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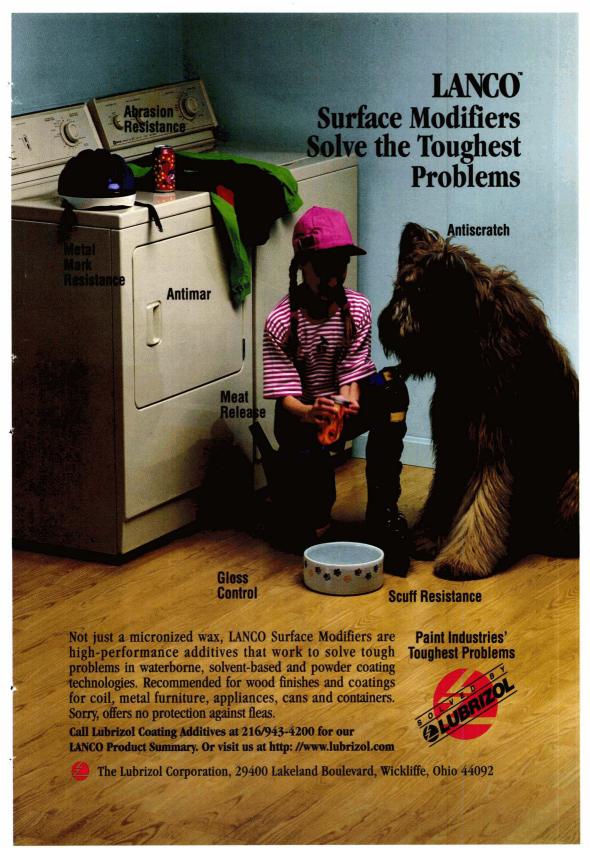


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SEPTEMBER 1997

VOL. 69, NO. 872

Technical Focus

- PALS Free Volume Study of Dry and Water Saturated Epoxies—M.M Madani, R.R. Miron, and R.D. Granata
 The purpose of this paper is to characterize the free volume properties as determined by positron annihilation lifetime
 spectroscopy (PALS) of two type of epoxies in respect to their behavior as protective coatings and their mixtures and
 the effects of three corrosion inhibitors.
- 84 Listing of Resin Suppliers to the Coatings Industry

Technical Articles

- Interfacial Studies of Crosslinked Polyurethanes; Part I. Quantitative and Structural Aspects of Crosslinking Near Film-Air and Film-Substrate Interfaces in Solvent-Borne Polyurethanes—A.M. Kaminski and M.W. Urban

 The authors present the criteria whereby the degree of cure of polyurethane coatings can be detrmined using FTIR.
- **69** Study of Anionic Polyurethane Ionomer Dispersant—Y.S. Huang et al.

 This paper describes the synthesis of six anionic polyurethane ionomers and their evaluation as dispersants in an acrylic baking enamel.
- 77 Influence of Crosslink Density, Glass Transition Temperature and Addition of Pigment and Wax on the Scratch Resistance of an Epoxy Coating—J. Lange, A. Luisier, and A. Hult The scratch resistance of a series of cationically cured epoxy coatings with varying crosslink densities is investigated.

Federation News

- 21 ICE '97—Preliminary Program/Housing/Registration Information
- 38 Current List of ICE '97 Exhibitors with Booth Numbers
- **40** ICE '97 Floor Plan of Exhibitors



Departments

JCT Guide for Authors	4	Product Overview—Lab Apparatus	94
Comment		People in the News	95
Technical Abstracts	8	Obituary	96
Industry News	11	New Products	97
Meetings Update	16	Calendar of Events	100
Regulatory Update		Humbug from Hillman	106

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GENERAL

The JOURNAL OF COATINGS TECHNOLOGY is published monthly by the Federation of Societies for Coatings Technology for its membership of approximately 7,000 in 26 Constituent Societies in the United States, Canada, Great Britain, and Mexico. The JOURNAL is devoted to the advancement of knowledge in the science and technology of surface coatings, the materials comprising such coatings, and their use and performance.

The Editors invite submission of original research papers, review papers, and papers under the special headings *Open Forum* and *Technology Forum*, and *Letters to the Editor*. All manuscripts will be assumed to be previously unpublished writing of the authors, not under consideration for publication elsewhere. When review papers contain tables or graphs from copyrighted articles, the authors will be required to obtain permission for use from the copyright holders. When the organization with which the authors are affiliated requires clearance of publications, authors are expected to obtain such clearance before submission of the manuscript. Papers presented to associations other than the Federation must be released by written communication before they can be considered for publication in the JOURNAL OF COATINGS TECHNOLOGY. Authors are obligated to reveal any exceptions to these conditions at the time a manuscript is submitted.

The JOURNAL OF COATINGS TECHNOLOGY has first right to the publication of papers presented at the Annual Meeting of the Federation and at local regional meetings or symposia of the Constituent Societies.

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

Letters to the Editor. The JOURNAL will consider for publication all correspondence relevant to the coatings industry and to the contents of the JOURNAL. When a letter concerns an article appearing in the JOURNAL, the original author is usually given an opportunity to reply.

...by Constituent Societies For Annual Meeting Presentation

Ten complete copies of the manuscript are required for committee review. The set of copies should be addressed to Mike Bell, Director of Educational Services, FSCT, 492 Norristown Rd., Blue Bell, PA 19422.

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Ten complete copies of the manuscript are required, and should be submitted to Mike Bell at the address previously listed. (For complete details, see "Roon Awards" section of the JOURNAL in the January 1997 issue.)

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In general, authors are advised to use the "Handbook for Authors" published by the American Chemical Society as a guide to the preparation of manuscripts (ACS, 1155 Sixteenth St., Washington, D.C. 20036). Another excellent reference work is "How to Write and Publish a Scientific Paper," by Robert A. Day (ISI Press, 3501 Market St., University City Science Center, Philadelphia, PA 19104).

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

Original Research Papers: Original research papers are the main technical content of the JOURNAL OF COATINGS TECHNOLOGY. Editors support the trend in scientific writing to a direct, less formal style that permits limited use of personal pronouns to avoid repetitious or awkward use of passive voice.

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

Open Forum: Topics for this category need not be research papers and may be non-technical in nature, dealing with any aspect of the coatings industry. The subject may be approached informally. Editors encourage submission of manuscripts, including articles dealing with business and policy issues, that constructively address industry problems and their solutions.

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

If a submitted paper consists of the text of a presentation made previously to a monthly or special meeting of a Society for Coatings Technology, or to another technical group, the name of the organization and the date of the presentation must be given. If someone other than the author of the paper made the presentation, this information, too, should be noted. Papers originally composed for oral presentation must be revised or rewritten by the author to conform to the style described in this guide.

Manuscripts should be typed with double spacing on one side of 8¹/₂ x 11 inch (22 x 28 cm) paper, with at least one-inch (2.5 cm) margins on all four sides. All paragraphs should be indented five spaces, and all pages should be numbered at the top center, or upper right corner.

Electronic submissions are requested as a supplement to the hard copies and original figures normally required. The text should be submitted on 3.5" disk formatted for IBM or Apple Macintosh (or compatible system). Text files should be saved in the word-processing format in which they were prepared. The file on disk MUST exactly match the accepted hard copy version. Graphics (figures, drawing, etc.) should be in a separate file. Submitted disks must be labeled with the author's name, paper title, computer platform type (e.g., IBM compatible), software (and version) used, and filenames. Complete instructions for electronic submission can be obtained from the Editor.

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The title should be as brief and informative as possible. Selection of titles that are key word-indexable is a helpful and recommended practice.

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A 75-100 word abstract must be part of the manuscript, and should be a concise statement of the key methods and findings or teachings of the work described in the paper. The abstract should not repeat the title or include reference numbers, nor should it duplicate the Conclusion or Summary.

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

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

Only as much review as is necessary to set the work in context and to make clear the significance of the work presented should be given as an introduction to the subject; the main burden for extensive background should be placed on the list of references.

Standard scientific and technical terminology should be used to convey clear and unambiguous meaning, but the use of technical jargon or slang should be avoided. Authors should bear in mind that the JOURNAL has an international audience, for many of whom English is a second, not native, language. Use of regional idioms or colloquialisms should be avoided. The use of obscure abbreviations is also discouraged. When appropriate, abbreviations should be made in parenthesis immediately following first mention of the term in the text, and then used alone whenever necessary.

Recent issues of the JOURNAL should be consulted for desired style and technical level.

Metric System

Metric system units should be used wherever applicable with the equivalent English units shown afterwards in parentheses. The ASTM Metric Practice Guide, E 380-72 (American Society for Testing and Materials, 100 Barr Harbor Dr., W. Conshohocken, PA 19428-2959) is a convenient reference.

Tables, Graphs, Drawings, and Photographs

Tables, rather than descriptive text, should be used only when they are genuinely helpful. Each table should be typed on a separate sheet, rather than included in the text, and appended to the manuscript. Each table should be numbered and have a descriptive caption. Tables should be referenced in the text (e.g., "See Table 1"). In numerical data in tables, numbers less than one should have a zero before the decimal point.

Graphs should be on good quality white or nonphotographic bluelined $8^{1}/2 \times 11$ inch paper. Each graph should be drawn on a separate sheet, numbered, and the captions listed on a copy of the original graph.

Drawings should conform to the guidelines given for Graphs and should be proportioned to fit the height-to-width ratio of the JOURNAL'S pages and columns.

Photographs should be sharp, clear, black-and-white prints no larger than 8×10 inches in size. Photos should be clearly labeled on the reverse side, taking care not to mar the image.

Color prints, slides, and graphics are generally not acceptable, but the selective use of color will be considered on a case-by-case basis when required for unambiguous presentation of scientific information. When illustrations are secured from an outside source, the source must be identified and the Editor assured that permission to reprint has been granted.

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Whenever possible, generic names should be used in preference to trade names. When trade names must be used to avoid ambiguity, and the name is a registered trademark, the symbol R, in a circle or parentheses, should be given immediately following, and the manufacturer listed as a footnote. In general, trade names should be used only in footnotes or in an appendix, rather than in the text.

If special nomenclature is used, include a nomenclature table giving definitions and dimensions for all terms.

Nomenclature of chemical compounds should conform to the style of *Chemical Abstracts* and the IUPAC rules. For oligomeric or polymeric materials, characteristics such as molecular weight, polydispersity, functional group content, etc. should be provided.

Equations

Equations must be typed, or written clearly, with equations numbered sequentially in parentheses to the right. If Greek letters are used, write out their names in the manuscript margin at the first point of use. Place superscripts^a and subscripts_b accurately. Avoid the use of superscripts in a manner that can lead to their interpretation as exponents.

Summary

The paper should be concluded with a summary which is intelligible without reference to the main text. The summary may be more complete than the abstract, listing conclusions drawn from the text. A well written summary can serve to inspire the busy reader to turn back to the paper, to read it thoroughly.

Acknowledgment

If used, it should follow the summary.

References

These should be listed in the numerical order in which they are cited in the text, and should be placed at the end of the manuscript. Names of authors may or may not be shown in the text with reference numbers. If possible, include titles of articles referenced in the literature. The following are examples of acceptable reference citations for periodicals, 1.2.3 books, 4 and patents.5

- (1) Pascal, R.H. and Reig, F.L., "Pigment Colors and Surfactant Selection," Official DIGEST, 36, No. 475 (Part 1), 839 (1964).
- (2) Davidson, H.R., "Use and Misuse of Computers in Color Control," JOURNAL OF COATINGS TECHNOLOGY, 54, No. 691, 55 (1982).
- (3) Stephen, H.G., "Hydrogen Bonding—Key to Dispersion?," J. Oil & Colour Chemists' Assoc., 65, No. 5, 191 (1982).
- (4) Patton, T. (Ed.), *Pigment Handbook*, Vol. 1, John Wiley & Sons, Inc., New York, 1973.
- (5) Henderson, W.A. Jr. and Singh, B. (to American Cyanamid Co.), U.S. Patent 4,361,518 (Nov. 30, 1982).

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COMMENT

Look Back with Us



History is hot! Far removed from the dry memorization of dates and locations of important events, today's study of history uses modern technology integrating photography, video, sound, and narrative—to bring the past to life.

At ICE '97 in Atlanta, on November 3-5, the Federation of Societies for Coatings Technology will take advantage of all these media—and more—to commemorate its 75th anniversary. This is one celebration that you can all enjoy!

For those attending ICE '97, activities begin on Monday morning, November 3, at the Georgia World Congress Center for the FSCT Annual Meeting Opening Session. Reflecting the convention theme, "75 Years: Tradition . . . Discovery . . . Opportunity," the Opening Session presentations will lead the audience on a fascinating trip back to the organization's beginnings, highlighting significant milestones in its growth, and revealing its plans for the future. Daniel Burrus, popular science forecaster, will preview innovative technologies and predict the impact these technologies will have on how we live and work.

Throughout its history, the FSCT's development has reflected the interests and concerns of the industry it serves. No anniversary celebration would be complete without an acknowledgment of the relationship between the Federation and the coatings industry. ICE '97 participants can visit the FSCT "History Walk" in the registration area of the Georgia World Congress Center. This attractive historical display, contributed to by ICE Exhibitors and FSCT Constituent Societies, will feature the equipment, instruments and artifacts used by the industry in days gone by. When was the last time you saw a one-ton stone mill?

One of the most exciting changes to debut at this year's Federation event is the ICE '97 Convention Guide. This newly expanded tabloid-sized publication will be fully distributed at ICE functions, throughout the show floor, and at headquarters hotels. In addition to all of the essential details regarding ICE '97, the Convention Guide will offer readers articles by key figures in the FSCT, leading industry suppliers, as well as contributions by Constituent Societies and Exhibitors.

Of course, observance of the Federation's anniversary is not limited to those attending the events in Atlanta. The *Convention Guide* will be mailed to all members in November. We invite you to share in the celebration as we recall the milestones achieved in the past and anticipate the Federation's role in the industry's next 75 years.

Patricia D. Ziegler Director of Publications

Technical Abstracts

Spanish translations provided by Marina Estevez, Technical Chief, Institute Mexicano de Técnicos en Pinturas y Tintas (ANAFAPYT)

PALS Free Volume Study of Dry and Water Saturated Epoxies—M.M. Madani, R.R. Miron and R.D. Granata

JCT, Vol. 69, No. 872, 45 (Sept. 1997)

Estudio del Volumen Libre
"Porosidad" de Epóxicos secos y
saturados de agua Mediante el
Método PALS—M.M. Madani, R.R.

