of reaction with aminoplast resins, and the improvement in film properties is slow, thus, the resulting films may have water permeable domains. The speaker then outlined the dispersion investigated for this study.

A low molecular weight hydroxy/carboxyl functional PUD, labeled XM was studied. A high molecular weight PUD labeled XP was used for comparison. After clear waterborne coatings of each PUD were made with 10, 15, 20, 25, 30, and 35% of hexakis(methoxymethyl)melamine (HMMM), additional XP coatings were formulated with

0, 2.5, and 5% HMMM. All formulations were catalyzed with 1% of amine-blocked p-toluenesulfonic acid on resin solids.

The crosslinked films were analyzed for typical mechanical properties and stress-strain studies and solvent swelling experiments were used to characterize the films. Dr. Tramontino revealed that the morphology of the films from the two different PUDs were not similar. The speaker determined that superior stain resistance, more uniform crosslinking, superior mechanical properties—at the proper HMMM level—and a lack of HMMM homopolymer on the cured surface were evident in XM. In addition, superior stain resistance of the bulkpolymer—as exposed by light surface abrasion—was found.

*Q. With the conventional urethane dispersions, cost is often the limiting factor to usage. What is the prospective for competitiveness with this chemistry?*

A. There is a prospect for competitiveness with this product.

WILLIAM E. JELF, III, Secretary

## CLEVELAND—SEPTEMBER

### "Lightfastness of Organic Pigments"

A moment of silence was held in memory of Federation Past-President Eugene H. Ott and James Brogгинi, retired from W.J. Roscoe.

Jim Benduhn, of Dar-Tech, presented President Constance Williams, of The Lubrizol Corp., with the Huls gavel.

Ms. Williams then presented the Past-President's Pin to Fred Anwari, of BFGoodrich Co.

It was announced at the meeting that the Educational Committee is seeking volunteers to act as Science Fair Advisors. Those interested in participating should contact Sharie Moskaluk, of The Sherwin-Williams Co.

Charles Beck read two By-Laws that will be published in the October meeting notice and will be voted on at the October meeting.

Ben Carlozzo, of Mameco International gave an overview of the Technical Committee's 1994 Annual Meeting presentation "CORRELATION OF ACCELERATED EXPOSURE TESTING AND EXTERIOR EXPOSURE SITES, PART II."

Mr. Carlozzo began with a review of Part I, which dealt with the six-month evaluations of nine different paint systems. Part II covers the one-year evaluations. Next, he



Pacific Northwest Society Officers for 1994-95 (from left) — Secretary—Edward Linton, Treasurer—Kenneth Wenzel, Member-at-Large—Beverly Spears, Society Representative—William Schackelford, President—John Westendorf, and Vice President—Richard Tomczak.

## Constituent Society Meetings and Secretaries

**BALTIMORE** (Third Thursday—Martin's West, Baltimore, MD). CONNIE SAUER, Duron, Inc. 10460 Tucker St., Beltsville, MD.

**BIRMINGHAM** (First Thursday—Strathallan Hotel, Birmingham, England). DAVID C. MORRIS, PPG Industries (UK) Ltd., P.O. Box 359, Rotton Park St., Birmingham, B16 0ADS, England.

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

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

**CLEVELAND** (Third Tuesday—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 NEWCOMB, 1448 N. Joe Wilson Rd., Cedar Hill, TX 75104.

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

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

**HOUSTON** (Second Wednesday—Medallion Hotel, Houston, TX). GUY SULLAWAY, Courtaulds Coatings, P.O. Box 4806, Houston, TX 77210.

**KANSAS CITY** (Second Thursday—Cascone's Restaurant, Kansas City, MO). RANDALL L. EHMER, Walsh & Associates, Inc., 500 Railroad Ave., N. Kansas City, MO 64116.

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

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

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

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

**NEW ENGLAND** (Third Thursday—Sheraton Lexington Hotel, Lexington, MA). GENE C. ANDERSON, Chemcentral Corp., 38 Spindlewick Dr., Nashua, NH 03062.

**NEW YORK** (Second Tuesday—Landmark II, East Rutherford, NJ). JOHN W. DU, Hils America, Inc., P.O. Box 365, Piscataway, NJ 08854.

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

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

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

**PIEDMONT** (Third Wednesday—Ramada Inn Airport, Greensboro, NC). ROY MODJEWSKI, Akzo Nobel Coatings, Inc., 1431 Progress St., High Point, NC 27261.

**PITTSBURGH** (Second Monday—Montemurro's Restaurant, Sharpsburg, PA). JAMES REDISKE, Miles Inc., Mobay Rd., Pittsburgh, PA 15205.

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

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

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

**TORONTO** (Second Monday—Meeting sites vary). NATALIE JANOWSKI, Degussa Canada Ltd., 4261 Mainway Dr., Burlington, Ont., L7R 3Y8, Canada.

**WESTERN NEW YORK** —MARKO MARKOFF, 182 Farmingdale Rd., Cheektowaga, NY 14225.

stated that this study was not conducted to evaluate which paint system is better but to see how outdoor exposure correlates to accelerated exposure methods.

The salt fog tester, the prohesion cabinet, and the prohesion cabinet with QUV and Envirotest were used to analyze the systems. Mr. Carlozzo explained how the results differed from the exposure sites. He also said that some of the test results that were borderline in correlation at six months, now correlate and there is now better correlation with those that correlated after six months.

The evening's guest speaker was Romesh Kumar, of Hoechst Celanese Corp. Dr. Kumar discussed "OPTIMIZING LIGHTFASTNESS OF ORGANIC PIGMENTS THROUGH PARTICLE SIZE CONTROL." Joining Dr. Kumar was Willie Schaud, Technical Manager, at Hoechst.

Dr. Kumar began by dispelling several myths (transparency, opacity, weatherfastness, and rheological properties) about organic pigments, saying that some desired properties can be achieved. The speaker then explained the difference between pigments and dyes. Pigments disperse and dyes dissolve.

Coatings are used for two main reasons: (1) decorative purposes; and (2) hiding the substrate. The physical mechanisms for hiding power are light scattering and light absorption. Dr. Kumar cited an example of light scattering in daily life. He said that milk is white, however, if you add something to precipitate the fat, it becomes clear.

The chemistry of the pigment determines its color. Paints must be stable and stability depends on the resin system, the pigment used, and processing. The more pigment that is added, the darker the color becomes.

Pigments can come in spheres, nodules, cubes, needles, agglomerates, and aggregates, said Dr. Kumar. As particle size increases, viscosity increases and it affects lightfastness, opacity, and rheological properties. When particle size decreases, tinting strength and transparency increase. More resin is needed if surface area increases. The speaker stressed the importance of pigment selection in relation to lightfastness/weatherfastness, since the end use and application are different.

In typical paints, pigment breakdown is different due to different exposures. In full shade, they tend to darken with color pigments. On the other hand, in partial shade, they tend to lighten with titanium dioxide. Dr. Kumar concluded that to increase colorfastness, lightfastness, and opacity, particle size must be increased. Larger particle size also requires less resin. Gloss is not really dependent on the pigment except if too much is used; it depends on particle size and how much resin gets to the surface.

*Q. Do you change tint strength with small changes in particle size?*





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002	022	042	062	082	102	122	142	162	182	202	222	242	262	282
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005	025	045	065	085	105	125	145	165	185	205	225	245	265	285
006	026	046	066	086	106	126	146	166	186	206	226	246	266	286
007	027	047	067	087	107	127	147	167	187	207	227	247	267	287
008	028	048	068	088	108	128	148	168	188	208	228	248	268	288
009	029	049	069	089	109	129	149	169	189	209	229	249	269	289
010	030	050	070	090	110	130	150	170	190	210	230	250	270	290
011	031	051	071	091	111	131	151	171	191	211	231	251	271	291
012	032	052	072	092	112	132	152	172	192	212	232	252	272	292
013	033	053	073	093	113	133	153	173	193	213	233	253	273	293
014	034	054	074	094	114	134	154	174	194	214	234	254	274	294
015	035	055	075	095	115	135	155	175	195	215	235	255	275	295
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017	037	057	077	097	117	137	157	177	197	217	237	257	277	297
018	038	058	078	098	118	138	158	178	198	218	238	258	278	298
019	039	059	079	099	119	139	159	179	199	219	239	259	279	299
020	040	060	080	100	120	140	160	180	200	220	240	260	280	300

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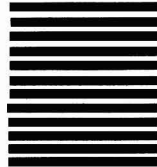
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A. Typically large particles will not have less tint strength.

RICHARD MIKOL, *Secretary*

## CLEVELAND—OCTOBER

### "Melamine Crosslinking Agents—Present and Future"

Carl Knauss was unanimously elected an Honorary Member of the Cleveland Society.

President Constance Williams, of The Lubrizol Corp., acknowledged those who had won awards at the FSCT Annual Meeting and Paint Industries' Show in New Orleans, LA. Jennifer Rumberg, of Mahoning Paint, received the Women in Coatings Award—Purchasing.

Ms. Williams also announced that the Cleveland Society won second place in the A.F. Voss/APJ Awards for the paper "Correlation of Accelerated Exposure Testing and Exterior Exposure." Ben Carlozzo, of Mameco International, presented the check for \$300 to the Society.