Miron and R.D. Granta

Free volume cavity sizes and fractions of epoxy specimens were determined using positron annihilation lifetime spectroscopy (PALS). PALS data were obtained before and after specimen water equilibration. Specimens were bisphenol A epoxide (B) and/or glycol epoxide (G) cured with a polyamide. Free volume sites increased linearly and cavity sizes decreased linearly with epoxide B:G ratio. Glass transition temperature (T_g) increased with epoxide B:G ratio. Water molecules in wet epoxy B filled approximately six percent of the cavities. epoxy G cavity size increased 11.4% after water equilibrium and was ascribed to cavity expansion. PALS results differed for commercial corrosion inhibitors in wet and dry mixtures of these epoxides.

Fueron determinados los volúmenes libres de las cavidades "porosidad" y la fracción de grupos epóxicos usando espectroscopia del tiempo de vida de aniquilación de l positrón (PALS). Los datos PALS fueron obtenidos antes y después de que la muestra se encontraba en equilibrio con el agua. Las muestras fueron exóxicos (B) tipo bistenol A y/o epoxi glicol (G) curados con una poliamida. Los espacios libre se incrementan linealmente y el tamaño de las cavidades disminuye linealmente con la relación epoxica (B):(G). La temperatura de transición vítrea (Tg) se incrementa con la relación epóxida. (B):(G). Las moléculas de agua llenan aproximandamente 31 6% de las cavidades del epóxico (B). El tamaño de las cavidades en el epóxico (G) se incrementan en 11.4% después de que se obtiene equilibrio con el agua y fue atribuida a la expansión de las cavidades. Los resultados obtenidos con el método PALS difieren para los inhibidores comerciales de corrosión en mexclas húmedas y secas de esos epoxidos.

Interfacial Studies of Crosslinked Polyurethanes; Part I. Quantitative and Structural Aspects of Crosslinking near Film-Air and Film-Substrate Interfaces in Solvent-Borne Polyurethanes—A.M. Kaminski and M.W. Urban

JCT, Vol. 69, No. 872, 55 (Sept. 1997)

One of the reactions leading to the formation of polyurethane (PU) crosslinked networks is the reaction of NCO and OH functionalities. In this study, we examined how crosslinking reactions of hexamethylene diisocyanate isocyanurate and polyacrylate near the filmair (F-A) and film-substrate (F-S) interfaces in urethane coatings may effect crosslink density as well as other network properties. While at the initial stages of the crosslinking reactions, solvent evaporation competes with the urethane network formation and isocyanate consumption changes at various depths from the F-A and F-S interfaces. Quantitative analysis of the NCO consumption as a function of depth showed that the NCO concentrations change from 2.35 ¥ 10-5 to 2.09 ¥ 10-5 M, while going from 0.27 to 1.14 µm. During reaction times not exceeding two to three hours, the NCO consumption at the F-A and F-S interfaces is consumed more rapidly. At low relative humidity conditions, excessive amounts of unreacted NCO exists at both the F-A and F-S interfaces. However, at the extended reaction times, NCO concentration levels at the F-S are greater than at the F-A interface, and the NCO concentration differences can be as high as 3 ¥ 10-5 M. In this study we also examined how crosslinking reactions of hexamethylene diisocyanate (HDI) isocyanurate and polyacrylate near the film-substrate (F-S) interfaces in urethane films may affect orientation and distribution of urethane functionalities. It appears that at initial stages of the crosslinking reactions, driving forces resulting from interfacial tension effects compete with the urethane network formation. Orientation of the H-bonded urethane carbonyl functionalities change as a function of distance from the F-S interfaces, ranging from 0.35 µm to 1.51 µm into a coating. Molecular migration and rearrangements near the F-S interfaces result from interfacial tensions between PU and substrates.

Estudio Interfacial de la Reticulación de los Poliuretanos; Part I. Aspectos Estructurales y Cuantitativos de la Reacción de Reticulación cerca de las Interfaces Película—Aire y Película—Sustrato en los Poliuretanos Base Solvente—A.M. Kaminski y M.W. Urban

Una de las reacciones más importantes para la formación de la malla de reticulación de un poliuretano (PU), es la reacción de los grupos funcionales NCO y OH. En este estudio, nosotros examinaremos como la reacción de reticulación del Hexametilen Diisocianato (HDI) y poliacrilatos cerca de las interfaces película - aire (F-A) y película - sustrato (F-S) en los recubrimientos de poliuretano puede afectar la densidad de la reticulación así como otras propiedades. Mientras que en las etapas iniciales de la reacción de reticulación la evaporación del solvente compite con la formación de la malla de uretano, el consumo de isocianato cambia a diferentes espesores desde las interfaces F-A y F-S. Análisis cuantitativos de el consumo de NCO como una función del espesor, mostraron que los cambios en la concentración van desde 2.35 x 10 E - 3 a 2.09 x 10 E -% M, a los espersores de 0.27 a 1.14 micras. Durante tiempos de reacción no mayores de 2.3 horas, el consumo de NCO en las interfaces F-A y F-S es más rápido. A condiciones de humedad relativa bajas, se obseraron cantidades excesivas de NCO sin reaccionar existente e ambas interfaces F-A y F-S. Sin



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Hercules Incorporated Aqualon Division Hercules Plaza 1313 North Market Street Wilmington, DE 19894-0001 (302) 594-5000 embargo, a medida que el tiempo de reacción transcurre, los niveles de concentración de NCO en las interfaces F-A es mayor que en las interfaces F-A, y las diferencias en la concentración de NCO fueron mayores de 3 x 10 E-5 M. En este estudio nosotros también examinamos como la reacción de reticulación del isocianurato de HDI y los poliacrilatos cerca de la interfaces película sustrato (F-S) en una película de uretano puede afectarse por la orientación y distribución de los grupos funcionales uretano. Pareciera que en las etapas iniciales de la reacción de reticulación, las fuerzas resultantes de dirigir los efectos de tensión interfacial compiten con la formación de la malla de uretano. La orientación de los enlaces de hidrógeno (H), de los uretanos con funcionalidad carbonílica cambia como una función de la distancia desde las interfaces F-S, variando desde 0.35 a 1.15 micras dentro del recubrimiento. La migración molecular y los rearreglos cerca de las interfaces F-S dan como resultando la tensión interfacial entre el PU y los sustratos.

Study of Anionic Polyurethane Ionomer Dispersant-Y.S. Huang et

JCT, Vol. 69, No. 872, 69 (Sept. 1997)

Estudio de Dispersantes Aniónicos de Poliuretano Ionomérico-Y.S. Huang et al.

Anionic polyurethane ionomers designed for use as dispersants in water-soluble acrylic baking enamels were synthesized, and their structures were confirmed by infrared analysis. In an aqueous media, the surface tension of the polyurethane ionomers with different ionic or mixed ionic groups was found to increase with increased concentration of ionic groups, because of the orientation of hydrophobic groups at the air-water interface. It was also found that viscosity in water is higher for a polyurethane ionomer with 1-2,5-diaminovaleric acid hydrochloride than for a polyurethane ionomer with dimethylol propionic acid.

Polyurethane ionomers with dimethylol propionic acid, p-amino benzoic acid, and mixed dimethylol propionic acid/p-amino benzoic acid were better dispersants for the wettability and dispersion of titanium dioxide in the water-soluble acrylic enamels. The degree of dispersion of titanium dioxide was found to improve with increased agitation time and anionic polyurethane ionomer concentration. In addition, polyurethanes with dimethylol propionic acid or p-amino benzoic acid or mixtures of these acids improved titanium dioxide dispersion

Fueron sintetizados poliuretano aniónicos ionoméricos para ser usados como dispersantes en esmaltes acrilicos de horneo solubles al agua, sus estructuras fueron confirmadas mediante análisis infrarrojo. Se encontró que en medios acuosos, la tensión superficial de el ionómero de poliuretano con diferentes iones o mezcla de grupos ionicos, se incrementa cuando aumenta la concentración de grupos ionicos debide a la orientación de los grupos hidrofóbicos en las interfaces aire-agua. También se encontró que la viscosidad en el agua es mayor para un poliuretano ionomérico con ácido 1-2-5 diamino valérico-hidrochlorhídrico que para un poliuretano ionomérico con ácido dimetilol propiónico. Los poliuretanos ionomériocos con ácido dimetilol propiónico, ácido pamino benzóico y una mezcla de estos dos ácidos fueron mejores para la humectanción y dispersión de el bióxido de titanio (TiO2) en los esmaltes acrílicos solubles en agua. Se encontró que el grado de dispersión del TiO2 se mejora con un aumento en el tiempo de agitación y aumentando la concentración del poliuretano aniónico ionomérico. En resumen, poliuretanos con ácido dimetilol propiónico o ácido p-amino benzóico o mezclas de estos ácidos mejoran la estabilidad de la dispersión en los esmaltes de horneo.

Influence of Crosslink Density, Glass **Transition Temperature and Addition** of Pigment and Wax on the Scratch Resistance of an Epoxy Coating-J. Lange, A. Luisier and Á. Hult

JCT, Vol. 69, No. 872, 77 (Sept. 1997)

Influencia de la Densidad de Reticulación, Temperatura de Transición Vítrea y de la Adición de Pigmentos y Ceras en la Resistencia al Rayado de un Recubrimiento Epóxico-J. Lang, A. Luisier y A. Hult

The scratch resistance of a series of cationically cured epoxy coatings with varying crosslink densities has been investigated using five different scratch tests. The tests simulate a variety of scratch modes, ranging from single scratches to repeated surface wear. The relative influences of crosslink density and glass transition temperature on scratch resistance, as well as the effects of adding pigment and wax to the coatings, were examined. The results show the scratch resistance to increase both with increasing crosslink density and increasing glass transition temperature in all tests. It is also shown that adding pigment or wax either increased or decreased the scratch resistance of the coating, depending on which type of scratching was performed.

La resistencia al rayado de una serie de recubrimientos, epóxicos curados catiónicamente con variación en la densidad del reticulado han sido investigados usando cinco diferentes pruebas de rayado. Estas pruebas simulan una variedad de formas de rayado, que varían desde el rayado sencillo hasta el rayado repetido de una superficie desgastada. Fueron estudiadas la influencia relativa de la densidad de reticulación y de la temperatura de transición vítrea sobre la resistencia al rayado, así como también el efecto de la adición de pigmentos y ceras en el recubrimiento. Los resultados mostraron que la resistencia al rayado se incrementa cuando se incrementa la densidad de reticulación y la temperatura de transición vítrea al mismo tiempo en todas la pruebas. También se demostró que la adición de pigmentos y ceras cualquiera de los dos, aumenta o disminuye la resistencia al rayado del recubrimiento dependiendo de cual tipo de rayado se va a mejorar.

Witco Corp. and Ciba Specialty Chemicals Inc. Sign Letter of Intent to Exchange Businesses

itco Corp., Greenwich, CT, and Ciba Specialty Chemicals Inc., Basel, Switzerland, have signed a letter of intent to exchange Witco's epoxy systems and adhesives business and Ciba's PVC heat stabilizers business in a one-for-one transaction. Both businesses had 1996 sales of approximately \$120 million (CHF 180 million). Further terms of the letter of intent were not disclosed. The transaction is expected to be completed in late 1997 and is subject to the approval of relevant regulatory authorities and the execution of a definitive agreement between the parties.

Under the agreement, Witco will acquire Ciba's worldwide PVC heat stabilizers business based in Lampertheim, Germany. This transaction includes the tin, mixed metal, non-metallic stabilizers, epoxy plasticizers and lubricants for PVC and other PVC stabilizers.

Ciba will acquire Witco's worldwide epoxy systems and adhesives business located in Bergkamen, Germany, and minor production facilities in Singapore and Italy. These epoxy systems and adhesives are used for industrial protective coatings to protect both steel and concrete structures against chemical and mechanical wear. It represents one business within Witco's Metal Organics/ Coatings and Adhesives Strategic Business Units.

Covered in the exchange are the assets within the facilities that are directly associated with these businesses in Lampertheim and Bergkamen, as well as minor production facilities in Singapore and Italy.

Upon completion of the deal, Witco will continue to manufacture metal organics and tin intermediates at its Bergkamen plant, and will invest nearly

J&W Scientific Acquires Two California Companies

J&W Scientific Inc., Folsom, CA, has acquired two Rancho Cordova, CA, companies: Humonics, Inc. and R&D Separations, Inc.

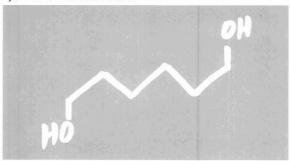
Humonics Inc. manufactures gas and liquid flow measurement devices for chromatographic science while R&D Separations, Inc. manufactures carrier gas purification traps/systems and other accessories for gas chromatography.

\$20 million to increase its capacity and enhance production at this site.

For Ciba Specialty Chemicals, Lampertheim will remain a key production, development, and marketing facility for the Additives Division, as well as the company's Central European headquarters. The company will continue with its plans to consolidate activities from Wehr and Frankfurt/Main to the Lampertheim facility, including the construction of a new office and laboratory.

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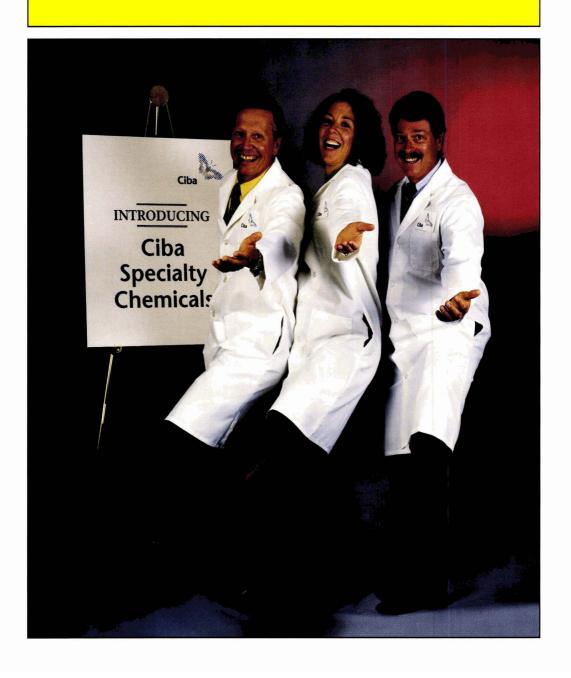
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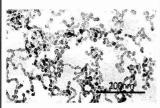
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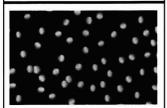


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Lyondell Petrochemical Co. Forms Joint Venture With Millennium Chemicals Inc.

A definitive agreement to form a new venture has been signed by Lyondell Petrochemical Co., Houston, TX and Millennium Chemicals Inc., Iselin, NJ. The new company, with expected revenues of approximately \$5 billion, will be comprised primarily of the olefins and polymers businesses of the two companies.

The new venture company will consist of 13 manufacturing facilities on the Gulf Coast and in the Midwest, producing ethylene, propylene, polyethylene, polypropylene, ethyl alcohol, and associated products. In addition, the company formed by this venture, which will be operated as a partnership, will be owned 57% by Lyondell and 43% by Millennium.

Lyondell's contribution to the new company includes the businesses associated with the following operations: (1) the olefins business comprised of two olefins plants at the Channelview Petrochemical Complex near Houston, TX; (2) the polymers business consisting of three manufacturing facilities located in Pasadena, Victoria, and Matagorda County, TX; and (3) the technology group and

certain business functions located in and near Houston.

Meanwhile, Millennium will contribute the following operations: (1) the olefins business comprised of plants at Clinton, IA, LaPorte, TX, and Morris, IL; (2) the polymers business comprised of five manufacturing facilities in Clinton, IA, Morris, IL, LaPorte, Port Arthur, and Chocolate Bayou, TX; (3) the performance polymers business with manufacturing facilities in Tuscola, IL, Fairport Harbor and Heath, OH, and Crockett, TX; (4) The synthetic ethanol and ethyl ether businesses with facilities at Tuscola, Newark, NJ, and Anaheim, CA; and (5) research and technology groups and a research center in Cincinnati, OH.

Not included in the venture are Millennium's interest in acetic acid, vinyl acetate, and methanol assets and operations. Also excluded from the transaction are Millennium Inorganic Chemicals, Millennium's sequity interest in Suburban Propane Partners.

The transaction is expected to close by year end, following approval by both companies' stockholders and satisfaction of certain other conditions.

Shell to Increase Capacity at Geismar, LA, Facility

A 40% manufacturing capacity increase has been announced by Shell Chemical Co., Houston, TX, for its 1,3 propanediol (PDO) unit at the Geismar, LA, facility. A raw material, PDO is necessary for the

Union Carbide Begins Work on Butanol Plant in Louisiana

Union Carbide Corp., Danbury, CT, has announced that work has begun on its second butanol production unit at Taft, LA. The 300-million pounds-per-year facility will start up in June of 1999 and bring the company's butanol capacity to 1.2 billion pounds.

The new plant will duplicate the Taft butanol unit that began production in December 1995 using Union Carbide/ Kvaerner low-pressure oxo technology. The oxo process is based on a new rhodium catalyst system that improves operating costs and product mix while lowering investment costs.

In the past two years, Union Carbide has added capacity in butanol derivatives worldwide, including butyl acrylate in North America, butyl glycol ethers in North America and Europe, and butyl acetate in North America and Asia. production of Corterra $^{\text{TM}}$ polymers, a polytrimethylene terephthalate, or PTT.

Shell plans to construct additional PDO and Corterra polymer plants in Louisiana for start-up in early 1999. The increased PDO manufacturing capacity at Shell's Geismar unit will require minor equipment changes which should be completed by the third quarter of 1997.

Corterra polymers are initially being marketed to the carpet, textile, and engineering thermoplastics industries.

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EFFECTIVE: September 1, 1997



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Waterborne Coatings Explored During Cleveland Society's Technical Symposium

he Cleveland Society for Coatings Technology conducted their 40th Annual Technical Symposium "Waterborne Coatings: Sink or Swim" on May 22-23, 1997. Held on the Case Western Reserve University campus in Cleveland, OH, the symposium attracted over 170 attendees.

The following topics were discussed during the two-day event:

"Two-Component Waterborne Polyurethane Coatings—Maturation of an Idea" Myron Shaffer and Douglas Wicks, Bayer Corp.;

"Shear Thickening and Time-Dependent Rheological Behavior in Aqueous Polyacrylic Ester Dispersions"—Alex Jam-ieson, Case Western Reserve University;

"Formulating with P-Series Glycol Ethers"—Carol Fox, ARCO Chemical;

"Polysiloxanes for High Temperature Waterborne Coatings"—James D. Green, Wacker Silicones;

"Environmental Issues Facing the Ohio Paint Industry"—Robert L. Toth, ICI Paints;

"Waterborne Epoxy Coatings—A Perspective on Dry"—Joseph Mulvey, Henkel Corp.; "Aluminum Pigments in Water—Fact or Fallacy?"—Martha Davies, Reynolds Aluminum;

"The Effect of Rheological Additives on the Properties of Water-Based Coatings"—David Bryant, Rheox, Inc.;

"On-Line Troubleshooting in Waterborne Systems"—Carol Williams and Charlie Tabbi, Reichhold Chemical Co.; and

"Update on VOC Regulations"—Madelyn Harding, The Sherwin-Williams Co.

David Bryant, of Rheox, was presented with the Symposium's Best Speaker Award.

In addition, the symposium featured table-top displays by nearly 40 companies. Personnel from the exhibiting companies were on-hand to answer any attendees' technical questions.

Plans are already underway for next year's symposium. The 41st Annual Technical Symposium will be a collaboration between the Cleveland and Pittsburgh Societies. Be sure to make plans to attend the 1998 Symposium on April 23-24. An added feature to the agenda next year is a Manufacturing Symposium slated for April 22.



Attendees at the CSCT's 40th Annual Technical Symposium discuss the latest technologies in waterborne coatings with table-top display personnel.

For more information on the Technical Symposium, contact Vicki Fisher, Jamestown Paint Co., 108 Main St., P.O. Box 157, Jamestown, PA 16134; (412)932-3101. For additional information on the Manufacturing Symposium, contact James Currie at the same address and

Biodeterioration Research Group to Meet on October 29-31, 1997

phone number.

Troy Corp., Florham Park, NJ, will host the autumn meeting of the International Biodeterioration Research Group on October 29-31 at the Meadowlands Hilton in Secaucus, NJ. The event marks the first time in IBRG history that the group has assembled outside Europe to hold its meeting.

IBRG is a consortium of scientists and other chemical industry professionals dedicated to improving biological testing procedures for industrial products. Members conduct round-robin tests in such areas as national and international standard testing programs, new testing procedures, contamination problems, and culture maintenance, then meet twice annually to discuss their findings. The consortium consists of a variety of working groups, concentrating on paint and emulsions, plastics protection, leather treatment, biocidal products directive, rapid methods, and metalworking fluids.

IBRG members and non-members are invited to attend. For more information, contact Marie Marabuto, Troy Corp., 8 Vreeland Rd., P.O. Box 955, Florham Park, NJ 07932-0955; (201) 443-4200.

Production Planning and Scheduling Workshop Slated for November 13-14 in Chicago, IL

CSCT Educational Chair

Vicki Fisher, of Jamestown

Paint Co. presents David

Bryant of Rheox, Inc. with

the Best Speaker Award

On November 13-14, 1997, the National Paint and Coatings Association (NPCA), Washington, D.C., will hold a "Production Planning and Scheduling Workshop" at the Rosemont Suites Hotel O'Hare, Chicago, IL.

The workshop is geared toward the needs of the process industries and will cover topics such as refocusing a company around the supply chain; theory and practical applications of planning and scheduling; finite capacity scheduling and material requirements planning logic; state-of-the-art planning and scheduling technologies; 15 points to consider when scheduling production;

and blending business strategies with the reality of limited capacity and resources.

The workshop is designed for individuals whose day-to-day work activities have impact on production schedules, inventory management, and customer service who wish to explore implementation of production planning and scheduling techniques to increase productivity and improve performance.

The registration fee is \$795 for NPCA members and \$995 for non-members. For more information, contact Melina Jimenez, NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005-5597.



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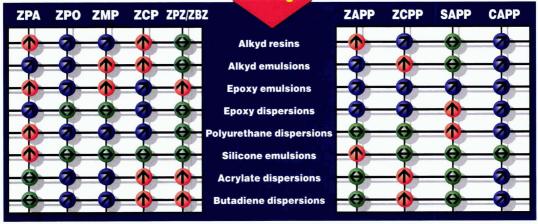
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Research Conference on Emulsion Polymers/Polymer Colloids To Be Held November 3-5, 1997 at Hilton Head Island, SC

The Institute of Materials Science, State University of New York, New Paltz, NY, will conduct the Second North American Research Conference on Emulsion Polymers/Polymer Colloids on November 3-5, 1997. The event will be held at Hilton Head Island, SC. The topics to be discussed include the following:

"Emulsion Polymerization-An Exciting Example of Applied Colloid Chemistry"-Klaus Tauer, Max Planck Kolloid Institut für Grenzflachenforschung;

"Some Scientific and Technological Challenges in Emulsion Polymerization"-Anton L. German, Technische Universiteit Eindhoven;

"Modeling and Compositional Control in Emulsion Co- and Terpolymerization"-Alex van Herk, Technische Universiteit Eindhoven;

"Synthesis of Micron-Sized, Monodispersed Functional Polymer Particles by Seeded Polymerization Utilizing the Dynamic Swelling Method"— Masayoshi Okubo, Kobe University;

"Evaluation of Dimethyl Meta-Isopropenyl Benzyl Isocyanate (TMI) in Emulsion Polymerization"—Eric Daniels, Lehigh University;

"Utilization of CO, in Heterogeneous Polymerizations: From Dispersion Polymerizations to CO, Modified, Aqueous-Based Emulsion Polymerizations"—J.M. DeSimone, University of North Carolina, Chapel Hill;

"Experimental Study of Emulsion Polymerization Kinetics with Crosslinking"—Hua Guo, General Electric Co.;

"The Use of Infrared and Calorimetry in Emulsion Polymerization"-Robert J. Warner, SC Johnson Polymer;

"Controlled Aggregation of Anionic Latexes with Cationic Surfactant"-Grazyna E. Kmiecik-Lawyrnowicz, Xerox Corp.;

"Interparticle Potentials of Latex and Titanium Dioxide Particles"-Stuart G. Croll, Millenium-Inorganic Chemicals;

"Modeling of Interactions Between Latex Particles and Macro-Surfaces"-Edward Kostansek, Rohm and Haas Co.;

"Interactions and Dispersion Structure in Nonspherical Latex Dispersions"-Michael S. Wolfe, E.I. DuPont de Nemours & Co., Inc.;

"Polymer Colloid Partitioning in Mixtures of Water-Soluble Polymers"-Peter R. Sperry, Rohm and Haas Co.;

"Viscoelastic Measurements of Carboxylated Latexes"-Yohannes Chonde, Dow Chemical Co.;

"Polymer Colloids for Diagnostic Applications"—C. Pichot, Ecole Normale Superieure de Lyon;

"Nanostructured Latexes: Synthesis, Characterization, and Properties"-Do Ik Lee, Dow Chemical Co.;

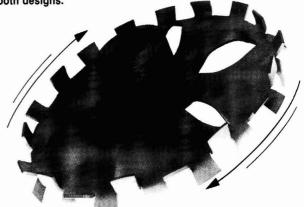
'Role of Latex Particle Size on Film Formation"-Charles S. Kan, Dow Chemical Co.:

"The Evolution of Interfaces in Single-Component and Blended Latex Films" Jianrong Feng, Thomson-Gordon Group;

"Ripening and Interdiffusion in Structured Latex Film"-M. Joanicot, Rhône-Poulenc S.A.

For more information, contact Angelos V. Patsis, Institute of Materials Science, State University of New York at New Paltz, New Paltz, NY 12561.

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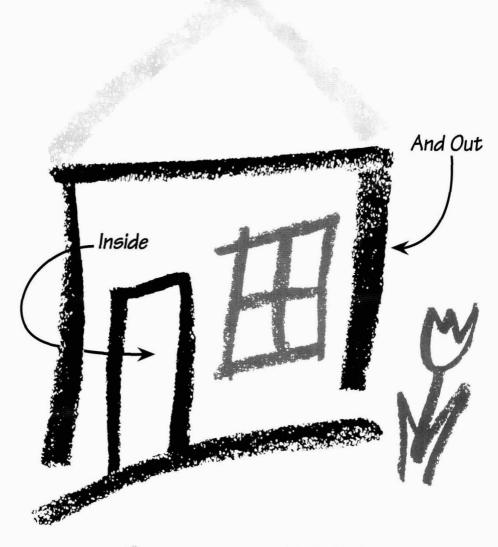
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Preliminary Program Information



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November 3-5, 1997

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Conference

FSCT's "ICE '97" Marks Our 75th Anniversary

ncompassing the spectrum of coatings manufacturing, the new format of the FSCT's annual convention will again feature an exhibition of over 300 supplier companies at the International Coatings Expo, on November 3-5, 1997 at the Georgia World Congress Center, in Atlanta, GA. Held in conjunction with the FSCT Annual Meeting and the Technology Conference, the Expo will present the industry's largest exhibition of suppliers. Come meet these suppliers for a face-to-face discussion of your needs and concerns. Problem-solving opportunities abound at "ICE '97"!

The FSCT's concurrent Technology Conference will provide a forum for learning at all levels of the coatings industry, from the newly hired technician to top level management. Each Course has been designed for specific areas of your organization, including lab personnel, sales and marketing staff, manufacturing, research and development, and quality assurance.

Highlights of the Technology Conference include:

- Two Executive Forums, covering new product development and technology management on Monday, November 3.
- Nine one-day Courses, scheduled over three days: November 3, 4, and 5, allowing attendees ample time to visit the International Coatings Exposition.
- Four two-day Courses, scheduled for Monday and Tuesday, November 3 and 4, which again allows attendees time to visit the Exposition on November 5.
- Course attendees will be given *complimentary attendance* to the International Coatings Expo and the technical presentations given at the FSCT's Annual Meeting.
 - Set of Course Materials is provided to the attendees of each Course.

Both you and your company benefit from attendance at this event. Your personal knowledge increases, which in turn improves your value within your organization, while the company stands to benefit from the new ideas and solutions you've learned by attending the conferences. The conferences also provide an opportunity for all coatings personnel to participate in the industry's premier event and learn the latest advances in the coatings industry.

ICE '97 Opening Session: Keynote Address by Daniel Burrus

Daniel Burrus, respected Science and Technology Forecaster, will provide the Keynote Address at the Opening Session on Monday, November 3.

Recent innovations in science and technology have provided us with a "new" set of tools to work with which will greatly increase our productivity and efficiency in all

areas. Knowing what these tools are, and how to apply them creatively is rapidly becoming a matter of business survival and a key to personal gain. In his presentation, Mr. Burrus uses down-to-earth terminology, insight and humor to provide an informative, provocative and fascinating look at the impact of scientific innovations on how we will live and work in the near future.



Technical Focus Lecture

One of the most popular features of the Annual Meeting, the Technical Focus Lecture kicks off the technical portion of the Annual Meeting technical program. The Lecturer is chosen by the Chairs of several FSCT Committees for on-going work in critical technical areas. This year's lecturer is Douglas A. Wicks, of Bayer Corp., and his topic is "Progress Towards Solvent Free Polyisocyanate Based Coatings."