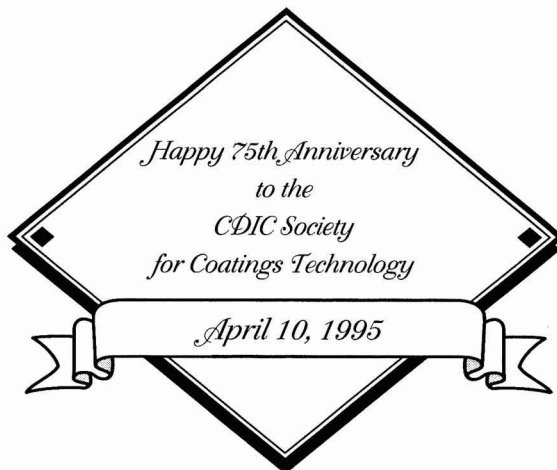
Environmental Control Committee Chairman Robert Toth, of The Glidden Co., reported on the Ohio Paint Council's October meeting. Mr. Toth also informed the members that the CSCT Environmental Symposium will be held on November 15, 1994 at the Hilton South in Independence, OH.

President-Elect Michael A. Wolfe, of Seegott, Inc., reminded members that Bosses' Night will take place on November 15, 1994. Invitations were sent to 150 bosses/technical directors of various coatings companies.

The meeting's guest speaker was New York Society member Nick Albrecht, of Cytec Industries, who spoke on "MELAMINE CROSSLINKING AGENTS—PRESENT AND FUTURE."

Mr. Albrecht described the ultimate coating as one with zero toxicity, zero VOCs, ease of application with 25-year exterior durability, chemical, heat and water resistance, and low cost. He also showed typical melamine-formaldehyde resins for surface coatings.

According to the speaker, alkylation of the melamine resins improves compatibility and solubility, increases stability, lowers self condensation, polymerization, improves film flexibility, lowers viscosity and VOCs, and decreases reactions. The type of alkylation can lower viscosity, increase film flow and leveling, increase E-spray resistivity, increase intercoat adhesion, increase weight loss on curing, and decrease film hardness.



Other coatings highlighted by Mr. Albrecht included the following: performance/compliance/cost ratings of OEM acrylic topcoats; VOC, formaldehyde emissions; environmental etch; low temperature cure response; and powder coatings.

Formaldehyde emissions on cure are lower in higher extents of alkylation. The type of amine resin also affects the formaldehyde emission on cure. Glycoluril resins give lower formaldehyde emissions than melamine resins. Mr. Albrecht predicted future trends that will lower free formaldehyde to less than 0.1%. Nonformaldehyde melamine crosslinking agents (CLA) will be at a higher cost and will also be for specific application areas.

Environmental etch resistance is increased using glycoluril and ISO CLAs. To improve etch resistance, the speaker recommended lowering the amino resin level and baking at a higher temperature. In addition, the catalyst concentration must be increased and the backbone of the resin optimized. Future technology will require innovation in CLA design, backbone resin design, and use of melamine CLAs as co-crosslinkers.

The higher the concentration of para toluenesulfonic acid (pTSA) catalyst with a highly alkylated melamine CLA, the lower the bake temperature needed for cure. However, with this higher concentration, concerns arise in formulation stability, crosslink stability, formaldehyde emission from cured coating, and water and corrosion resistance. A highly alkylated acrylic/CLA resin shows softer cure and longer stability than a lower alkylated acrylic/CLA resin. Innovation is required in catalysis for lower temperature cure.

The powder coating mentioned was tetramethoxy methyl glycoluril with a nonvolatile of > 98%. Improvements in formulation latitude and innovation in eliminating reaction volatile is required in powder coatings.

Mr. Albrecht concluded that the strength of melamine crosslinking agents is performance/cost. However, some concerns do exist, such as VOCs, formaldehyde, E-etch resistance, and low temperature cure.

Q. Are there any room temperature cure melamines?

A. Probably not. pTSA at 5 to 6% is needed, but reversal reactions are a problem.

RICHARD A. MIKOL, *Secretary*

## CLEVELAND—NOVEMBER

### Federation Officer Visit

The meeting featured a visit from FSCT Executive Vice President Robert F. Ziegler and FSCT President-Elect Darlene R. Brezinski, of Consolidated Research Inc.

Mr. Ziegler relayed the success of the 1994 Annual Meeting and Paint Industries' Show in New Orleans citing that over 7,900 attendees and 308 exhibiting companies were present. He noted that the 1995 Annual Meeting and Paint Show, themed "Creativity + Adaptability = Gateway to Success," will be held on October 9-11 in St. Louis, MO.

The 1996 Annual Meeting and Paint Show in Chicago, IL, will be concurrent with NPCA's Annual Meeting.

Mr. Ziegler also stated that FSCT and NPCA are working together on the Panorama™ MSDS on CD-ROM Retrieval System, as well as the State Paint Councils.

President-Elect Darlene R. Brezinski addressed the current activities of the Publications Committee of FSCT. She stated that FSCT's Paint Stone is available to publicize upcoming events. She also revealed that the updated, encyclopedic *Paint and Coatings Dic-*

tionary will be available on CD-ROM in 1995.

The following question was addressed to Dr. Brezinski:

*Q. What about using the NAFTA Harmonized Tariff codes, will this be on the MSDS CD ROM?*

A. Yes.

The evening's speaker was Cleveland Society member Robert Toth, of The Glidden Co., who spoke on "REGULATIONS OF VOCs IN ARCHITECTURAL AND INDUSTRIAL MAINTENANCE (AIM) COATINGS."

Mr. Toth began his presentation by showing a videotape on VOC regulations. The contents of the tape addressed the determination of VOCs and the need for precise VOC testing due to poor analytical techniques. A poor technique can lead to a decline in sales and production. The video suggested procedures to reduce the variations of volatile organic determination. (The tape is available to anyone in the Cleveland Society who is interested in viewing it.)

Mr. Toth discussed the dangers of ground level ozone, stating that it reduces lung functions and inhibits plant growth. Ground level ozone is formed by the reaction of hydrocarbons (most paint solvent vapors-VOCs) with nitrogen oxide in the presence of sunlight. The speaker defined a VOC as volatile material containing carbon atoms.

All traditional paint solvents are included in AIM coatings. In 1992, annual sales of paints were \$12.2 billion and AIM coatings accounted for 50% of the total paint industry.

Due to this high number, VOCs must be regulated. Some sources of VOCs include vehicle emissions, organic solvent evaporation, surface coatings, etc. The speaker

stressed that the objective of VOC regulations is not to reduce VOC emissions, as such, but rather to reduce the formation and therefore the concentration of ground level ozone.

Currently, there are six states that regulate AIM coatings: Arizona, California, New York, New Jersey, Texas, and Kentucky. The major challenge in regulating VOC levels in AIM coatings is to construct product categories that will allow higher VOC limits for certain coatings that could not be produced for technical and/or economic reasons if a single limit were placed on all AIM coatings.

In February 1992 EPA convened a negotiated rulemaking committee to address the VOC content of paints for AIM coatings. However, after almost three years of negotiations between EPA and AIM, Reg-Neg Committee was terminated. Neither the EPA proposal nor the industry proposal had the potential to form the basis for a consensus.

At the present, EPA has proposed a three-phase program reducing VOC emissions by 40% by the year 2004. Three tables, one each for the years 1996, 2000, and 2004, which consist of 17 main categories plus 27 low volume categories have been proposed. EPA is expected to release a proposed national AIM rule in early 1995, with the final rule implemented in 1996. Due to the delay, eight states have issued their own rules.

NPCA has formed the AIM Coatings Steering Committee that will track and monitor state rules; offer a model rule to states who show interest in writing their own VOC rules; and prepare to comment on Federal EPA (National) rule. Their overall goal is to pursue a consistent, uniform, and equitable approach to regulating AIM coatings.

MICHAEL A. WOLFE, *President-Elect*

## NORTHWESTERN—DECEMBER

### "New Developments in High-Solids Technology"

Mike Maters, of Hüls America Inc. presented President Mike Grivna, of Hirshfield's Paint Mfg. Inc., with the Hüls Gavel.

The members were informed that Technical Symposium Chairman Russ Clevenger, of Eastman Chemical Co., has selected "Formulating Techniques for the 21st Century" as the topic.

Educational Committee Chairman, Mustapha Bacchus, of The Valspar Corp., reported that he has been invited to serve on the Federation's Educational Committee. Mr. Bacchus is currently seeking new members for the Society's Educational Committee.

The evening's speaker, Keith Gosse, of McWhorter Technologies, presented "NEW DEVELOPMENTS IN HIGH-SOLIDS TECHNOLOGY."

Mr. Gosse stated that a polymer's molecular weight is determined by its performance properties. Lower molecular weights have lower viscosities for high-solids systems, but they lack in performance. He then introduced two products that overcome these performance obstacles.

According to the speaker, a nonaqueous air dry dispersion and a high-solids acrylic acid have been developed for general industrial coatings. Mr. Gosse also presented several VOC compliant coating formulations, including an aerosol and two-component urethane with hydroxy functionality.

JOSEPH J. MILLS, *Secretary*

## PACIFIC NORTHWEST (VANCOUVER)—NOVEMBER

### "Titanium Dioxide Update"

Manufacturing Committee Chairman Valerie Braund, of General Paint Ltd., reported that she is planning to initiate a manufacturers' lecture circuit. The other Society chapters agreed.

Dave Pasin stated that three of the four sections of the paint course have been revised, leaving one to complete.

Ed Linton, of Cloverdale Paint Co., requested that anyone with pertinent data for the archives submit the information to Dick Stewart, of Firestop Systems, Inc.

Bob Hopkins, of SCM Chemicals-Americas, delivered the evening's presentation titled "TITANIUM DIOXIDE UPDATE."

Kelvin J. Hugert, *Secretary*



Serving as Officers for the Winnipeg Section of the Northwestern Society for 1994-95 are (from left) — Secretary—Ron Chemerika, Program Chairman—Sandra Madray, Social Chairman—Mark Rathwell, Secretary—Cecilla Chong, and President—Dennis Adams.



## CDIC

### Active

- Favstritsky, Nick A.—Great Lakes Chemical Corp., West Lafayette, IN.  
 Frazier, Tim R.—Concrete Sealants, New Carlisle, OH.  
 Young, Thomas J.—PPG Industries, Inc., Powell, OH.

### Associate

- Barnett, Gary D.—Neyra Industries, Cincinnati, OH.  
 Eichinger, James A.—M.F. Cachet, Union, KY.

## CHICAGO

### Active

- Braun, Kevin G.—Technical Coatings, Melrose Park, IL.  
 Cleope, Maria S.—Morton International, Batavia, IL.  
 Cooper, Jerri L.—Finishes Unlimited, Sugar Grove, IL.  
 Faris, Ismail A.—McWhorter Technologies, Carpentersville, IL.  
 George, Mathew—McWhorter Technologies, Carpentersville.  
 Haines, Roger E.—McWhorter Technologies, Carpentersville.  
 Malhan, Ramesh C.—McWhorter Technologies, Chicago Hts., IL.  
 Mallisk, Julie R.—McWhorter Technologies, Carpentersville.  
 Nguyen, Binh D.—McWhorter Technologies, Carpentersville.  
 Studt, Jane M.—McWhorter Technologies, Carpentersville.  
 Traub, Lance M.—McWhorter Technologies, Carpentersville.

### Associate

- Hanson, Lisa N.—Ashland Chemical Co., Countryside, IL.  
 Long, Carl J. III—McWhorter Technologies, Carpentersville, IL.  
 Rahrig, Douglas B.—McWhorter Technologies, Carpentersville.  
 Roeder, James W.—TAB Chemicals, Inc., Chicago, IL.  
 Seline, Elizabeth A.—J.M. Huber Corp., Orland Park, IL.  
 Whittaker, James E.—Thor Chemical Inc., Norwalk, CT.  
 Wachala, Kathleen M.—Eastman Chemical, Frankfurt, IL.  
 Wray, Linda S.—TAB Chemicals, Chicago, IL.

## CLEVELAND

### Active

- Dolbinzno, Ted T.—The Glidden Co., Strongsville, OH.

73rd Annual Meeting  
 & 60th Paint Industries' Show

October 9-10-11, 1995

Cervantes Convention Center  
 St. Louis, MO

- Meade, Cynthia L.—Singleton Corp., Cleveland, OH.  
 Sabo, Lynn O.—The Sherwin-Williams Co., Cleveland.  
 Wickert, Frank A.—Tremco Inc., Cleveland.

### Associate

- Charvat, Robert A.—Charvat and Associates, Cleveland, OH.  
 Sabo, Edward—Specialty Chemical Sales, Lyndhurst, OH.

## DETROIT

### Associate

- Fillmore, Ronald L.—Dow Corning Corp., Plymouth, MI.

## LOS ANGELES

### Active

- Beardall, Carwin B.—Classic Paint, Prescott Valley, AR.  
 Dimas, Jesus M.—Dunn-Edwards Corp., Los Angeles, CA.  
 Haanstra, L. Lloyd—Deft Inc., Irvine, CA.  
 Mird, Gustavo J.—Ellis Paint Co., Los Angeles, CA.  
 Primmer, Robert H.—Behr Process Corp., Santa Ana, CA.

### Associate

- Campbell, Mary L.—SPC, Fullerton, CA.  
 Imfeld, Mark A.—Pacific Coast Chemicals, Los Angeles, CA.  
 Lukens, Clark E.—United States Testing, Los Angeles.  
 Mastrop, Niels, S.—World Minerals, Lompoc, CA.  
 Morris, Geoffrey H.—SPC, Fullerton.  
 Westfall, Ronald—SPC, Fullerton.

## LOUISVILLE

### Active

- Cromley, Steve J.—Courtaulds Coatings, Louisville, KY.  
 Crutcher, James W.—Kelley Technical Coatings, Louisville.  
 Eichinger, James A.—M.F. Cachet, Union, KY.  
 Greenwald, John R.—Alcoa, Newburgh, IN.  
 Hazelwood, Steve—Akzo Nobel Coatings Inc., Louisville.  
 Kaiser, John H.—Akzo Nobel Coatings Inc., Louisville.  
 Mettelle, Michael J.—Akzo Nobel Coatings Inc., Louisville.  
 Nolan, Steven R.—Courtaulds Coatings, Louisville.  
 Turnley, Peter—Courtaulds Coatings, Louisville.

### Associate

- Burdick, Kenneth—Chem-Materials Co., Cleveland, OH.  
 Clarkson, William F.—C&S Process Technology, Louisville, KY.  
 Harris, Janet L.—Akzo Nobel Coatings Inc., Louisville.  
 Hessian, Harry J.—Devoe Coatings Co., Louisville.

- Stitsinger, Donald R.—International Specialty Products, Glen Ellyn, IL.

## MONTREAL

### Active

- Mootoosamy, Camini G.—Nacan Products Ltd., Boucherville, Que.  
 Pedneault, Dominique—Can-Lak, Daveluyville, Que.

### Associate

- Martel, Johanne—L.V. Lomas Ltd., Dorval, Que.

## NORTHWESTERN

### Associate

- Meister, John S.—Union Carbide Corp., Lisle, IL.

## PHILADELPHIA

### Active

- Buckley, William O.—Air Products and Chemicals, Allentown, PA.  
 Burdinski, Robert E.—Air Products and Chemicals, Allentown.  
 Gaboury, Scott—Elf Atochem, King of Prussia, PA.  
 Jannicky, Peter J.—Damakos Inc., West Chester, PA.  
 Makor, Paul—Hüls America Inc., Piscataway, NJ.  
 Metz, Andrea J.—Rohm and Haas Co., Spring House, PA.  
 Schwartz, Joel—Air Products and Chemicals, Allentown.  
 Zaborsky, Herbert J.—Hamburger Color Co., King of Prussia.

### Associate

- Debourke, Scott—Whitford Corp., West Chester, PA.  
 Lane, Larry G.—Johnson Matthey Inc., West Chester.  
 Malloy, Charles F.—Nalco Chemical Co., Springfield, PA.  
 Maurer, John C.—Superior Materials, Garden City, NY.  
 O'Connor, J. Brian—McWhorter Technologies, Erha, NJ.  
 Sooby, Steven D.—Premier Mill Corp., Lawrenceville, NJ.  
 Subluskey, Lee A.—Magmar Associates Inc., Hockessin, DE.  
 Weaver, Gary—Hüls America Inc., Piscataway, NJ.  
 Wollenweber, Arthur P.—Zeneca Resins, Bedminster, NJ.  
 Yerger, Carl D.—Premier Mill Corp., Reading, PA.

## PITTSBURGH

### Active

- Hindash, Abba A.—PPG Architectural, Springdale, PA.  
 Troutman, Mark D.—Westinghouse Electric, Manor, PA.

# Future Society Meetings

## SOUTHERN

### Active

- Ayrer, Maurice A.—Thompson Minwax, Olive Branch, TN.  
Gerhard, David E.—Thompson and Formby, Olive Branch.  
Katres, Todd—Hüls America Inc., Atlanta, GA.  
Lynch, Frank T.—Aqua Borne Coatings Systems Inc., Jonesboro, GA.  
Morris, Richard H.—Delta Industrial Coatings, Arlington, TN.  
Paulin, Patrick P.—Erfurt Paint and Color Co., Inc., W. Palm Beach, FL.  
Sharma, Mahendra K.—Eastman Chemical Co., Kingsport, TN.  
Shubert, Randy—Shubert Paints, Tucker, GA.  
Taylor, Charles B.—Centurion Paints Supply Inc., Kissimmee, FL.

### Associate

- Carter, Sherry E.—CheMarCo., Inc., Marietta, GA.  
Wiley, Rosalyn—Dry Branch Kaolin Co., Dry Branch, GA.  
Fishman, Philip M.—Birmingham, AL.  
Graham, Victor W.—Deeks & Co., Inc., Nashville, TN.

## TORONTO

### Active

- Bailey, Jane H.E.—Rohm and Haas Canada, West Hill, Ont.  
Faldas, Filomeno—Sico Inc., Etobicoke, Ont.  
Hulme, Mike—L.V. Lomas Co., Brampton, Ont.  
Lawrence, Paul K.—Dominion Colour Corp., Etobicoke.  
Mainville, Alain—Tiger Drylac Canada, Guelph, Ont.  
Martinek, Miroslav—Rohm and Haas Canada, West Hill.  
Massillamany, Emil—Rohm and Haas Canada, West Hill.  
Meneghetti, Luigi—Gand-Ho Ink Co., Brampton.  
Oliphant, Ken—Rohm and Haas Canada, West Hill.  
Ross, Curtis J.—Dominion Colour Corp., Etobicoke.  
Salazar, Christina—K-G Packaging, Etobicoke.  
Shrilliff, Geoff L.—K-G Packaging, Toronto, Ont.  
Vella, Tony—Canadian General Tower, Cambridge, Ont.