Mattiello Memorial Lecture

The 1997 Mattiello Lecturer is F. Louis Floyd, Vice President for Technology of Duron Paints and Wallcoverings, Baltimore, MD. He will discuss "Reducing Product Development Cycle Times *Without* Increasing Risk." The Mattiello Lecturer is an individual who has made outstanding contributions to science, technology, and engineering, related to the coatings industry.

Schedule of Events

All events will be held at The Georgia World Congress Center (unless otherwise noted)

SUNDAY, NOVEMBER 2

8:00 am-5:00 pm

REGISTRATION SERVICES

6:00 pm-8:00 pm **EXECUTIVE FORUM RECEPTION AND DINNER**

MONDAY, NOVEMBER 3

7:00 am-5:00 pm **REGISTRATION SERVICES**

TECHNOLOGY CONFERENCE

(One-Day)

8:30 am-4:30 pm Executive Forum I: Strategically Effective New Product Develop-

ment: An Enterprise-Wide Approach from Concept Through

Commercialization

Executive Forum II: Advanced 8:30 am-4:30 pm

Topics in Technology Manage-

8:00 am-4:00 pm Effective Employee Motivation Strategies: Take This Job and Love

It—Creating an Inspired and

Motivated Workforce

Effective Technical and Scientific 8:30 am-4:30 pm

Writing Workshop

TWO-DAY CONFERENCES

(Part 1)

9:00 am-4:30 pm Introduction to Radiation Curing 9:00 am-4:00 pm Methodology of Evaluating

Corrosion Resistance

9:00 am-4:30 pm Back to Basics: Resins, Pigments,

Solvents & Additives

Polymer Chemistry for the 8:00 am- 4:00 pm

Coatings Formulator

8:30 am-10:00 am **ICE OPENING SESSION**

10:00 am-5:00 pm **EXPO HOURS**

1:00 pm-2:00 pm **TECHNICAL FOCUS LECTURE**

ANNUAL MEETING PROGRAM 1:00 pm-4:00 pm

2:00 pm-4:00 pm **SOCIAL GUEST WELCOME**

RECEPTION

TUESDAY, NOVEMBER 4

REGISTRATION SERVICES 7:00 am-5:00 pm

TECHNOLOGY CONFERENCE

8:30 am-4:30 pm Winning Technical Presentations

8:30 am-4:30 pm Effective Negotiating Skills 8:30 am-4:30 pm Creative Decorating

TWO-DAY CONFERENCES

(Part 2) Introduction to Radiation Curing 9:00 am-4:00 pm

9:00 am-3:30 pm Methodology of Evaluating

Corrosion Resistance

Back to Basics: Resins, Pigments, 9:00 am-4:30 pm

Solvents & Additives

Polymer Chemistry for the 8:00 am- 4:00 pm

Coatings Formulator

EXPO HOURS 9:00 am-5:00 pm

SOCIAL GUEST OPTIONAL 9:30 am-3:30 pm

TOURS—Depart from GWCC

ANNUAL MEETING PROGRAM 9:00 am-3:30 pm

6:30 pm-7:30 pm INTERNATIONAL RECEPTION

Marriott Marquis

WEDNESDAY, NOVEMBER 5

7:30 am-12:00 Noon REGISTRATION SERVICES

8:00 am-9:30 am **FSCT ANNIVERSARY** RREAKEAST

> TECHNOLOGY CONFERENCE (One-Day)

8:30 am-4:30 pm Surfactant Chemistry and

Application

8:30 am-4:30 pm Industry Leadership with Product Stewardship

Spray Applications Workshop 8:00 am-5:00 pm Binks-Sames, Norcross, GA

Chemistry and Formulation of 8:00 am-4:00 pm

Powder Coatings

9:00 am-12:00 Noon **EXPO HOURS**

10:30 am-11:30 am

ANNUAL MEETING PROGRAM 9:00 am-11:30 am

MATTIELLO MEMORIAL

LECTURE



Monday, November 3 10:00 am - 5:00 pm

Tuesday, November 4 9:00 am - 5:00 pm

Wednesday, November 5 9:00 am - 12:00 Noon



FSCT 75th Anniversary Annual Meeting Program



NOVEMBER 3-5, 1997 ATLANTA, GA

Celebrating 75 years as the premier technical organization in the coatings industry, the FSCT has planned an event that will truly reflect the theme "75 Years . . . Tradition . . . Discovery . . . Opportunity." ICE '97 will provide a fascinating look back to the organization's origins. Even further, it will offer attendees insights into the coatings industry as it moves into the future—previewing

innovative technologies and solutions to current challenges. Participants of ICE '97 will have the opportunity to attend Annual Meeting technical sessions as part of their registration. Highlights of this year's technical program

include:

MONDAY, NOVEMBER 3, 1997

Technical Focus Lecture 1:00 pm - 2:00 pm

"Progress Towards Solvent Free Polyisocyanate Based Coatings"—Douglas A. Wicks, Bayer Corp.

Roon Award Competition Papers 2:30 pm - 4:00 pm

"Oxidative Crosslinking of Alkyd Resins Studied with Mass Spectrometry and NMR Using Model Compounds"— W.J. Muizebelt, J.J. Donkerbroek, M.W.F. Nielen, J.B. Hussem, M.E.F. Biemond, Akzo Nobel Central Research, Arnhem, The Netherlands and R.P. Klaasen and K.H. Zabel, Akzo Nobel Coatings Research, Sassenheim, The Nether-

"Recent Advances in Epoxy Curing Agent Technology for Low Temperature Cure Coatings"-Kalyan Ghosh and Patricia Garcia, Shell Development Co., Westhollow Technology Center, Houston, TX

"Analysis of Water in Paints by Near Infrared Spectroscopy"-John L. Massingill, S. Wang, and Z. Wu, NSF I/U Cooperative Research Center in Coatings, Eastern Michigan University, Ypsilanti, MI

TUESDAY, NOVEMBER 4, 1997

APJ/Voss Award Competition Papers 9:00 am - 11:30 am

"Temperature Dependence on the Rheological Behavior of Modified Acrylic Latex Coatings"—Cleveland Society for Coatings Technology

'Correlation of Accelerated Exposure Testing and Exterior Exposure Sites, Part IV"-Cleveland Society for Coatings Technology

"Low Cost, In-House VOC Determination of Latex Paints"—Los Angeles Society for Coatings Technology

"Pearl and Aluminum Pigments in Waterborne Coatings: What Variables Determine Optimum Performance?"— Louisville Society for Coatings Technology

"Reactive Diluents for Two-Component Polyurethane Coatings"—New York Society for Coatings Technology

Women in Coatings 2:00 pm - 3:30 pm

Roundtable discussion on the role and impact of women in the coatings industry. Participants include: Darlene Brezinski, Consolidated Research, Inc.; Susannah I. Dobbs, Crown Technology II, LLC; and Sandra B. Skommesa, Ellis Paint Co.

WEDNESDAY, NOVEMBER 5, 1997

International Papers 9:00 am - 10:30 am

"The Performance of Building Materials in Australia"-Donald J. Bartlett, Australian Paint Approval Scheme

Title to be determined—John Gillatt, Oil and Colour Chemists' Association

"One-Pack Ambient Curing, Waterborne Coating Composition Using Michael Addition Which Has Good Stability and Gives a Highly Crosslinked Film"-Katsuhiko Sho, Koichi Saito, Tohru Yagi, Japan Society of Colour Material

Mattiello Memorial Lecture 10:30 am - 11:30 am

"Reducing Product Development Cycle Times Without Increasing Risk"-F. Louis Floyd, Duron Paints and Wallcoverings

Coatings Societies International to Present Medallion Honoring Technical Achievement at FSCT 75th Annual Meeting

Coatings Societies International will sponsor an award honoring outstanding technical achievement in the presentation of papers at the FSCT Annual Meeting in Atlanta, on November 3-5, 1997. This award, first presented at the XXIII FATIPEC Congress in June 1996, is given at selected annual congresses of CSI-member organizations.

In addition to the Federation of Societies for Coatings Technology, member organizations of CSI include: FATIPEC (Federation d'Association des Technicians des Peintures, Vernis, Emaux, et d'Imprimerie de l'Europe Continentale); JSCM (Japan Society of Colour Material); OCCA (Oil & Colour Chemists' Association); SLF (Skandinaviska Lackteknikers Forbund); SCAA (Surface Coatings Association Australia); and SCANZ (Surface Coatings Association New Zealand).

Coatings Technology Conference

All Courses held at Georgia World Congress Center (unless otherwise noted)

75th Annual Meeting **International Coatings Expo and Technology** Conference

SUNDAY, NOVEMBER 2

Sunday Evening Executive Forum (Reception and Dinner)

(Included with registration for Executive Forum I or II)

Time: 6:00 pm - 8:00 pm

MONDAY, NOVEMBER 3, 1997

One-Day Courses

EXECUTIVE FORUM I: STRATEGICALLY EFFECTIVE NEW PRODUCT DEVELOPMENT: AN ENTERPRISE-WIDE APPROACH FROM CONCEPT THROUGH COMMERCIALIZATION

Time: 8:30 am - 4:30 pm

Course Description

A one-day interactive workshop introducing participants to the "Why's" and "How's" of enterprise-wide New Product Development (NPD), fuzzy front-end processes, concept generation, portfolio management, supporting systems and tools and fast development. The course is geared toward company executives and decision makers with key responsibilities for improving their organization's NPD output.

Instructors

Paul J. O'Connor, Adept Group, Ltd. Christopher W. Miller, Innovation Focus

Registration is limited to 30 attendees.

EXECUTIVE FORUM II: ADVANCED TOPICS IN TECHNOLOGY MANAGEMENT

Time: 8:30 am - 4:30 pm

Course Description

Targeted to R&D and business executives who have strategic leadership responsibility in their organizations, this executive level workshop will introduce participants to three management topics focused on maximizing technology to achieve commercial success in the coatings industry.

Instructors

Stephen E. Rudolph, A.D. Little John F. Martin, A.D. Little Masha V. Markusova, A.D. Little

Registration is limited to 30 attendees.

EFFECTIVE EMPLOYEE **MOTIVATION STRATEGIES:** TAKE THIS JOB AND LOVE IT—CREATING AN INSPIRED AND MOTIVATED WORKFORCE

Time: 8:00 am - 4:00 pm

Course Description

Designed for all managers, this course will provide attendees with the necessary techniques to keep employees all pulling in the same direction for a common cause. Participants will learn the benefits of different types of motivational strategies and why some techniques work on certain people and not others. Attendees will also learn the value of incorporating these strategies into their management style.

Instructor

Jane Shuman, Shuman and Associates

Registration is limited to 30 attendees.

EFFECTIVE TECHNICAL AND SCIENTIFIC WRITING WORKSHOP

Time: 8:30 am - 4:30 pm

Course Description

For all levels of laboratory and R&D personnel, along with applicators and anyone with responsibility for writing memos, letters, reports, manuals, specifications and proposals on a routine basis. The session includes inclass writing exercises designed for practical application and allows time for individual instructor attention. Participants are invited to submit writing samples in advance for confidential review by the instructor.

Instructor

Salvatore J. Iacone

Registration is limited to 25 attendees.

MONDAY-TUESDAY, **NOVEMBER 3-4, 1997**

Two-Day Courses

INTRODUCTION TO RADIATION CURING

Co-Sponsored by RadTech International

Time: 9:00 am - 4:30 pm (Monday) 9:00 am - 4:00 pm (Tuesday)

Course Description

Radiation curable coatings are fast becoming an efficient method to meet the regulatory challenges facing the industry. This course is designed for formulating chem-



NOVEMBER 3-5, 1997 ATLANTA, GA

Coatings Technology Conference

ists, R&D Chemists, technical directors and company decision makers in the adhesives, inks, plastics and coatings industries, and will provide details on the basics of radiation cure as a low VOC, low energy cure technology.

"Digital Imaging for the Rating of Weathered Products"— Fred Lee, South Florida Test Service

"Project Management and Statistical Approach to Corrosion Study"—Eric J. Carlson, A.D. Little

Registration is limited to 125 attendees.

Topics and Instructors

"Introduction to Radiation Curing (Electron Beam, Free Radical, Cationic)"—Latoska N. Price, Akzo Nobel

"Photoinitiators and Reactive Diluents"—William R. Schaeffer, Sartomer

"Equipment for Radiation Curing"—Richard W. Stowe, Fusion UV Systems

"Toxicology, Safety and Handling"—Ronald Golden, UCB Radcure

"Formulating for Radiation Curing: Coatings for Wood"— Roy J. Modjewski, Akzo Nobel

"Formulating for Radiation Curing: Coatings for Metal"— John Braddock, Union Carbide

"Formulating for Radiation Curing: Radiation Curing for Printing Inks"—Paul Gupta, Flint Ink

Registration is limited to 125 attendees.

METHODOLOGY OF EVALUATING CORROSION RESISTANCE

Developed by FSCT Corrosion Committee

Time: 9:00 am - 4:00 pm (Monday) 9:00 am - 3:30 pm (Tuesday)

Course Description

This course is designed to review what corrosion is, its mechanism, methods of prevention, and a discussion of standardizing the process of testing for corrosion resistance. Attendees will learn important details of corrosion, the principles of protection, and then participate in and discuss possible methods of standardizing the evaluation process. It is designed for formulators, R & D chemists, technical service personnel, and technical management.

Topics and Instructors

- "The Definition and Mechanism of Corrosion and Environmental Variation"—John DeLuccia, University of Pennsylvania
- "Surface Preparation"—Lloyd Smith, Corrosion Control Consultants
- "Contribution and Value of Surface Treatments"—Peter W. Dority, Henkel
- "Theoretical Methods Coatings Use to Protect a Substrate"— James A. Ellor, Corrpro
- "Practical Formulation Techniques"—Vince McGinnis, Battelle
- "Cabinets and Exterior Exposure for the Purpose of Evaluating Corrosion Resistance"—Douglas Grossman, Q-Panel
- "Electrochemical Means to Evaluate Corrosion"—Stephen Tait, Johnson Polymers

BACK TO BASICS: RESINS, PIGMENTS, SOLVENTS & ADDITIVES

Time: 9:00 am - 4:30 pm (Monday) 9:00 am - 4:30 pm (Tuesday)

Course Description

For chemists, lab technicians, sales, marketing, and field support personnel new to the industry or with minimal experience. Participants will gain a better understanding of the four main components that make up the basic composition of coatings (resins, pigments, solvents, and additives), the role each plays in the development of coatings, the effect of various application methods on coatings performance and will be given tips on trouble-shooting techniques to effectively formulate coatings.

Topics and Instructors

- "Resins and Solvents"—George M. Schmitz, Reichhold Chemical
- "Pigments and Additives"—Curtis P. Bailey, Bailey and Associates

Registration is limited to 125 attendees.