### Associate

- Cincinato, Eddie—Stochem Inc., Brampton, Ont.  
Dewitte, Claude M.—BFGoodrich Co., Waterloo, Ont.  
Heise, W.J.—L.V. Lomas Co., Brampton.  
Hulme, Mike—L.V. Lomas Co., Brampton.  
Judge, Lisa C.—Coatings Magazine, Oakville, Ont.  
Kay, Jennifer—Coatings Magazine, Oakville.  
MacFarlane, Steve—Dominion Colour Corp., Pickering, Ont.  
McKay, Gary B.—Hüls Canada Inc., Brampton.  
Nucara, Basilio—Dominion Color Corp., Etobicoke, Ont.  
Schertzer, Richard A.—Whittaker, Clark & Daniels, Inc., Rexdale, Ont.  
Sinclair, Paul W.—Pioneer Executive Consultants, Etobicoke.  
Taylor, Alan—Canadian General Tower, Cambridge, Ont.

## CDIC

- (Mar. 13)—Manufacturing Program.  
(Apr. 10)—CDIC 75th Anniversary.  
(May 8)—"EVALUATION OF NEW GENERATION COALESCING AGENTS FOR INDUSTRIAL ACRYLIC LATEXES"—Thomas M. Larson, Exxon Chemical Co.

## Chicago

- (Mar. 6)—"SOLVING PRACTICAL ADHESION PROBLEMS IN COATINGS WITH FUNDAMENTAL STUDIES IN SURFACE AND BULK EFFECTS OF ADHESION"—Kishan C. Sehgal, Union Carbide Chemical & Plastics Co., Inc.  
(Apr. 4)—"NEW TECHNOLOGY IN SMALL MEDIA MILLING"—Harry Way, Netzsch, Inc.  
(May 12)—Annual Awards Banquet.

## Cleveland

- (Feb. 21)—"FOAM & FOAM CONTROL AGENTS"—Andrew Romano, Drew Division/Ashland Chemical.  
(Mar. 21)—"USING KAOLIN PIGMENTS TO REPLACE CRYSTALLINE SILICA"—Thad T. Broome, J.M. Huber Corp.  
(Apr. 11)—Manufacturing Symposium.  
(Apr. 18)—Annual Meeting. "UNIQUE WETTING & DISPERSING ADDITIVES"—Edward Ort, BYK-Chemie.  
(May 4-5)—Educational Symposium.  
(May 16)—Awards/Past-Presidents/Spouses' Night.

## Golden Gate

- (Mar. 13)—"USE OF SMECTITE CLAY MINERALS IN WATERBORNE COATINGS"—Dwayne Siptak and Bob Briell, Southern Clay Products.  
(Apr. 17)—"EASILY DISPERSIBLE, LOW OIL ABSORPTION, OPACIFYING ORGANIC PIGMENTS ENCAPSULATING TITANATED LITHOPONE"—Michael Issel, Sino American Pigments.  
(May 15)—"HIGH-SPEED DISPERSION TECHNIQUES"—Rocky Courtain, Morehouse Industries, Inc.  
(June 19)—Manufacturing Committee Seminar.

## Los Angeles

- (Feb. 20-22)—Western Coatings Societies' Symposium and Show, San Francisco, CA.  
(Mar. 8)—Past Presidents' Night. "USE OF SMECTITE CLAY MINERALS IN WATERBORNE COATINGS"—Dwayne Siptak and Bob Briell, Southern Clay Products.  
(Apr. 12)—Bosses' Night. "EASILY DISPERSIBLE, LOW OIL ABSORPTION OPACIFYING ORGANIC PIGMENTS ENCAPSULATING TITANATED LITHOPONE"—Mike Issel, Sino American Pigments.  
(May 10)—Awards Night. "HIGH-SPEED DISPERSION TECHNIQUES"—Rocky Courtain, Morehouse Industries, Inc.  
(June 14)—Annual Meeting.

## Montreal

- (Mar. 1)—"ACCELERATED TESTING"—Dwight Weldon, KTA-Tator.  
(Apr. 5)—"RHEOLOGY MODIFIERS"—Bob Briell, Southern Clay Products.  
(May 3)—"ADHESION PROMOTERS"—Eastman Chemicals.

## New England

- (Feb. 16)—"THE EFFECT OF ASSOCIATIVE THICKENERS ON THE PROPERTIES OF WATER-BASED COATINGS"—David Bryant, Rheox.  
(Mar. 16)—Past-Presidents' Night. "NEW LATEX TECHNOLOGY FOR SEMI-TRANSPARENT STAINS"—Arthur Leman, Rohm and Haas Co.  
(Apr. 20)—Joint Meeting with New England Paint and Coatings Association. "NEW COALESCING SOLVENTS FOR WATERBORNE COATINGS"—Peter Doty, Dow Chemical Co.; and "COATINGS IN FORENSICS"—James Corby, FBI.  
(May 25)—"NEW CHEMISTRIES IN DEFOAMERS AND WETTING AGENTS"—Fred Lewchik, BYK-Chemie.

## Pacific Northwest (Puget Sound Section)

- (Mar. 15)—"USE OF SMECTITE CLAY MINERALS IN WATERBORNE COATINGS"—Dwayne Siptak and Bob Briell, Southern Clay Products.  
(Apr. 19)—"EASILY DISPERSIBLE, LOW OIL ABSORPTION OPACIFYING ORGANIC PIGMENTS ENCAPSULATING TITANATED LITHOPONE"—Mike Issel, Sino American Pigments.  
(May 4-6)—48th Annual Spring Symposium, Portland, OR.  
(May 17)—Joint Meeting with Puget Sound Paint and Coatings Association. "HIGH-SPEED DISPERSION TECHNIQUES UPDATE"—Rocky Courtain, Morehouse Industries, Inc.

## Pittsburgh

- (Apr. 10)—Joint Meeting with SSPC, PDCA, NACE, and PSCIT.  
(May 8)—"COLOR TRENDS FOR THE COMING YEAR"—PPG Industries, Inc. Past-Presidents' and Spouses' Night.

## Toronto

- (Mar. 6)—"RADIATION CURING TECHNOLOGY FOR THE 90S AND BEYOND"—Robert A. Lie Berman, Henkel Corp.  
(Apr. 10)—Technical Symposium.  
(May 8)—"Non-Toxic Anticorrosive Pigments in Aqueous Media"—Robert E. Snyder, A.R. Monteith (77) Ltd. (Toronto Society Technical Committee Presentation).

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## Design and Analysis of Experiments Volume 1: Introduction to Experimental Design

Written by: Klaus Hinkelmann and Oscar Kempthorne

Published by: John Wiley and Sons, Inc., 605 Third Ave., New York, NY 10158  
xvi + 495 pages, \$59.95

Reviewed by: Dave Sartori, PPG Industries, Inc., Allison Park, PA



This text is the first of two volumes that update Oscar Kempthorne's 1952 work of the same name. It is intended to serve as a textbook for a first-year graduate course in experimental design. As such, the book's orientation is very much towards the theoretical with essentially no emphasis on data-driven examples or case studies. While broad coverage to various types of designs is given, the main emphasis is on "error-control" designs (completely randomized designs, randomized blocks, and Latin square designs). The reader will also require an upper-level undergraduate grasp of linear algebra to successfully navigate the text.

The clear intent of the authors is to build the subject from first principles. Considerable discussion is given in the first two chapters on the need for experimental design and its place within the general setting and philosophy of the scientific method. Chapter 3 provides a concise description of the designs covered in greater detail in later chapters. This overview includes a general discussion of the difference between error-control designs, in which  $k$  treatments are compared, and treatment designs, in which the  $k$  treatments take on a factorial structure. It is noted in section 3 of this chapter that designs such as incomplete block designs for factorial experiments and split-plot designs combine ideas from both the error-control and treatment schemes. The authors conclude this chapter by saying of the designs discussed, "They are the building blocks of almost all designs, and it is the aim of this book to elucidate them in a rigorous way, emphasizing the statistical and mathematical aspects" (p. 59).

Indeed, this point is where the rigor takes hold—starting with a thorough overview of linear model theory in Chapter 4. Clearly the linear algebra needed is assumed as neither definitions nor an appendix of the essential concepts such as dimension, rank, orthogonality, and idempotency, are offered. This chapter goes well beyond the matrix development of ordinary least squares estimation into such topics as the Moore-Penrose Generalized Inverse, conditioned linear models, analysis of covariance models, ordered models, and nested models.

The authors point out that the analysis of all intervention studies is based on the concept of randomization, and Chapter 5 lays out in a qualitative manner the general philosophy of randomization. It is here that the contributions of Sir Ronald Fisher receive the most discussion. The theme of randomization is carried over into Chapter

6 where completely randomized designs are introduced. One of the more enlightening parts of this chapter is a simulation study that justifies the F-test as an approximation to the randomization test. Also starting with Chapter 6, the last section of each chapter is devoted to providing a brief description

(Continued on next page.)