POLYMER CHEMISTRY FOR THE COATINGS FORMULATOR

Developed by FSCT Professional Development Committee

Time: 8:00 am - 4:00 pm (Monday) 8:00 am - 4:00 pm (Tuesday)

Course Description

Provides current information on polymer chemistry for coatings formulators, R&D chemists, and sales and marketing personnel with strong technical backgrounds or interests. Attendees will realize a greater understanding of the essential concepts of polymer science and the underlying principles to determine coating performance. The course helps attendees to develop coatings using specific scientific principles as opposed to trial and error and is also relevant for ink, sealant, and adhesive industry personnel.

Topics and Instructors

- "Polymer Fundamentals"—John L. Gardon, Akzo Nobel
- "Step-Growth Polymerization"—Frederick H. Walker, Air Products
- "Chain Growth Polymerization"—David J. Nordstrom, DuPont Automotive
- "Alkyds and Polyesters"—Paul R. Baukema, Akzo Nobel

Coatings Technology Conference

75th Annual Meeting

International Coatings

Expo and Technology

Conference

"Polyurethanes"—Peter Schmitt, Bayer

"Melamine Crosslinking Agents"—Colin Brogan, Cytec

"Emulsion Polymerization"—Mark A. Kesselmayer, Rohm

"Vinyl Latexes"—Jennifer Cogar, McWhorter Technologies

"Acrylics"-Mark R. Winkle, Rohm and Haas

"Epoxy Resins"-David A. Dubowik, Air Products and Chemicals

"Rheology"-Richard R. Eley, ICI Paints

"Mechanical Properties"—Michael Neag, ICI Paints

"Polymer Degradation"—John Gerlock, Ford Motor Co.

Registration is limited to 125 attendees.

TUESDAY, NOVEMBER 4, 1997

One-Day Courses

WINNING TECHNICAL PRESENTATIONS

Time: 8:30 am - 4:30 pm

Course Description

For laboratory and R&D personnel at all levels, in addition to marketing and sales staff and anyone else responsible for delivering technical presentations. Attendees will learn how to design an effective technical presentation, such as learning how to develop effective visuals; proper speaking techniques and data organization; how to handle question and answer sessions; tips on transferring written information to speaking terms; and how to communicate clearly to all audiences. This program offers a combination of lecture, interaction and small group projects.

Instructor

Carter Johnson, Buying Time Seminars

Registration is limited to 25 attendees.

EFFECTIVE NEGOTIATING SKILLS

Time: 8:30 am - 4:30 pm

Course Description

Every day, in every aspect of our lives, we are faced with negotiations. This program, designed for both technical staff and sales personnel, will provide attendees with proven techniques to enable your organization to benefit, both financially and time wise, from improved negotiations.

Instructor

Mark K. Schoenfield, of Schoenberg, Fisher, Newman & Rosenberg, Ltd.

Registration is limited to 30 attendees.

CREATIVE DECORATING

Time: 8:30 am - 4:30 pm

Course Description

Designed for decorators, applicators, those who develop trade sales paints, forward marketers, painting contractors, store owners, and those interested in do-it-yourself projects, this course will provide current information on the latest techniques being used to decorate homes and the workplace. The course will cover techniques such as rag painting, sponge painting and many other methods used to provide the current "looks" for home and office decoration.

Instructors

John Catalanotto, Pro Faux Greg Frohnapel, Pro Faux

Registration is limited to 50 attendees.

WEDNESDAY, NOVEMBER 5, 1997

One-Day Courses

SURFACTANT CHEMISTRY AND APPLICATION

Time: 8:30 am - 4:30 pm

Course Description

For R&D personnel, technicians, formulating chemists and application chemists, along with technical service personnel in the coatings and inks industries, this course will provide attendees with a better understanding of the various aspects of surfactant chemistry and its effect on the development of coatings and inks. Attendees will also develop an open thinking approach to surfactants and problem solving. This course content will include case studies and live demonstrations. Attendees are encouraged to submit questions in advance.

Topics and Instructors

- "Overview of Surfactant Types"—Robert E. Stevens, Air Prod-
- "General Surfactant Properties"—Steven Snow, Dow Corn-
- "Wetting and Dispersion Phenomena"-Edward W. Orr, Byk-Chemie
- "Application Case Study: Surface Tension Coatings Defects and Substrate Wetting"—Joel Schwartz, Air Products
- "Application Case Study: Wetting and Dispersing"-Frederick M. Lewchik, Byk-Chemie
- "Application Case Study: Defoamers"—Paul Fox, Ultra Ad-

Registration is limited to 75 attendees.



Coatings Technology Conference

INDUSTRY LEADER-SHIP WITH PRODUCT STEWARDSHIP

Time: 8:30 am - 4:30 pm

Course Description

This course has been developed for senior officers, hygienists, regulatory personnel, and those individuals involved in regulatory enforcement from the DOT, military, refinishing industry as well as the coatings and chemical industries. The program will give attendees a good description of the benefits of developing a Responsible Care $^{\rm IM}$ or Coatings Care $^{\rm IM}$ program and the benefits to the company. This overview will give a basic understanding to attendees on how to install and manage such a program.

Topics and Instructors

- "Introduction: Why Responsible Care/Product Stewardship Will Change Your Business"—Steven R. Sides, NPCA
- "What is Product Stewardship and Responsible Care?"— Harry Finkbaum
- "Training Aids Available to Help Your Customers"—Richard Sayad, Dow Chemical
- "Communicating with Your Customers"—Charles M. Bartish, Air Products
- "Personal Protective Equipment"—R. Myers, Bayer Corp.
- "VOC's: How Quickly and How Far Must We Move?"— James Bassett, Eastman Chemical
- "Disposal Issues"-Instructor to be determined

Registration is limited to 100 attendees.

Responsible $Care^{\mathbb{M}}$ is a registered trademark of the Chemical Manufacturers Association and Coatings $Care^{\mathbb{M}}$ is a registered trademark of the National Paint and Coatings Association.

SPRAY APPLICATIONS WORKSHOP

Time: 8:00 am - 5:00 pm

Location: Binks-Sames, Norcross, GA

Course Description

Provides both experienced and novice applicators, field service personnel, specifiers and formulators with infor-

mation on current and upcoming technologies as they apply to the application of coatings and finishes. Considered a "Learning Exchange Seminar," attendees will learn how to properly select, maintain and operate spray finishing equipment and to answer a variety of questions related to spray finishing. This course will be held at the Binks-Sames facility in Norcross, GA. Transportation from the Georgia World Congress Center will be provided.

Instructor

Jerry Hund, Binks-Sames

Registration is limited to 25 attendees.

CHEMISTRY AND FORMULATION OF POWDER COATINGS

Time: 8:00 am - 4:00 pm

Course Description

Designed to provide formulators, R&D chemists, technical directors, and company decision makers with information on the basic processes of formulating powder coatings. This course will be beneficial to both newcomers to the powder coatings industry as well as give information to those companies that are considering adding powder coatings as a product line.

Topics and Instructors

- "What is Powder Coating/Powder Coating Chemistry"— Frances Salvagio, Ciba Specialty Chemicals
- "Powder Coating Manufacture/Recent Advances"—Debra Gill, Herberts O'Brien
- "Powder Coating Application/Formulation"—Alan Pekarik, S.C. Johnson

Registration is limited to 100 attendees.

Coatings and Inks Division

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Guest Accommodations

There are 10 hotels providing convention rates for ICE '97. All have undergone extensive renovations to prepare for the Olympics held in Atlanta in 1996. Properties have been included to match every travel budget.

The Atlanta Convention Bureau's housing department is the official housing coordinator for ICE '97. To obtain a reservation for the convention, you must complete the official housing form and send it to ICE Housing. Only international attendees using a credit card may fax to 404-521-6392. The Bureau will provide an acknowledgment of all reservations. The hotel assigned will then forward a confirmation of the reservation. Please read your confirmation carefully for the hotel's cancellation policy.

The deadline for housing is **October 3**. After October 3, the Housing Bureau will place reservations on a space available basis. A deposit is required on all reservations. The deposit amount is \$125 per room reservation with three exceptions: the Ritz Carlton, the Westin, and Omni at CNN, each of which requires a deposit equal to onenight's stay. If paying by check, make the check payable to the ICE Housing Bureau. Credit card deposits will be processed 30 days prior to arrival. Before October 3, contact the Housing Bureau at 404-521-6630 with changes required. After October 3, contact the hotels directly.

The ICE Housing Bureau strives to place your reservation in the hotel of your choice. If the property desired is not available, the reservation will be placed based on the next choice available. Please be sure to indicate three choices on your form.

Rates

		Double or			X indicates
Hotel	Single	Twin	1 BR Suites	2 BR Suites	Shuttle stop
FSCT Headquarters Atlanta Marriott Marquis* Marquis level	\$150 194	\$170 214	\$500 - \$1,250	\$650 - \$1,400	x
Atlanta Hilton and Towers	150	170			- x
Hyatt Regency Atlanta Concierge level	155 189	175 209	605 - 925	825 - 1,075	Х
Westin Peachtree Plaza Concierge Level	147 177	167 197			X
Radisson	91	101			X
Omni at CNN*	159	179	675 - 825	825 - 1,800	
Courtyard by Marriott	129	139			X
Best Western American Concierge level	99 119	114 134			X
Ritz Carlton	165	180			X
Comfort Inn	105	115			x

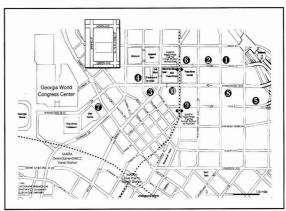
Key: Single = room with one occupant; Double = room with two occupants, 1 bed; Twin = room with two occupants, 2 beds; 1 bedroom suites = living room parlor and one connecting bedroom; 2 bedroom suites = living room parlor and two connecting bedrooms

*Exhibitors will be limited to a block of 10 rooms at the Marriott Marquis and the Omni at CNN. A one-bedroom suite counts as 2 rooms. A two-bedroom suite counts as 3 rooms.

= SOLD OUT

KEY to HOTEL MAP

- 1. Atlanta Hilton and Towers
- 2. Atlanta Marriott Marquis
- 3. Best Western American Hotel
- 4. Comfort Inn Downtown
- 5. Courtvard by Marriott Downtown
- 6. Hyatt Regency Atlanta
- 7. Omni at CNN
- 8. Radisson Hotel Atlanta
- 9. Ritz Carlton Atlanta
- 10. Westin Peachtree Plaza Hotel



1997 Housing Application Form

Reservations must be placed with the ICE Housing Bureau to receive preferred rates. Hotels will not accept reservations directly. Reservations must be received by October 3, 1997.

1. To Make Reservations

- ◆ Please print or type all items to ensure accuracy
- ◆ Complete each part below in detail for correct and rapid computer processing
- Form may be duplicated or supplemental room list must be attached using same format as below



MAIL or FAX Send completed form and payment (in U.S. dollars only) to:

ICE Housing Bureau 233 Peachtree St., N.E. Suite 2000 Atlanta, GA 30303

International Attendees may fax to 404-521-6392

Prior to October 3, all reservations, changes, and cancellations must be made with ICE Housing Bureau. After this date, these changes must be made with the hotel directly. Cancellation policies vary by hotel, please read confirmations carefully.

2. Hotel Information

it	Rate	3rd		Rate
nd	Rate		No. of roo	oms requested
Names of Occupants	Room	Type*	Arrival	Departure
1.	☐ single☐ double	□ dbl/dbl		
2.	☐ single☐ double	☐ dbl/dbl		
3.	□ single □ double	☐ dbl/dbl		
4.	☐ single☐ double	☐ dbl/dbl		
5.	□ single □ double	☐ dbl/dbl		
the same of the sa		posits may be	made by major credit card	exception of the Omni @C or check (U.S. dollars).
☐ Enclosed is my check payable Please bill my: ☐ AMEX ☐ M				or check (U.S. dollars).
	C U Visa	for \$		or check (U.S. dollars).
Please bill my: AMEX M	C 🗅 Visa	for \$	_	or check (U.S. dollars).
Please bill my: AMEX M Credit Card Number Name of Card Holder	C U Visa	for \$	Expiration Date	or check (U.S. dollars).
Please bill my: AMEX M Credit Card Number Name of Card Holder	C UVisa	for \$	Expiration Date	or check (U.S. dollars).
Please bill my: AMEX MCredit Card Number Name of Card Holder Send Confirmations to Contact Name: Company:	C Uvisa	for \$	Expiration Date Signature Telephone:	or check (U.S. dollars).
Please bill my: AMEX MCredit Card Number Name of Card Holder Send Confirmations to Contact Name: Company: Address:	C UVisa	for \$	Expiration Date Signature Telephone:	or check (U.S. dollars).
Please bill my: AMEX MEX MEX MEX MEX MEX MEX MEX MEX MEX	C Uvisa	for \$	Expiration Date Signature Telephone: FAX:	or check (U.S. dollars).
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Registration Information

November 3-5, 1997 ATLANTA, GA

Registration Procedures

By Mail Complete ICE '97 Registration Form and mail with pay-

ment to:

ICE Registration P.O. Box 998 Park Ridge, IL 60068

By Fax 24 hours a day, 7 days a week

U.S. 800-952-9812

International 847-698-9245

By the Internet The FSCT's ICE '97 Registration has gone on-line. To register via the Internet, use the

address: "www.coatingstech.org"

If you have any questions on how to complete the registration form, contact the ICE Registration Help Line:

All cancellations and refund requests must be in writing and sent to ICE Registration. Cancellations received after October 26 are non-refundable.

All pre-registered attendees will receive a confirmation statement of their registration selection. Badges and materials will be available for pickup at the ICE Registration Services Area located in the Georgia World Congress Center beginning on Saturday, November 1.

Registration Rates

		ADVANCE PRICING (After June 30, 1997)		TION FEES
	Member	Non-Member	Before Oct. 3	After Oct. 3
ICE Expo/Annual Meeting Attendee	\$75	\$100	\$25	\$25
Retired Members	30			
Students	15	15	_	-
Social Guests	40	40	-	
Swan House Tour Atlanta Tour	60 60	60 60		-
Conf. Package 1	250	350	50	100
Conf. Package 2	450	600	50	100
Conf. Package 3	550	750	50	100
Executive Forum	550	750	50	100

Registration Options

ICE Expo/Annual Meeting Attendee receives 3-days' admittance to the Expo and to the Annual Meeting program

Conference Package 1* Receives admittance to 1 ONE-DAY course

Conference Package 2* Receives admittance to 2 ONE-DAY courses or 1 TWO-DAY course

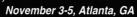
Conference Package 3* Receives admittance to 3 ONE-DAY courses or 1 TWO-DAY & 1 ONE-DAY course

^{*}All conference packages include admittance to the Expo and Annual Meeting Program



Do Not Use This Form After October 3, 1997

ICE '97, PRE-REGISTRATION FORM





Early-bird pricing is in effect until JUNE 30. For regular pre-registration, this form must be postmarked no later than OCTOBER 3, 1997. Form must be filled out completely for processing. A confirmation of your registration will be sent to you. ICE badges must be worn for admission to the ICE exhibits and all Technical Conference programs.