## Introduction to Surface Chemistry and Catalysis

Edited by: Gabor A. Somorjai

Published by: John Wiley and Sons, Inc., 605 Third Ave., New York, NY 10158  
xxiv + 627 pages, \$59.95

Reviewed by: Robert F. Brady Jr., Naval Research Laboratory, Washington, D.C.

This book summarizes the current understanding of the properties of surfaces and interfaces between surfaces. Studies of surfaces at the molecular level conducted in industrial and academic laboratories during the past 20 years are evaluated and summarized. The characteristics and behavior of the molecules on a surface are shown to be the foundation of the macroscopic properties of the surface.

The first chapter gives an introduction to surfaces and to the techniques used to study them. The remaining seven chapters treat the structure, thermodynamics, dynamics, and electrical properties of surfaces, bonding at a surface, catalysis by surfaces, and mechanical properties of surfaces. Each of the chapters ends with a summary of important concepts, problem sets, and copious references. Solutions to most of the problems, and a complete subject index are included at the end of the book. Clear photographs and line drawings are used throughout. A weakness of the book, from the point of view of our industry, is that much of the discussion focuses on the solid—gas and solid—vacuum interfaces because most of the results of modern surface science investigations have come from such work. Nevertheless, the concepts unearthed in these studies are valuable for processes which concern us, such as absorption and pigment wetting.

The book is as much a handbook as a textbook, for 40% of its pages are devoted to tables of data. A particularly useful table decodes and explains the numerous acronyms used for the analytical techniques which have been developed for modern surface science. Data on the structure of the surfaces of pure metals and alloys is provided in several extensive, well-referenced tables.

The book provides many insights into industrial processes commonplace in the coatings industry, such as the absorption of resins onto pigments. Properties of interfaces such as adhesion, tribological behavior, friction, and lubrication are also covered in some detail. People concerned with the use of catalysts for resin synthesis will find value in the comprehensive discussion of materials used for catalysts and their preparation, deactivation, and regeneration. A case study of the absorption of hydrocarbons on platinum provides an instructive example. Those seeking coatings with easy-release properties will also find useful concepts throughout the book.

## Literature Review

Continued from previous page.

of how the topics covered in that chapter can be implemented using SAS®. No numerical examples are offered, however. Comparing several treatments is the subject of Chapter 7. Included in this chapter are brief descriptions of the more popular multiple comparison methods such as Bonferroni *t*-statistics, Tukey's Studentized Range Procedure, Duncan's Multiple Range Test, The Scheffé Procedure, and Dunnett's Test for comparisons with a control. Chapter 8, entitled "Use of Supplementary Information," motivates and describes the use of the analysis of covariance model. Chapter 9, "Randomized Block Designs," and Chapter 10, "Latin Square Type Designs," complete the discussion of the standard error-control designs.

Chapters 11 and 12 delve into factorial and response surface designs. Some treatment is given to the use of ideas from error control designs, such as blocking, in factorial and response surface settings. A feature of Chapter 11 that is uncommon in more data-driven texts is a discussion of confounding in three-level and mixed-level factorial designs. Chapter 12 also makes passing reference to mixture response surface designs and combined mixture-process variable designs. The final chapter of the book is dedicated to split-plot type designs, including some coverage of repeated measures problems.

One feature of the authors' style that is somewhat bothersome is the incessant use of acronyms. This was particularly noticeable in Chapters 6 and 7. A few examples include CRD (completely randomized design), EU (experimental unit), CWE (comparisonwise error rate), FWE (familywise error rate), and PFE (per family error rate). While one can surely appreciate that it is tiresome to repeatedly write (and perhaps read) "completely randomized design," acronyms are used in figure captions, section headings, and table titles. For example, the title of Table 6.6 on page 177 is "Values of  $\Delta^*$  to Determine Numbers of Replications in CDR\*." While I was reasonably certain that "CDR" should have been "CRD," I still found myself surfing back through the text to make sure I did not miss something.

There is little doubt, however, that this text will take its rightful place among the classic theoretical works in the field and will enjoy much deserved air time in graduate statistics programs. But the absence of numerical examples and emphasis on theory over application make this an unsuitable addition to the bookshelf of the industrial experimenter. For this purpose, I would direct the reader to Montgomery's *Design and Analysis of Experiments* or Box, Hunter, and Hunter's *Statistics for Experimenters*.



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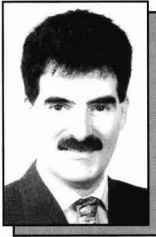
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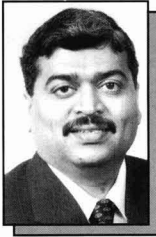
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**P. Figlioti Sr.**



**M. Sohoni**

**Peter Figlioti Sr.**, has been named Senior Project Chemist for McWhorter Technologies, Carpentersville, IL. Mr. Figlioti will be responsible for product evaluation and technical service for the powder coating resin line. He is a member of the Northwestern Society.

The company has also appointed **Milind Sohoni** to Project Chemist. In this capacity, Dr. Sohoni will work on the research and development of resins and curing agents for powder coatings. He is a member of the Northwestern Society.

In addition, **Joseph F. Schwan** has accepted the position of Senior Analytical Chemist. Dr. Schwan's new position entails supervision of the R&D Analytical Group in development of new analytical methods relating to resins and other non-routine analyses.

**James J. Egizio** was promoted to National Sales Manager, Industrial Coatings for Spraylat Corp., Mt. Vernon, NY. In his new role, Mr. Egizio will be managing the sales force handling all of the company's industrial coatings.

**David Philbrook** has been named Western Regional Sales Manager for Ultrablend Systems, Inc. In this position, Mr. Philbrook will coordinate marketing and sales activities in 11 western states, Alaska, Hawaii, and western Canada and Mexico. Prior to joining Ultrablend, he served as Marketing Consultant with Titan Corp. and Supreme Chemical.

**Leonard C. Afremow**, Vice President of Marketing and Research of Dexter Corp.'s Packaging Products Division, Waukegan, IL, has announced his retirement. He was with Dexter for 14 years.

Mr. Afremow, a member of the Chicago Society, was Chairman of the Technical Committee that wrote the first edition of the Federation's "Infrared Spectroscopy and its Use in the Coatings Industry."

In other news, **Stephen R. Postle** has been appointed Director of Research and Development. Mr. Postle was previously Director of Research and Development, the Americas. While retaining his previous duties, Mr. Postle will assume responsibilities for global technical strategy, including coordinating resource allocation and project selection of four regional laboratories in conjunction with regional General Managers.

The company also named **Gary Niceswanger** Manager of Financial Reporting. Mr. Niceswanger's new role embraces the preparation, consolidation, and analysis of monthly, quarterly, and annual financial statements and maintenance of the division's accounting records.

**Charles G. Munger**, of C.G. Munger Associates, Fallbrook, CA, has been presented with the Steel Structures Painting Council's (SSPC) 1994 Honorary Life Member Award. The award, which is only the second of its kind ever presented, recognizes Mr. Munger's years of service to the SSPC and the protective coatings industry as an author, editor, and industry spokesperson.



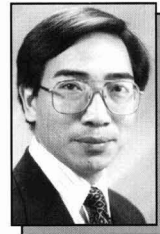
Mr. Munger is also a member of the Los Angeles Society and a NACE Past-President. Special Conference Awards were presented to **Reginald D. Coots**, Houston Lighting and Power; **Tony Liu**, Benjamin Moore & Co.; and **Steven P. Roetter**, Tank Industry Consultants.

In addition, awards were presented to **Daniel P. Adley**, KTA/SET Environmental, and **Clive Hare**, Clive H. Hare, Inc.

SSPC also presented its Executive Director, **Bernard R. Appleman**, with a special award recognizing his achievements and dedication to SSPC. Mr. Appleman is also a member of the Pittsburgh Society.

**Rohm and Haas Co.**, Philadelphia, PA, has appointed persons to two new positions in the company's Architectural Coatings Sales organization. **Jacalyn L. Thompson** was named Technical Sales Trainee. In this capacity, she will support technical communications, including customer correspondence, while in training at the company's research facility in Spring House, PA.

**Stephanie J. Smith** was promoted to her first field assignment from Technical Sales Trainee. She will assume sales responsibilities in the Midwest.



**W.Q. Xu**

**Wen-Qing Xu** has joined the Environmental Products Group of Johnson Matthey's Catalytic Systems Division, Wayne, PA, as a Staff Scientist. Mr. Xu will oversee the development of new catalysts for destruction of VOCs at low temperatures.

**Thomas M. Von Lehman** has been appointed Vice President of Purchasing and Distribution for PPG Industries, Pittsburgh, PA. Mr. Von Lehman, who joined PPG in 1980, was formerly Director of Corporate Planning.

**Michael C. Beatty** has accepted the position of Business Manager for the Organics Division of Hilton Davis, a unit of Freedom Chemical Co., Cincinnati, OH. Mr. Beatty was formerly Product Manager responsible for the aliphatic and aromatic raw materials used in polyurethane coating applications at Miles Inc., Pittsburgh, PA.

**Robert P. Serretti** has been elected Vice President of Manufacturing and Engineering of the Oleo/Surfactants Group at Witco Corp., Greenwich, CT. In this capacity, Mr. Serretti will be responsible for the manufacturing and engineering services at the company's 14 North America plants which manufacture oleochemicals and surfactants.

**Engelhard Corp.**, Iselin, NJ, has named **Daniel W. Parker** President of the Surface Technologies business unit. Mr. Parker was formerly President of General Plasma, Inc., a supplier of thermal spray coating technologies to the automotive, diesel engine, aircraft, land-based turbine, and medical implant markets, which was acquired by Engelhard in 1994.

**Brian Myerscough** has accepted the position of General Manager of Americas Operations for Datacolor International, Lawrenceville, NJ. Headquartered in Charlotte, NC, he will direct operations of North and Latin American technical sales, support, and service organizations.

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The National Decorating Products Association, St. Louis, MO, has elected its 1994-95 Board of Directors. Elected as Officers are: President—**Ed Makarenko**, Fargeys Paint & Wallcoverings Ltd.; Vice President—**Arlan Hatloe**, Hatloe's Decorating Center Inc.; Treasurer—**Gary Fahndrich**, West Side Decorating Center Inc.; and Advisory Committee—**Jan Satrom**, Grand Forks Glass & Paint Co.; and **C. Robert Perschon**, Perschon Paint & Wallcovering.

New Directors on the Board include: **Gary Hume**, Guyton Paint Products; **Stephen Ozab**, Goleta Valley Paint; **Carl Reineke**, Reineke Decorating Center; and **Robert Vernier**, Cy Vernier Paint Co. Inc.

**Frederick A. Shinnars**, Group Vice President responsible for worldwide oleochemicals and surfactants operations for Witco Corp., Greenwich, CT, has accepted the position of Corporate Officer. Mr. Shinnars has also served as Vice President of GE Silicones, President of GE Plastics Japan, and Vice President of Operations of Borg Warner.

OM Group, Inc., Cleveland, OH, has named **Lawrence A. Dominey** Vice President of Research and Development. In this position, Mr. Dominey will be responsible for expansion of existing business sectors with emphasis on product improvements and new product development. He will focus on specialty areas such as PVC stabilizers, catalysts, and advanced battery materials.

Degussa Corp., Roswell, GA, has appointed **Thomas J. Lahey** Regional Sales Representative of its Pigment Group, Carbon Black Div. Mr. Lahey will be responsible for the sales of carbon black and iron blue pigments in the southern United States. In addition to maintaining existing accounts, he is charged with the development of distributor sales.

The Adhesive and Sealant Council has elected **Allan C. Buchholz**, the Director of Research and Development for the Darworth Co., Simsbury, CT, to its Board of Directors. Dr. Buchholz is a member of the New England Society as well as the American Chemical Society, the American Society for Testing and Materials (Caulk Committee), and the New England Coatings Association.

**James A. Eichinger** has accepted the position of Sales Representative for The M.F. Cachat Co., Cleveland, OH. He will maintain a satellite office for the company in Union, KY.

## Obituaries

**Keith Vander Hyde**, Past-President and Executive Committee member of the National Paint and Coatings Association, died on October 5, 1994. He was 66 years old.

Mr. Vander Hyde, former Chairman and Chief Executive Officer of Guardsman Products, Inc., Grand Rapids, MI, retired last year. He started his career at Guardsman in 1959 as a Department Manager. At the time of his death, Mr. Vander Hyde served as Guardsman's Chairman Emeritus.

He is survived by his wife, Barbara; two sons, Kip and Bill; three daughters, Betsy, Kristi, and Anne; and seven grandchildren.

**Frederick D. Rotar** passed away on November 18, 1994. Mr. Rotar was Director of Sales and Marketing for Union Process, Inc., Akron, OH.

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## Laboratory Apparatus

### Water Analyzer

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Circle No. 30 on Reader Service Card

### Thermogravimetric Analyzer

Shimadzu Scientific Instruments presents the TGA-51 Macro Thermogravimetric Analyzer. This instrument is designed for QA/QC in polymer and inorganic chemical industries. The unit is designed for easy switching between sample atmospheres, and measures sample weight changes as a function of temperature over time.

Circle No. 31 on Reader Service Card

### Spectrophotometers

Two new multi-angle spectrophotometers for measuring metallic colors are introduced. Minolta Corp.'s CM-512m1 and CM-512m2 are designed with three-angle illumination/single-angle viewing geometry for facilitating measurements of metallic color and pearlescent finishes. A true-angle function automatically positions the spectrophotometers perpendicular to the specimen surface regardless of the contour, reportedly improving accuracy and repeatability.

Circle No. 32 on Reader Service Card

### EDXRF Spectrometer

The ED<sup>2000</sup> energy dispersive X-ray fluorescence spectrometer has been developed for qualitative, full qualitative, and semi-qualitative analysis of plastics materials. This instrument, offered by Oxford Instruments, Inc., features simultaneous analysis of up to 80 elements for qualitative analysis or up to 50 elements for full qualitative analysis between Na<sup>11</sup> and U<sup>92</sup>.

Circle No. 33 on Reader Service Card

### Infrared Microscope

Spectra-Tech Inc., along with Brookhaven's National Synchrotron Light Source, Grumman Corp. Research Center, and the Carnegie Geophysical Institute, has installed a Spectra-Tech Irtm™ infrared microanalysis system to Brookhaven's synchrotron light

source, which produces infrared radiation about 100 times brighter than customary light sources. Reportedly, with this combination, spectra from samples 5 mm in diameter were obtained with signal-to-noise comparable to conventional measurements with 100 mm apertures.

Circle No. 34 on Reader Service Card

### Viscosity Measurement

An in-line viscosity measurement system for the paint and coatings industry is available. Cambridge Applied Systems' viscometers operate with one moving part—a polished stainless steel piston whose movement is resisted by the viscous flow of fluid around it. Piston travel time is measured and displayed in units of absolute viscosity.

Circle No. 35 on Reader Service Card

### Air Toxics Analyzer

Information is available on the Air Toxics Analyzer I monitoring system for air toxics in canisters or on-line air streams. The analyzer includes a cryogen-free focusing trap integrated into the AutoSystem gas chromatograph. This Perkin-Elmer analytical tool also features automated analysis for air streams and up to 16 canisters and simultaneous analysis of polar and nonpolar components.

Circle No. 36 on Reader Service Card



## Testing Equipment

### Accelerated Weathering

The Emmaqua® and Emmaqua®+ are designed to perform the equivalent of three years of weathering testing in six months. Ten highly reflective and specially coated mirrors in this Heraeus DSET mechanism concentrate natural sunlight onto specimens with an intensity of about eight suns. The device tracks the sun and exposes specimens to the full spectrum of sunlight.

Circle No. 37 on Reader Service Card

### Testing Apparatus

A source directory of paint testing apparatus and instruments called for in the ASTM *Annual Books of Standards*, Section 6, Volumes 06.01, 06.02, and 06.03 on paints, coatings, and allied materials has been assembled. This eight-page complimentary pamphlet from Paul N. Gardner Co., Inc. lists apparatus and instruments called for in more than 300 specifications.

Circle No. 38 on Reader Service Card



## Sealants/Caulks

### Fluoroelastomer Adhesives

Properties, uses, and technical specifications for PLV 2000 and PLV 2100 fluoroelastomer adhesives and sealants are detailed in a press release. Pelmor Laboratories, Inc.'s two-part liquid products have been designed for resistance to harsh environments. The data sheet describes characteristics, industrial uses, physical properties, typical values, and application data for these adhesives.

Circle No. 39 on Reader Service Card



## Raw Materials

### Coating Surfactants

A brochure describes typical properties and appropriate paint and coating applications for a range of surfactants. "Witflow® Surfactants for Paints and Coatings" presents data on 22 surfactant grades for more than 12 applications. A list of causes and cures of 20 common coatings defects is also featured in this full-color Witco Corp. publication.

Circle No. 40 on Reader Service Card

### Pigment Grind Aid

A new pigment grind aid designed to promote pigment wetting and facilitate the dispersion of inorganic and organic pigments used in architectural, graphic arts, and industrial coatings applications is available from Air Products and Chemicals, Inc. Reported attributes of Surfynol® CT-324 include high pigment solids at optimal working viscosities, particularly in pigment slurries and bases, and a low dispersing agent requirement.

Circle No. 41 on Reader Service Card

### Antifoam Emulsions

GE Silicones introduces two new solvent-free, water-based antifoam emulsions. AF9010 and AF9030 are designed to be effective at very low concentrations, and can be used in both acid and alkaline systems. The antifoams are reportedly easily dilutable and fast-acting, and can be used in a variety of industries, such as paints and coatings, chemical processing, pharmaceuticals, agricultural chemicals, wastewater, food processing, and pulp and paper.

Circle No. 42 on Reader Service Card

## Epoxy Resin

D.E.R.® 353 diluted bisphenol-A/bisphenol-F epoxy resin is now available from Dow Plastics. This resin, a blend of bisphenol-F and bisphenol-A epoxy resins with alkyl glycidyl ether reactive diluent, is designed for crystallization resistance, low viscosity, and chemical resistance. Other reported features include higher filler loading, facilitated self-leveling formulations, improved wet-out, and resistance to aromatic, chlorinated, and ketone solvents.