STEP 1. Industry Attendee Badge Information	STEP 2. Registrant Profile Are you a first-time attendee? 1 □ Yes 2 □ No
FIRST NAME (Nickname)	FSCT Society Affiliation
EIROT HAVE	Information below must be completed for registration to be processed:
FIRST NAME LAST NAME	Your Company (Check one only)
COMPANY	31 Manufacturers of Paints, Varnishes, 42 Environmental Services 43 Other
COMPANY	32 Manufacturers of Printing Inks
MAILING ADDRESS (or P.O. Box)	33 ☐ Manufacturers of Sealants, Caulks, Adhesives Your Position (Check one only)
	34 ☐ Manufacturers of Powder Coatings 51 ☐ Management/Administration
CITY STATE/P	PROV. 35 Manufacturers of Raw Materials 52 Mfg. & Engineering 53 Quality Control 53 Quality Control
	Containers 54 ☐ Research & Development
COUNTRY (other than U.S.) POSTAL CODE	Equipment 56 🗇 Sales & Marketing
	38 ☐ Government Agency 39 ☐ Research/Testing/Consulting 57 ☐ Consultant
TELEPHONE NO. FAX NO.	40
	41 Paint Consumer
STEP 3. Social Guest Badge Information	
FIRST NAME (Nickname)	FIRST NAME LAST NAME
CITY	ATE/PROV.
	Social Guest Registration
COUNTRY (other than U.S.) POSTAL CODE	(Welcome Social and Expo)\$40
TELEPHONE NO.	Optional Activities (Select only one—include selection in Step 5): Tues. Tour to Swan House\$60
	Tues. Tour of Atlanta Highlights\$60
STEP 4. Conference Registration: Conference registrant	is must check boxes of session they wish to attend (Not more than one box per day)
Monday, Nov. 3	
3,	uesday, Nov. 4 Wednesday, Nov. 5
ONE-DAY COURSES ONE-DAY COUR	ISES ONE-DAY COURSES
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General Information

Social Guest Program

ATLANTA, GA

This year's Social Guest program includes registration to attend a welcome program featuring Decorating Trends for Your Home, on Monday, November 3, as well as admittance to the FSCT Opening Session on Monday, November 3, and three days admittance to the Expo.

Social Guest registration is offered only to the spouse or guest of the industry attendees. To register, complete the Social Guest portion of the official FSCT registration form. Advance Registration fee is \$40. On-Site fee in Atlanta is \$50.

Optional Tours

Social Guests have the opportunity to register for optional guided tours to be conducted on Tuesday, November 4. Two choices are offered: The Goodlife in Historic Buckhead and A Taste of the Peach. Each will run from 9:30 am to 3:30 pm.

The tours include transportation on deluxe motorcoaches, admission fees to attractions specified, and lunch. To participate Social Guests must complete the Activities portion of the Social Guests Registration Form.

Tour 1: The Good Life in Historic Buckhead

features a visit to the Atlanta History Center offering participants a chance to tour the regal Swan House, the Tullie Smith House, and browse through the Atlanta History Museum. Following the morning tours, participants will travel to 103 West for a lunch at the posh Four Star restaurant. After lunch, participants will travel to Phipps Plaza and Lenox Square, two of Buckhead's favorite malls!

Due to limited availability, pre-registration is encouraged. \$60/person

Tour 2: A Taste of the Peach is a city tour highlighting the Olympic Village, the State Capitol, as well as the Historic District on Sweet Auburn Avenue. Participants will visit the Fox Theater as well as the Shops of Bennett Street. Following the morning tours, participants will travel to 103 West for a lunch at the posh Four Star restaurant.

After lunch, participants will travel to Phipps Plaza and Lenox Square, two of Buckhead's favorite malls!

Due to limited availability, pre-registration is encouraged. \$60/person

Shuttle Service

Shuttle service will be provided between the participating hotels and the Georgia World Congress Center.

Hours of Operation

Sunday, Nov. 2	8:00	am	to	5:00 pm
Monday, Nov. 3	7:00	am	to	6:00 pm
Tuesday, Nov. 4	7:00	am	to	6:00 pm
Wednesday, Nov. 5	7:30	am	to	3:00 pm

Pick-up locations and up-to-date schedule information will be posted at the Georgia World Congress Center and in the lobby of each participating hotel.

Travel Notes

The official carrier is Delta Airlines. To receive the special Delta convention rates*, you may wish to arrange your travel through the FSCT Travel Desk <u>OR</u> through Delta's Meeting Network.

If you book your travel through the FSCT Travel Desk, you will receive a chance to win round-trip transportation for two to FSCT's ICE '98 in New Orleans. To earn a chance at the drawing, you must book through the FSCT Travel Desk at 1-800-448-FSCT and mention the ICE Show

Should you or your travel professional desire to make travel arrangements through the Delta Meeting Network contact them at 1-800-241-6760 and refer to the convention file number I-3697.

*Certain restrictions apply.

Airport and Ground Transportation

Hartsfield International Airport serves the Atlantametropolitan area. With 2,000 flights a day, it's one of the world's largest passenger terminals.

Located in Hartsfield International Airport near the baggage claim area is a station for MARTA, Atlanta's rapid transit system. MARTA provides a fast and economical transportation option to the downtown area. Call 404-848-4711 for further details. Other transportation options from the airport are shuttle service and taxis. Atlanta Airport Shuttle provides scheduled service from Hartsfield to the downtown hotels every 15 minutes from 7 a.m. to 11 p.m. One-way runs \$8.00 and round-trip is \$14.00. The trip takes approximately 30 minutes. Taxi service from Hartsfield operates on a flat fee: \$18 for one person, \$20 for two people, and \$24 for three people.

Weather

Temperatures average $64^{\circ}F$ (17.8°C) in Atlanta during early November. In the evenings expect the average to be $46^{\circ}F$ (7.8°C).

Attire

Business attire is appropriate for all events.

Visitor details on Atlanta are available on the world wide web at "www.atlanta.com"

JOURNAL OF COATINGS TECHNOLOGY

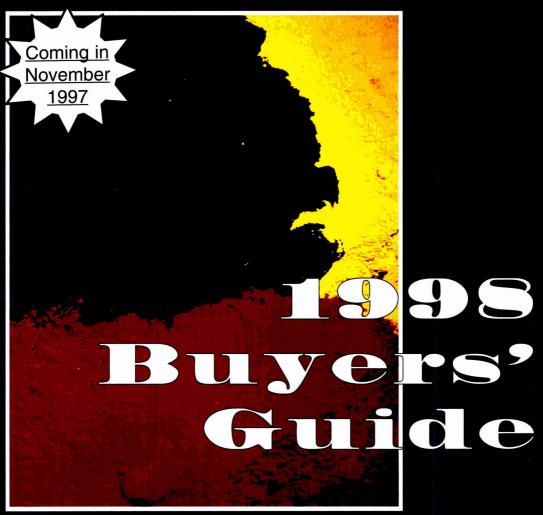


Photo courtesy of Sun Chemical Co.

The 1998 JCT Buyers' Guide will be published in the November 1997 issue of the JOURNAL OF COATINGS TECHNOLOGY. The JCT is produced by the Federation of Societies for Coatings Technology, sponsors of the International Coatings Expo and Technology Conference. Anyone wishing to participate should contact JCT, 492 Norristown Rd., Blue Bell, PA 19422, phone: (610) 940-0777; fax: (610) 940-0292.

For advertising information, contact Scott Walter, National Advertising Representative, 22785 Cass Ave., Woodland Hills, CA 91364: Phone (818) 224-3226; Fax: (818) 222-0194.

FEDERATION OF SOCIETIES



5K Run/Walk for Fun Registration Form



The ICE '97 5000 5K Run/Walk for Fun is the result of a cooperative effort by the Federation of Societies for Coatings Technology and Troy Corporation benefitting the Coatings Industry Education Foundation. We hope this activity provides a healthy and enjoyable start to your Tuesday morning. Complete this registration form and return it with your entry fee today!

RACE TIME: Tuesday, November 4, 7:00 am SHARP!!!

COURSE: 5 Kilometers around Lake Clara Meer in Piedmont Park

MEETING TIME: Meet at the Courtland Street entrance to the Atlanta

Marriott Marquis Hotel (Peachtree Center stop off MARTA) or the Hilton Towers (check hotel lobbies for

bus information) 6:30 am.

T-SHIRTS: YOU MUST PRE-REGISTER IN ORDER TO RECEIVE

A T-SHIRT!!!

TO REGISTER: Complete this form and return it with your \$5 per person

race fee to:

Troy Corporation/ICE '97 5000

8 Vreeland Road Florham Park, NJ 07932

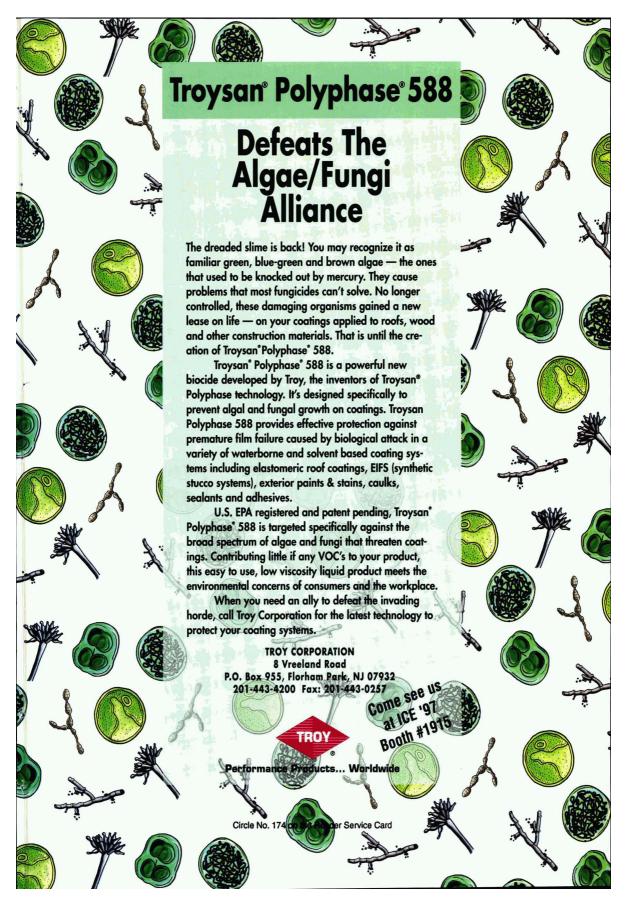


REGISTRATION:	Race Categories:
Name	O Male O Female
Company	O 5K Race O 5K Walk
Address City State Zip/Postal Code	○ 35 & Under ○ 36–49 ○ 50 & Over
Country	T-Shirt Size:
Phone Fax Date of Birth Age on Race Day	O Adult Large

WAIVER AND INDEMNITY: In consideration of this entry being accepted, I the undersigned intending to be legally bound, hereby for myself, my heirs, executors and administrators, waive and release any claims I may have against the Troy Corporation, The Federation of Societies for Coatings Technology, Georgia World Congress Center, The City of Atlanta, GA, race sponsors, their staff, officials, volunteers and any representatives, successors or assignees for any injuries that may be suffered by me in this event. I further hereby certify that I am physically fit and have sufficiently trained for the completion of this event.

SIGNATURE: _____ DATE: _____

YOU MUST SIGN THIS FORM IN ORDER TO PARTICIPATE!
RACE COMMITTEE RESERVES THE RIGHT TO MAKE ANY NECESSARY CHANGES OR ADJUSTMENTS.





World's Premier Coating Expo Features Over 300 Exhibitors

The largest coatings-related exhibition in the world—the FSCT International Coatings Expo (ICE)—will be held in conjunction with the FSCT 75th Annual Meeting and Coatings Technology Conference at the Georgia World Congress Center in Atlanta, GA. Over 300 supplier companies

1004

to the coatings manufacturing industry will be present to discuss their newest products and services. In over 95,000 sq. ft. of exhibits will be displayed a wide variety of raw materials, production equipment, containers and filling equipment, laboratory apparatus, and testing and application devices for the paint and coatings producer.

Key personnel from the top technical sales staff of supplier companies will be available to provide attendees with an opportunity to learn of the latest developments in their products and services.

ICE '97 Exhibitors with Booth Numbers (As of 8/15/97)

BASE Corn

Color indicates ICE '97 Sponsor

A.P. Dataweigh Systems2421
ABC Dispensing Technologies Inc.2414
Acetex Enterprises357
Aceto Corp921
ACT Laboratories1122
Adhesives Age503
Advanced Software Designs604
Air Products & Chemicals, Inc 929
Akzo Nobel Resins514
Alar Engineering Corp2048
Allied Colloids, Inc
AlliedSignal Inc
Alnor Oil Co., Inc701
Ambrose Co
ACS, Information & Services442
American Colors, Inc451
Amer. Paint & Coatings Journal 2512
Amoco Chemical Co1444
ANGUS Chemical Co 938
Anker Labelers USA Inc430
Anker Labelers USA Inc

D/131 COIP	1000
BatchMaster Software Corp	2004
Bayer Corp	1642
Blacoh Fluid Control, Inc	2349
Borden Chemical, Inc	
Boss Bulk Systems, Inc	
Bowers Process Equipment Inc	
Brookfield Engineering Labs., Inc	826
Buckman Laboratories, Inc	630
Buhler Inc	.2154
Bulkcon Systems USA	.2244
Burgess Pigment Co	.1422
BYK-Chemie USA	.1522
BYK-Gardner, Inc	.1624
Cabot Corp., CAB-O-SIL &	
Special Blacks Div	.1126
Calgon Corp	.2147
Cardolite Corp	.1543
CB Mills	
Cellulose Filler Factory Corp	.1549
Celotex-Testing Services	324
CEM Corp	.2538
Chemical & Engineering News	444
Chemical Marketing Reporter	824
Chemical Week	623
Chemicals Incorporated	505
Chemir/Polytech Laboratories, In	c. 602
Ciba Specialty Chemicals Additive	es,
Pigments, & Polymers Divs	.1230
CIBA Specialty Chemicals-	
Fluoro Chemicals	558
Cimbar Performance Minerals	.1045
Clariant Corp	722
Clawson Container Co	419
CMI International	

Coatings Magazine2103
Coatings World/Ink World Mag 402
Coldec Inc
Color Communications, Inc 1946
Color Corp. of America1910
ColorTec Associates2008
Columbian Chemicals Co 1726
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of Taotek NA2236
Corrosion Control Consultants
& Labs2107
Cortec Corp
CR Minerals Corp
Crosfield Co
Cuno, Inc
Custom Aerosol Packaging2548
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Sufficie Surfynol Surfactane

When it comes to formulating compliant coatings and inks, an ounce of prevention is worth a pound of cure. With Surfynol surfactants as part of your starting formulation, you can avoid the time consuming and costly process of balancing improved wetting with foam control.