Circle No. 43 on Reader Service Card

## Acrylic Copolymer

Carboset® CR 714, an acrylic copolymer emulsion from The BFGoodrich Co., is available. This product is designed to produce clear wood finishes with chemical, water, and print resistance. This thermoplastic polymer can be used to replace catalyzed varnishes and nitrocellulose lacquers, and is based on multiphase polymer technology to provide hardness at lower film-forming temperatures, purportedly reducing coalescent demand and VOCs.

Circle No. 44 on Reader Service Card



## Equipment

### Ball Mill

Paul O. Abbé Inc. has announced the availability of a new line of microprocessor-controlled ball mills. The heat sensitive product mills operate on the basis of a tumbling action which generates little heat. Particle size can be controlled by varying milling time and size of the grinding media in these mills, which can be appropriate for materials requiring minimal contamination.

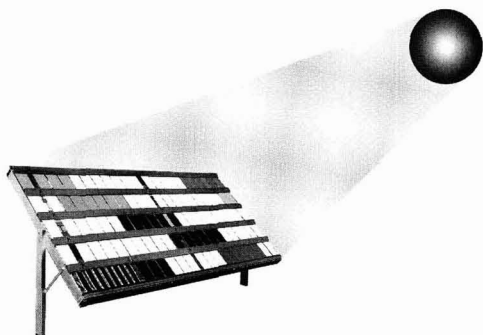
Circle No. 45 on Reader Service Card

### Agitator

A siphon agitator for closed-head drum mixing is available from Graco Inc. The Twistork™, which can run at speeds up to 800 rpm, features a helical blade that lifts thick solids from the bottom and keeps them in suspension for a more consistent mix. A built-in siphon tube allows complete evacuation of materials during or following agitation.

Circle No. 46 on Reader Service Card

## Quality, Time Tested Equipment, To Meet Your Exposure Testing Needs



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Circle No. 218 on Reader Service Card



## Books/ Publications

### Regulatory Compliance

A comprehensive regulatory compliance manual is designed to help epoxy resin formulators and their customers to be informed of the laws that affect them. The 800-page document includes information on laws in such areas as worker safety and health, the Clean Air Act, state/community right-to-know, pollution prevention, hazardous waste management, and transportation of hazardous materials. The volume is available from The Society of the Plastics Industry, Inc.

Circle No. 47 on Reader Service Card

### Plastics Processing

A technical paper highlighting the experimental results of a study on lubricants used to improve the injection molding of plastic fittings for PVC pipe has been printed. The paper examines numerous approaches to achieving an acceptable balance of performance properties and cost in lubricant formulas which facilitate the injection molding process. AlliedSignal Inc. can provide more information or copies of this literature.

Circle No. 48 on Reader Service Card



## Waterborne Systems

Shell Chemical Co. has developed a binder of literature on its line of resins for ambient cure waterborne coatings. Included are sections on formulating with waterborne systems, waterborne starting point formulations, waterborne product data sheets, epoxy resin dispersion handling and storage, and waterborne special topics. A waterborne products guide and selector guide to waterborne systems are also included in the "Shell Waterborne Systems for Ambient Cure Coatings" binder.

Circle No. 49 on Reader Service Card

## Powder Coatings Companies

A brochure entitled "Profiles of North American Powder Coatings Companies" discusses the size, growth, and composition of the powder coatings market, including coating manufacturers, equipment manufacturers, and raw materials suppliers. The P.D. Lovett & Co. publication profiles more than 140 companies, and includes a listing of products offered, manufacturing locations, and tables summarizing sales and market participation.

Circle No. 50 on Reader Service Card

## UV/EB Curing Manual

UV/EB curing is examined in a manual which is updated periodically. The publication contains a product catalog, product bulletins, application bulletins, starting point formulations, technical papers, safety and handling information, and a glossary of UV/EB curing terms. The guide also serves as a comprehensive reference to Sartomer Co.'s specialty oligomers, urethane acrylates, epoxy acrylates, monomers, and photoinitiators.

Circle No. 51 on Reader Service Card

## Functional Training

A booklet investigates the systematic application of functional training to assist manufacturing workers in becoming problem solvers, technical writers, and trainers. The Manufacturing Technology Strategies publication describes various ways systematic functional training has been used to solve a range of business problems through such methods as creating high performance teams, certifying employees to meet third-party standards and requirements, and conducting job analyses.

Circle No. 52 on Reader Service Card

## Controllers

A 40-page catalog describes Athena Controls' temperature, process, and power controllers for its line of microprocessor and analog instruments. Also described in the publication are single-board, nonindicating

temperature controllers for embedded controller applications and SCR power controls and contactors. Complete technical specifications are provided, along with a comprehensive glossary and tutorial on control fundamentals.

Circle No. 53 on Reader Service Card

## Coatings Topics

ASTM has released information on a number of books that are currently available. Volumes have been printed on such subjects as atmospheric analysis, biomedical engineering, composites, construction materials and engineering, environmental assessment, metals, petroleum products, and quality control. A new software program on benchmarking is also obtainable.

Circle No. 54 on Reader Service Card



## Drum Packer/Crusher

TeeMark Corp. introduces its drum packer/crusher series DPC85, which will crush drums up to 110-gallon capacity and compact material inside 85- and 55-gallon drums. A two-speed hydraulic pump powers the cylinder to an 85,000 pound crushing force, and the ram is automatically cycled by an all-hydraulic control valve.

Circle No. 55 on Reader Service Card

## Remote Sampling

Spectra-Tech, Inc. introduces infrared fiber optics to analyze samples at remote locations or samples that are not the optimum shape or size for traditional sampling accessories. Applications include hazardous environments, process and reaction monitoring, restricted access sampling vessels, non-destructive sampling, oddly shaped samples, and high temperature or pressure environments.

Circle No. 56 on Reader Service Card

## Spray Gun

A gravity feed spray gun that complies with air quality regulations reportedly atomizes and sprays as quickly as conventional air spray. A low-volume 93P air nozzle designed for automotive use allows for the spraying of basecoats, clearcoats, waterborne, and high-solids. Binks Manufacturing Co.'s M1-G HVLP gravity speed spray gun reportedly offers fast application speeds as well as material savings.

Circle No. 57 on Reader Service Card

## Pressure Cell

Rheometrics, Inc. introduces a new high-pressure cell for dynamic stress rheometer models SR-200 and SR-500. This accessory enables users to conduct rheological tests in both dynamic and steady test modes at pressures between 0 and 750 psi (50 bar) and temperatures ranging from -40 to 150°C. It is possible to test materials that are used or processed under temperature or pressure extremes with this cell.

Circle No. 58 on Reader Service Card

## Can Crusher

The CanDoo! can crusher model CC6 is designed to crush cans and pails up to six gallon capacity. Cycle time is about 30 seconds in this FabTech Corp. crusher, and the cycle is fully mechanical. An automatic bottom piercer eliminates the need to drain partially filled cans, and the punctured bottom vents internal pressure during crushing and drains residual liquids into the lower chamber.

Circle No. 59 on Reader Service Card



## Manufacturing Software

Imprimis™, an object-oriented, graphical user interface-based, client/server manufacturing software product, will be released in March 1995 by Tangible Vision, Inc. This integrated real-time system provides information on areas such as improving customer service, increasing productivity, enhancing quality, reducing costs, and increasing profitability. Other capabilities include system administration, inventory and warehouse control, purchasing, and sales order processing.

Circle No. 60 on Reader Service Card

## Color Analysis

Two new systems designed to measure products that are intentionally manufactured with nonuniform color and appearance are introduced. HunterLab, Inc. manufactures the On-Line Color Vision System, which consists of a sensor mounted over the production line and a separate equipment enclosure; and The Bench-Top Color Vision System, a self-contained quality control system. These systems use high performance video technology to capture, digitize, and analyze images.

Circle No. 61 on Reader Service Card

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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

(Mar. 21-22)—“Formulating for the New Clean Air Act.” Seminar sponsored by the Professional Development Committee. Cleveland Airport Marriott, Cleveland, OH.

(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 Coral Beach, Cancun, Mexico.

(June 20-21)—“Polymer Chemistry for the Coatings Formulator.” Seminar sponsored by the Professional Development Committee, Chicago, 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

(Mar. 8)—“Process Safety.” Chicago Society Manufacturing Seminar/Workshop. Marriott Hotel, Oak Brook, IL. (Stephen Brauer, Mozel Incorporated, 1110 Lake Cook Rd., Ste. 250, Buffalo Grove, IL 60089-1968; (708) 285-0065).

(Mar. 15)—“Clean Air & Technology.” Mini-Trade Show and Symposium sponsored by the Piedmont Society. Showcase on the Park, High Point, NC. (Richard Chodnicki, Van Horn, Metz & Co., 320 Townsontown Blvd., Ste. 205, Baltimore, MD 21204).

(Mar. 29-31)—Southwestern Paint Convention. Sponsored by Houston and Dallas Societies. Airport Hyatt Regency, Dallas, TX. (Benny Puckett, Kelly-Moore Paint Co., Inc., 301 W. Hurst Blvd., Hurst, TX 76053; (817) 268-3131).

(Mar. 29-July 5)—“Understanding the Basics of Coatings II.” Sponsored by the Joint Educational Committee of the New York Society for Coatings Technology and the Metropolitan New York Paint and Coatings Association. Course held Wednesday evenings for 15 consecutive weeks at Fairleigh Dickinson University, Hackensack, NJ. (Mildred Leonard, NYSCT Office, Rm. 208, 520 Westfield Ave., Elizabeth, NJ 07208; (908) 354-3200).

(Apr. 4)—“Compliant Coatings for 2000 and Beyond.” Sponsored by the Detroit Society. Michigan State University Management Center, Troy, MI. (Joe Lesnek, DSCCT, P.O. Box 2454, Riverview, MI 48192; (812) 428-9200).

(Apr. 11)—“Manufacturing Principles to Survive the 90s.” Seminar sponsored by the Cleveland Society and the Cleveland Paint & Coatings Association. Cleveland Hilton South, Cleveland, OH. (J.P. Walton, Jamestown Paint Co., 108 Main St., Jamestown, PA 16134; (412) 932-3101 or fax: (412) 932-5147).

(Apr. 19)—“Spectrum of Coatings Science.” Sponsored by the Louisville Society. Executive Inn, Louisville, KY. (Ilona DuVall, Red Spot Paint Co., 1107 Louisiana Ave., Evansville, IN 47711; (812) 467-2337).

(Apr. 19-21)—Southern Society Annual Meeting. Hyatt Regency, Savannah, GA. (Wayne West, Thompson & Formby, Inc., 10136 Magnolia Dr., Olive Branch, MS 38654).

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## 1995 FSCT SPRING WEEK TECHNICAL SEMINAR



“COATINGS IN THE AMERICAS:  
REGULATORY AND TECHNOLOGICAL ISSUES”

MAY 17-18, 1995

FIESTA AMERICANA CORAL BEACH  
CANCUN, MEXICO

(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, 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).

## OTHER ORGANIZATIONS

### 1995 — North America



(Mar. 5-8)—"Pigment Dispersions: Science and Technology." Orlando, FL. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(Mar. 6-8)—"Advances in Polymer Colloids/Emulsion Polymers." Orlando, FL. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(Mar. 6-8)—"Fundamentals of Adhesion." Orlando, FL. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(Mar. 6-8)—"Introduction to Polymer Colloids/Emulsion Polymers." Grosvenor Resorts, Orlando, FL. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(Mar. 8-10)—"Computer Modeling of Polymer and Solid Interfaces." Orlando, FL. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(Mar. 8-10)—"Polymer Blends and Alloys: Phase Behavior, Characterization, Morphology, and Alloying Technology." Orlando, FL. (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(Mar. 9-12)—"Supervisor/Competent Person for Deleading of Industrial Structures." Course sponsored by the Steel Structures Painting Council (SSPC). Palmer House, Chicago, IL. (Dee Boyle, SSPC, 4516 Henry St., Ste. 301, Pittsburgh, PA 15213-3728).

(Mar. 13-15)—Eighth Annual Lead Paint Abatement and Removal Conference. Sponsored by the Steel Structures Painting Council (SSPC). Palmer House, Chicago, IL. (Dee Boyle, SSPC, 4516 Henry St., Ste. 301, Pittsburgh, PA 15213-3728).

(Mar. 13-15)—"Second North American Research Conference on Stabilization and Degradation of Polymers." Hilton Head, SC. Sponsored by the American Chemical Society, Polymeric Materials: Science and Engineering (A.V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(Mar. 20-24)—"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).

(Mar. 26-31)—"Corrosion '95." Annual conference sponsored by National Association of Corrosion Engineers (NACE) International. Orange County Convention Center, Orlando, FL. (NACE International, P.O. Box 218340, Houston, TX 77218-8340).

(Apr. 2-7)—38th Annual Technical Conference sponsored by Society of Vacuum Coaters (SVC). Chicago Marriott Downtown, Chicago, IL. (SVC, 440 Live Oak Loop NE, Albuquerque, NM 87122-1407).

(Apr. 5-7)—10th Annual Conference sponsored by Architectural Spray Coaters Association (ASCA). Doral Ocean Beach Resort, Miami, FL. (ASCA, 230 W. Wells St., Ste. 311, Milwaukee, WI 53203).

(Apr. 18-19)—35th Annual Washington Paint Technical Group Spring Symposium. Ramada Hotel—Tysons Corner, Falls Church, VA. (Mark Padow, NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005-6272).

(Apr. 23-25)—ISCC Annual Meeting. 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)—"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 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)—"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. Knauss, 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).

(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 39406-0037).

(June 25-29)—"Coatings 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 17540-0056).

(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. 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. 20-22)—"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)—"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. 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).

(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. 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. 26-28)—"Surcon '95." Biennial International Conference sponsored by the Oil & Colour Chemists' Association. London, Heathrow. (Chris Pacey-Day, 967 Harrow Rd., Wembley HA0 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).

## 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, Daiichi Naka-Bldg. 4F, 3-4 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

(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

(Mar. 14-16)—"European Coatings Show '95." Congress and Exhibition. Exhibition Centre, Nuremberg, Germany. (Vincentz Verlag, Postfach 62 47, D-30062, Hannover, Germany).



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# Humbug from Hillman

**M**y thanks to Bob Link, who sent me some more of Richard Lederer's garbled English collection. This time from Lederer's *Anguished English*. In this group, Lederer gives the school kids a break and concentrates on "an array of adult and adulterated examples."

—Calf born to farmer with two heads.

—Yoko Ono will talk about her husband, John Lennon, who was killed in an interview with Barbara Walters.

—After years of being under a pile of dust, Chester D. Thatcher found all the old records of the Bangor Lions Club at the Bangor House.

—Some sources said shortly after his death, Mao Tse Tung had expressed a wish that his body be cremated.

—Locked in a vault for over 50 years, the owner of the jewels has decided to sell them.

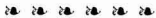
—The patient was referred to a psychiatrist with a severe emotional problem.

—Edwin Newman, author of two Book-of-the-Month Club books on the abuse of language, hinted in a speech to nearly 1,300 persons in the Memorial Theater that efforts to improve language may be the result of attacks on pompous, weird language such as his.



**A** little more than a year ago, Eric Simon suggested that I read *The Monkey's Wrench* by Primo Levi and possibly recommend it to my readers. I did both.

I have heard from Eric again and this time it was with reference to Primo Levi's *The Periodic Table* and the chapter entitled "Chromium." There is so much hilarious humor in Levi's description of experiences in the manufacture of synthetic varnishes and chromate based anti-rust paints that I could scarcely do the chapter justice with excerpts. I can only urge my readers to head for the nearest book store or library and secure a copy of *The Periodic Table*. You will thank Eric for the trip.



**W**illy C.P. Busch sent me a copy of a column by one Paul Harasim (sic) of the *Houston Post*. Highlighted in the article was a story told by a senior state district judge, Bill Hatten:

An 80-year old man decided to visit a house of ill repute. After knocking on the door, he was greeted by the madam.

"Old man," she said, looking at the stooped figure before her, "you'd better get out of here. You've already had it."

"Well," replied the old man, "if I had it, then what do I owe you?"

Dear Willy,

On behalf of all the vibrant "octos," I suggest you write to the old coot and tell him to raise that age to 90 before we sue him!!!



**Y**ou've seen Sid Lauren's name in the column frequently. Well, I'm very happy to welcome Sid's daughter, Barbara, to the column again. It seems that *Washington Post* columnist Bob Levey, from time to time, features "wordmanship" contests. For example, on May 6th, he announced the winners who responded to his, "You are thinking of dumping your significant other, but S.O. dumps you first. This phenomenon is called . . . . ."

The winner ..... Dearjohnimo  
Some runners up ..... One Dumpmanship  
Pre-dumptive Strike  
Seren-dumpity  
Beaumerang  
Frontal Dumpotomy



In his October 7th column, Bob Levey announced the winners to . . . . . "Children have the nasty habit of leaving their dirty clothes not in the hamper, not in the washer, not in the incinerator, but in a scraggly heap in the middle of the floor. This heap is called . . . . ."

The winner ..... Pedopile  
Some runners up ..... Messdeminor  
Tyke's Peak  
Kiddie Litter  
The Mother Load  
Frock Pile  
Habertrashery



**A**nd speaking of contests! Bob Athey found this "Mental Illness Exam." The following grocery list was written by a certifiable lunatic. Score yourself.

- (a) Grey V (That's a giveaway, folks)
- (b) Tighters
- (c) Uncle Toms
- (d) Glee Decks
- (e) Reef Range Czich Inn (Answers at bottom; no cheating, please)

Zero right—(Can't understand any) You're sane!! Congratulations.

One right—You talk to yourself when people aren't watching.

Two right—You talk to yourself when people ARE watching.

Three right—You think you're Elvis.

Four right—You're nuts.

Five right—You're sick—SICK.

Answers: (a) Gray, (b) Frottoes, (c) Tomatoes, (d) Kleenex, and (e) Free range chicken.

—Herb Hillman, *Humbug's Nest*, P.O. Box 135, Whitingham, VT 05361.



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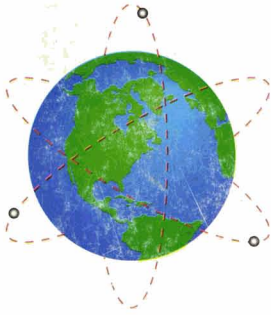
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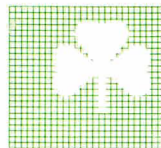
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