Since Surfynol surfactants

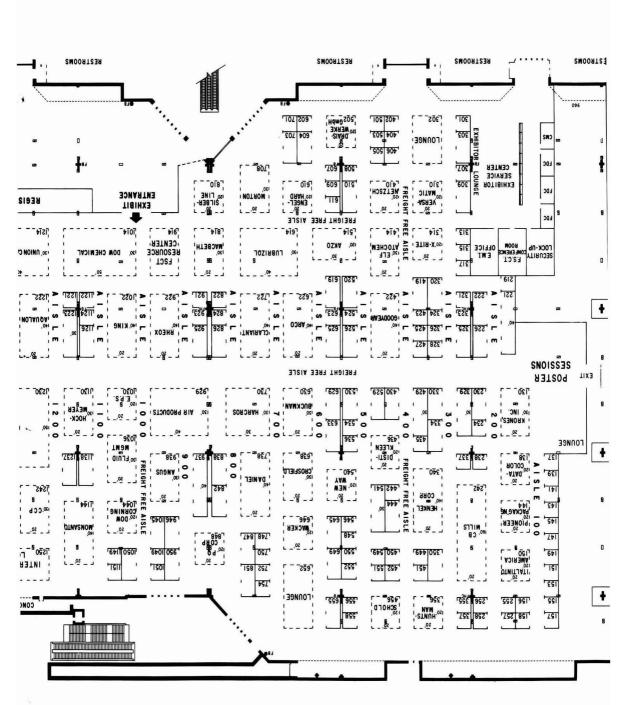
Since Surfynol surfactants lower both dynamic and equilibrium surface tension in waterborne systems, wetting is enhanced — which means customers get few or no defects, even with spray or very high application speeds.

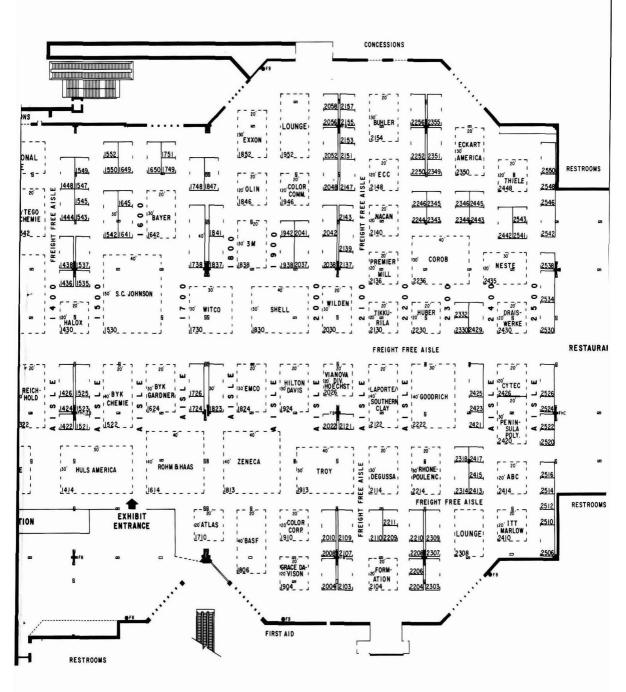
Surfynol surfactants also offer superior flow and leveling without foam. And they work in other compliant systems, including powder and high-solids. So make sure your time and talent are backed up by a wetting agent that's right from the start. Call 800-345-3148 or 610-481-6799 for a free sample. Or visit our web site at www.airproducts.com/chemicals.

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INTERNATIONAL COATINGS EXPO NOVEMBER 3-5,1997 GEORGIA WORLD CONGRESS CENTER ATLANTA,GEORGIA

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Estron Chemical, Inc
Fawcett Co
Coatings Technology 914 Fillite North America, Inc. 611 Filter Specialists, Inc. (FSI) 526 First Ten Angstroms 238 Fischer Technology Inc. 2121 Florida Drum Co. 337 Fluid Management 1036 Formation Systems, Inc. 2104 Franklin Industrial Minerals 2520 Fuji Silysia Chemical 1641 H.B. Fuller Co. 946 G A F Filter Systems 1542
GAF Filter Systems
Halox Pigments 1430 J.W. Hanson Co., Inc. 2309 Harcros Pigments Inc. 730 Henkel Corp. 340 HERO Industries Limited 847 Heucotech Ltd. A N.J. Limited Partnership Partnership 2210 Hi-Mar Specialties, Inc. 2332 Hilton Davis Co. 1924 Hockmeyer Equipment Corp. 1130

Horiba Instruments Inc
Minerals Div2230
Hüls America, Inc
HunterLab529
Huntsman Corp356
ICI Paints North America2522
ICI Surfactants1650
Ideal Manufacturing & Sales Corp. 309
IGT Reprotest Inc313
Indco, Inc
Industrial Copolymers Ltd2534 Infinium Software (formerly
Software 2000)633
Inmark, Inc1645
INOUE USA2550
Inpra-Latina2204
Interfibe Corp
International Compliance Center 334
International Speciality Chemicals .450
International Specialty Products (ISP)1542
Italtinto America Inc
ITT Marlow/ITT A-C Pump2410
Jevic Transportation Inc2256
S.C. Johnson Polymer 1530
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Technology 914
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Kelly Chemical Corp. 2355 Kenrich Petrochemicals, Inc. 2425 King Industries, Inc. 1022 Kline & Co., Inc. 425 KRONES, Inc. 130 KTA-Tator Inc. 510 Labelmaster 534 Laporte Inc./Southern Clay 2122 Products 2122 Laporte Inc./Aztec Peroxides, Inc. 2122 Laporte Inc./Laponite Rheological Additives 2122 Laporte Inc./Mapico 2122
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Kelly Chemical Corp. 2355 Kenrich Petrochemicals, Inc. 2425 King Industries, Inc. 1022 Kline & Co., Inc. 425 KRONES, Inc. 130 KTA-Tator Inc. 510 Labelmaster 534 Laporte Inc./Southern Clay Products 2122 Laporte Inc./Aztec Peroxides, Inc. 2122 Laporte Inc./Aztec Peroxides, Inc. 2122 Laporte Inc./Mapico 2122 Laporte Inc./Mineral Pigments 2122 Laque Corrosion Services 2514 Lawter International 429 The Leneta Co 1942 Liquid Controls Corp 1237 Littleford Day Inc. 2429 Longview Fibre Co 546 The Lubrizol Corp 614 Luzenac America 2445
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Kelly Chemical Corp. 2355 Kenrich Petrochemicals, Inc. 2425 King Industries, Inc. 1022 Kline & Co., Inc. 425 KRONES, Inc. 130 KTA-Tator Inc. 510 Labelmaster. 534 Laporte Inc./Southern Clay Products 2122 Laporte Inc./Aztec Peroxides, Inc. 2122 Laporte Inc./Laponite Rheological Additives. 2122 Laporte Inc./Mapico 2122 Laporte Inc./Mineral Pigments 2122 LaQue Corrosion Services 2514 Lawter International 429 The Leneta Co 1942 Liquid Controls Corp 1237 Littleford Day Inc. 2429 Longview Fibre Co 546 The Lubrizol Corp 614 Luzenac America 2445 3M/OSHD 1838 3M/Zeelan 1838 Macbeth (see Gretaq Macbeth) 814
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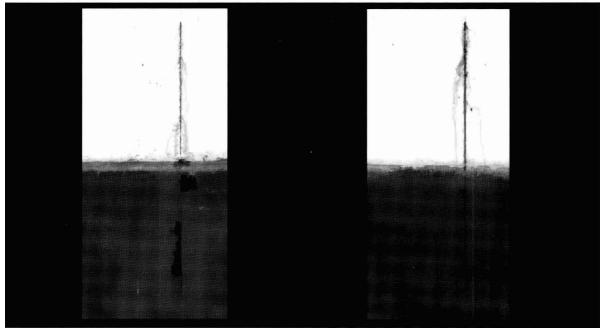
Michelman, Inc	.1151
Micro Powders, Inc	1823
Microfluidics International Corp.	2042
Micromeritics	
Micromet Instruments	
Microview USA, Inc	
Mid-States Eng. & Mfg	355
Ming-Zu Chemical Industries	
MiniFibers, Inc.	1050
Minolta Corp	838
Mississippi Lime Co	.2318
University of Missouri-Rolla	153
Mitsubishi Chemical	.1847
Modern Paint & Coatings	.2155
Monsanto Co	
Morton International	700
Muetek Analytic Inc	. 2041
Myers Engineering	520
Nacan Products Ltd	.2140
Nagase America Corp	
Nametre Co	2330
Neste OXO AB	
Netzsch Incorporated	
Neupak, Inc	937
New Way Packaging	
Machinery, Inc	540
Nichem Corp	609
Norman International	752
North American Oxide Inc	1/2/
North Dakota State University	
North Dakota State University	
Nyco® Minerals, Inc	
Nyco® Minerals, Inc	330
Nyco® Minerals, Inc Occidental Chemical Corp	330
Nyco® Minerals, Inc Occidental Chemical Corp	330
Nyco® Minerals, Inc	330
Nyco® Minerals, Inc	330 541 748 1846
Nyco® Minerals, Inc	330 541 748 1846 230
Nyco® Minerals, Inc	330 541 748 1846 230
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Nyco® Minerals, Inc	330 541 748 1846 230 2417 427 2137 219 2307 226 435
Nyco® Minerals, Inc	330 541 748 1846 230 2417 427 2137 219 2307 226 435 524
Nyco® Minerals, Inc	3305417481846230241742721372192307226435524
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Nyco® Minerals, Inc	330 541 748 1846 230 2417 427 2137 219 2307 226 435 524 2420 2420 1938 144 923
Nyco® Minerals, Inc	330 541 748 1846 230 2417 427 2137 219 2307 226 435 524 2420 2209 1938 144 923 1724
Nyco® Minerals, Inc	330 541 748 1846 230 2417 427 2137 219 2307 226 435 524 2420 2209 1938 1724 923 1724
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Silberline Mfg. Co., Inc
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Software)
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University of Southern Mississippi 149
Specialty Minerals, Inc
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Startech Services Inc
Startex Chemical, Inc
SSPC-The Society for Protective
Coatings2344
Stony Brook Scientific, Ltd 2153
Stretch-O-Seal Corp548 Summit Precision Polymers Corp607
summit Precision Polymers Corp 807
Tech Pak, Inc
Teemark Corp307
Tego Chemie Service USA1342
Testing Machines, Inc2056
Thiele Engineering Co2448
Thomas Scientific
Toyal America Inc1525
Troy Corp1915
U.S. Aluminum, Inc2442
U.S. Zinc Corp750
UCB Chemicals Corp./Radcure 1749
Union Carbide Corp1214
Union Process Inc1426
United Mineral & Chemical Corp. 1837

United Soybean Board649
Utah Clay Technologies, Inc 2206
Van Waters & Rogers, Inc329
VanDeMark Group328
R.T. Vanderbilt Co., Inc625
Versa-Matic Pump Co 310
Vianova Resins Inc2026
Victaulic Co. of America255
Vinavil Americas406
Vorti-Siv Div.
MM Industries, Inc619

Wacker Silicones Corp646
Western Equipment Co2516
Wilden Pump & Engineering Co 2030
Witco Corp1730
WSI Chemical, Inc449
X-Rite, Incorporated314
Zeiss Optical Systems, Inc
Zemex Industrial Minerals508
Zeneca Resins1813
Zeneca Biocides1813

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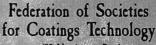
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PALS Free Volume Study of Dry and Water Saturated Epoxies

M.M. Madani, R.R. Miron and R.D. Granata—Lehigh University*

INTRODUCTION

rosslinked epoxy resins are used as protective coatings. A protective coating forms a barrier to prevent corrosion due to transport of the reactants or corrosives (such as water, oxygen, and ions) through the coating to the underlying substrate. The barrier properties of a protective coating are dependent on factors such as: (1) the solvent interactions between the reactants and the polymer matrix, i.e., dispersion, permanent dipole, and hydrogen bonding interactions¹; (2) the size and density of the free-volume cavities in the polymer network²; (3) the existence of defects in the coating in the form of voids, transport pathways, and capillaries3,4; and (4) the presence of voids at the coating/metal interface.2 The free volume of the polymer includes the molecular space between the polymer chains available for thermal motion and is an important component of reactant transport.

The purpose of this paper is to characterize the free volume properties as determined by positron annihilation lifetime spectroscopy (PALS) of two types of epoxies in respect to their behavior as protective coatings (a good coating based on a bisphenol A epoxide and a poor coating based on a poly(propylene glycol) epoxide⁵) and their mixtures and the effects of three corrosion inhibitors. The effect of water on the physical structure of the epoxy resins was also studied.

PALS Background

The concept of free volume is often applied to interpret behavior of a polymer melt or glass, or to study physical aging effects when configurational (e.g., crystallinity) thermodynamic quantities like volume and enthalpy or viscoelastic properties are investigated. Several techniques are available to study the free volume and physical properties of the polymer on a molecular level. Among these are photochromic and fluorescent spectroscopy,^{6,7} small angle diffractions,⁸ and positron annihilation lifetime spectroscopy (PALS).⁹⁻¹¹

Positrons formed from natural radioactive decay of ²²Na have a maximum kinetic energy of 0.545 MeV and penetrate condensed matter while dissipating kinetic energy via ionization, plasmon, electron-hole genera-



Free volume cavity sizes and fractions of epoxy specimens were determined using positron annihilation lifetime spectroscopy (PALS). PALS data were obtained before and after specimen water equilibration. Specimens were bisphenol A ep-

oxide (B) and/or glycol epoxide (G) cured with a polyamide. Free volume sites increased linearly and cavity sizes decreased linearly with epoxide B:G ratio. Glass transition temperature (T_g) increased with epoxide B:G ratio. Water molecules in wet epoxy B filled approximately six percent of the cavities. Epoxy G cavity size increased 11.4% after water equilibrium and was ascribed to cavity expansion. PALS results differed for commercial corrosion inhibitors in wet and dry mixtures of these epoxides.

tion, and elastic scattering in a process known as thermalization. 9-14 Thermalized positrons then annihilate with electrons yielding two photons each with an energy of 511 keV (gamma rays). The mechanism of the annihilation in molecular materials (such as insulators, polymers, gases, etc.) differs. In molecular materials a thermalized positron extracts an electron to form a bound state known as the positronium (Ps), a hydrogen-like atomic structure. Ps exists in two ground states depending upon the spin-spin interaction between an electron and a positron. 9-13 A singlet state (anti-parallel spin) is called a para-positronium (p-Ps) and has a lifetime of 0.125 ns in a vacuum. The triplet state (parallel spin) is called ortho-positronium (o-Ps) and has a lifetime of 140 ns in vacuum. The annihilation of p-Ps gives two photons each having 511 keV. In vacuum, o-Ps produces three photons of varying energies with a total energy of 1.022 Mev. In molecular materials, o-Ps undergoes many collisions with the surrounding electrons which can re-

Table 1—Formulation of Specimens Used for PALS Measurements

Specimens	Composition by Weight	Curing Conditions
Ероху В (100%)	100 parts B 66.7 parts polyamide 0.25 parts surfactant	100°C, 2 hr
Ероху В + Ероху G (70/30%)	35 parts B 15 parts G 33.3 parts polyamide 0.25 parts surfactant	100°C, 2 hr
Ероху В + Ероху G (50%/50%)	25 parts B 25 parts G 33.3 parts polyamide 0.125 parts surfactant	100°C, 2 hr
Epoxy B + Epoxy G (30%/70%)	15 parts B 35 parts G 33.3 parts polyamide 0.125 parts surfactant	100°C, 2 hr
Epoxy G (100%)	100 parts G 66.7 parts surfactant 0.25 parts surfactant	100°C, 2 hr
Epoxies + Inhibitor	87 parts epoxy mixture 15 parts inhibitor	100°, 2 hr

act with the o-Ps atom annihilating into two photons. This normal decay process known as pickoff reduces the lifetime of o-Ps to 1-9 ns. $^{14-19}$

* = Inhibitors (silicate, borate, or nitrophthalate)

Following thermalization, positron and positronium tend to become localized (trapped) in regions of low electron density as a result of repulsion from the surrounding atoms (exchange repulsion and Pauli exclusion). These low electron density sites can be surfaces or voids (for positron and Ps), defects such as vacancies and dislocations (for positron), or the free volume in polymers (for Ps). As the size of these regions increases, the local electron density decreases. This decrease causes an increase in the lifetime of the trapped o-Ps due to the reduced probability of an interaction with an electron. The intensity of o-Ps annihilation increases with the number of the low electron density regions (free volume) due to higher o-Ps formation and localization rates. 17 Formation probabilities and annihilation characteristics of Ps in free volume strongly depend on the chemical and physical properties of the materials.15

Three or four lifetimes are found in positron annihilation in polymers. The shortest lifetime (0.1-0.2) ns is usually associated with annihilation of the p-Ps and Ps-molecular species; the intermediate lifetime (0.2-0.4) ns with free annihilation of the positron; and the longer lifetime (1-9) ns with the decay of o-Ps by electron pickoff. ¹⁶⁻¹⁹

Polymer/Water Interactions

Diffusion and sorption of water in epoxies have been studied extensively. 20-28 These studies reported that the water molecules can cluster, craze, and plasticize epoxy matrices accelerating diffusion in the polymer. Moy and Karasz²⁶ have shown that the equilibrium sorption of water in an epoxy resin is markedly affected by curing conditions. It is expected that curing at higher tempera-

ture will increase the crosslink density as well as increase the number of hydroxyl groups in the polymer. They found a decrease in the rates of sorption with increasing cure temperature which suggests that sorption and transport of water vapor in the glassy state of the epoxy is predominantly controlled by morphological features, i.e., by the crosslink density rather than by increase in the number of possible sorption sites. Although in a more dense and crosslinked network the number of hydrophilic sites is higher, the ability of the moisture to diffuse into the available free volume is lower. In low glass transition temperature and less rigid epoxy systems, as expected for crosslinking occurring mainly by reaction of all amine groups, no appreciable sorption was found to occur by the hydrogen-bonding mechanisms. Most of the sorption can then be attributed to the dilution of the free volume.26 The o-Ps lifetimes and intensities are sensitive to the presence of water molecules in the epoxy matrix.²⁷ Relationships between water sorption and indirect measurements of

polymer free volume, such as specific volume and mechanical properties, have been reported.²⁸

PALS was employed for this investigation to study the relationship between free volume as determined by PALS and epoxy/water interactions. In addition, the behavior of the commercial inhibitors (silicate, a borate, and nitrophthalate) mixed with epoxy resins was also studied.

EXPERIMENTAL

Specimen Preparation

Two commercial liquid epoxide resins were used. Epoxy B is a diepoxide, the diglycidyl ether of bisphenol A (DGEBA)^d with an equivalent weight (EW) of 174. Epoxide G is a polyglycol diepoxide^e with an EW of 190 containing hydroxyl groups. A polyamide resinf was used for crosslinking the epoxide resins. A small amount of surfactant^g was added to each formulation. *Table* 1 provides formulation details.

Three commercial inhibitors—silicate, borate, and nitrophthalate—were evaluated in formulations prepared with the epoxy resins using a Hoover Automatic Muller^h Model 48.

⁽a) Halox SZP-391[™] (calcium strontium phosphosilicate), Halox Pigments, Div. of Hammond Lead Products, 1326 Summer St., Hammond, IN 46320-2240.

⁽b) Butrol 22™ (barium metaborate monohydrate), Buckman Laboratories International, Inc., 1256 N. McLean Blvd., Memphis, TN 38108-1241.

⁽c) SICORIN-RZ™ (1,3-benzene dicarboxylic acid, 5-nitro, Zn salt, 1:1) BASF Corporation, 491 Columbia Ave., Holland, MI 49423,

⁽d) D.E.R.™ 332, epoxide resin, Dow Chemical Co., Midland, MI 48641;

⁽e) D.E.R.™ 736, epoxide resin, Dow Chemical Co., Midland, MI 48641.

⁽f) UNI-REZ™ 2140 (high imidazoline, unsaturated polyamide), Union Camp Corporation, P.O. Box 3301, Princeton, NJ 08543.

⁽g) BYK 306™ (polyether modified dimethylopolysiloxane copolymer surfactant), BYK Chemie USA, 736 Bedford Rd., Grosse Pointe Park, MI 48230-1803.

⁽h) Hoover Color Corp., P.O. Box 218, State Highway 693, Hiwasee, VA 24347;

Inhibitor concentrations were 15% (w/w) in epoxy resin. All specimens were cured at 100°C for two hours in aluminum pans unless noted otherwise. After curing, the specimens were removed from the aluminum pans and the thicknesses were 2 to 4 mm, sufficient for stopping all positrons. Differential scanning calorimetry (DSC) was used for determining the glass transition temperature (Tg) of the epoxies and their mixtures (Figure 1). Measurements were made using 15-20 mg samples from 10 to 100°C for one set and from –50 to 100°C for a second set using scanning rates of 5 and 10 deg/min, respectively. The Tg values were calculated from the heat flow plots and the averages of several measurements were reported. The Tg increased as the epoxide B:G ratio increased.

PALS

A standard fast-fast coincidence system based on fast plastic scintillators, RCA photomultipliers, and EG&G Orteci electronics was used. The source for positrons was 20 μCi of ²²Na sealed in 25 μm thick Kapton foils. The positron source was placed between two identical specimens in a sandwich configuration. The details of the experimental setup have been reported elsewhere.11 The ²²Na source decays simultaneously to a positron and a gamma photon with an energy of 1.28 MeV. The time interval between the creation (1.28 MeV signal) and annihilation (511 keV signal) is the lifetime of the positron in matter. The spectrum was measured until 60,000 counts were accumulated in the peak channel. A 22.5% positron annihilation intensity with a lifetime of 365 ps in the Kapton source was subtracted from each spectrum as a source correction. The positron lifetimes, intensities, and uncertainties were obtained from the raw data using the program Positronfit.²⁹ Measurements were made under similar conditions of temperature and relative humidity for all specimens.

Figure 1—Glass transition temperature of the epoxy specimens as a function of Epoxy B concentration.

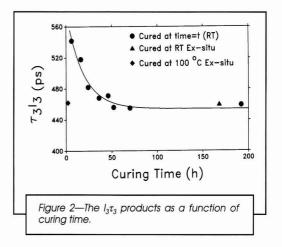
Table 2—The PALS Lifetimes and Intensities in Epoxy B In Situ

Curing Time (hr) at RT	τ_3 (ps)	l ₃ (%)
6	2294 ± 75	23.6 ± 0.8
16	2252 ± 60	23.0 ± 0.7
24	2024 ± 40	23.8 ± 0.6
36	2059 ± 35	22.7 ± 0.5
46	1975 ± 18	23.8 ± 0.5
52	2005 ± 17	22.8 ± 0.5
70	1995 ± 15	22.8 ± 0.5
192		23.1 ± 0.6
7 days, RT*, ex-situ	1976 ± 16	$23.9 \pm 05.$
2 hr, 100°C, ex-situ		23.4 ± 0.5

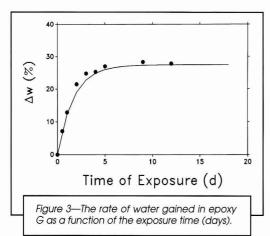
RESULTS AND DISCUSSION

PALS of 100% Epoxy B During Cure at Room Temperature

PALS data were obtained from 100% Epoxy B as a function of curing time at room temperature (RT). Spectra were taken during the first six hours of the cure cycle and then continued up to 192 hr. Three lifetimes and intensities were resolved in the PALS analyses. The results of these experiments are shown in Table 2 and *Figure* 2. The third lifetime intensity was $23.0 \pm 1.0\%$. The third lifetime decreased from 2.3 ± 0.06 ns to 1.97 ± 0.02 ns within 192 hr. The long lifetime during the first six hours in the curing mixture indicated that a large free volume cavity existed in the mixture which dramatically decreased as the epoxy crosslinked and cured. The I₃t₃ products decreased within the first 50 hr of curing time at RT (Figure 2). From Table 2, the third lifetime remains constant within experimental error after 50 hr, indicating that the free volume cavities formed and a specific cavity size was established even though the epoxy was



⁽i) EG & G Ortec, 100 Midland Rd., Oak Ridge, TN 37830-9102.)



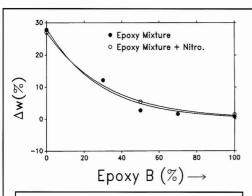
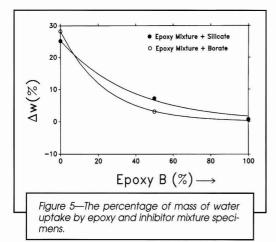


Figure 4—The percentage of mass of water uptake in epoxy and epoxy plus nitrophthalate inhibitor as a function of epoxy B concentration.



not cured completely. The solid line in the *Figure* 2 is the result of an exponential fit to the raw data given by:

$$I_3 \tau_3(t) = I_3 \tau_3(\infty) + C \times \exp(-\alpha t)$$

where t is curing time in hours; $C = 126.3 \pm 10.4$ ps is the $I_3\tau_3$ change associated with the transition from uncured to cured and depends only on the epoxy material; I₃τ₃ $(\infty) = 454.5 \pm 15.5$ ps is the maximum (final) $I_3\tau_3$ value after reaction takes place at room temperature. The value of α is related to the chemical constant of the reaction (first order). Two additional tests were made on the same type of epoxy: one cured at room temperature for seven days (triangle in Figure 2) and other cured at 100°C for two hours (diamond in Figure 2). For these cured materials the third lifetimes and intensities were equivalent. This indicates that the free volume fraction of epoxy B was independent of the degree of cure: DSC showed that 66.6% of epoxy B was cured at room temperature and 99.9% was cured after baking at 100°C for two hours.30 The subsequent experiments were performed on epoxy specimens prepared and cured at 100°C for two hours.

An interesting conclusion is that the epoxide and curing agent form a liquid structure which is not very different from the cured material. The concentration of cavities remains constant while the cavity size decreases during cure. The reactants align in the liquid state establishing the cavity concentration, while the cavity size decreases as the crosslink bonds are formed.

Direct Water Uptake and PALS Measurements

PALS was performed on the cured epoxy mixtures given in Table 1. The first measurements were taken (dry) before immersion in water. The specimens were weighed using a high sensitivity balance accurate to six significant figures under similar laboratory conditions. Next, all specimens were immersed in distilled water for 18 days. This was more than a sufficient time for water uptake to reach equilibrium (see Figure 3 and epoxies G and B).5,27 After exposure to water, the specimens were blotted dry with paper towels to eliminate the liquid water from the surface and then weighed again (within 10 min after removal from water) to determine the amount of water absorbed by the specimen (direct measurements). PALS measurements were made on the wet specimens. During the PALS measurements, the whole source/sandwich configuration was kept in a Kapton bag containing moist paper to maintain water equilibrium (100% RH). All spectra were analyzed with three lifetimes and intensities. The results of direct measurements are shown in Table 3 and Figures 4 and 5 for epoxy and epoxy plus inhibitor mixtures. The water absorption in the 100% epoxide B matrix was 0.8% after 18 days versus 25-28% for 100% epoxide G. The amounts of water gained by epoxy B and its mixtures with corrosion inhibitors were the same and independent of the presence of corrosion inhibitor (within experimental error). However, in *Figure* 5, the amount of water gained by the epoxy G containing silicate inhibitor is approximately two percent less than the amount of water gained by the epoxy G alone. The mixture containing borate inhibitor

Table 3—Water Uptake by Specimens in 18 Days

Specimens	Inhibitors	Mass (g) before Water Uptake	Mass (g) of Water Gained	Δ wt%
B (100%)	_	11.462	0.086	0.75
B + G (70%/30%)	 -	22.457	0.367	1.63
3 + G (50%/50%)	_	30.598	0.845	2.76
3 + G (30%/70%)	_	22.232	2.724	12.25
G (100%)	_	18.462	5.13	27.78
3 (100%)	Silicate	15.976	0.3	1.88
3 + G (50%/50%)	Silicate	31.470	2.227	7.08
∋ (100%)	Silicate	19.981	5.16	25.82
3 (100%)	Borate	11.684	0.09	0.77
3 + G (50%/50)	Borate	29.296	0.93	3.17
∋ (100%)	Borate	20.393	5.76	28.24
3 (100%)	Nitrophthalate	7.142	0.105	1.47
3 + G (50%/50%)		24.553	1.133	4.61
G (100%)		28.620	7.721	27.0

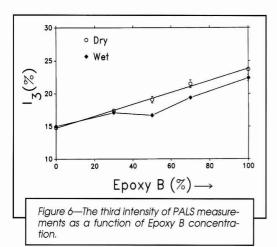
had 1.5% more water uptake than 100% epoxy G. The inhibitors provided useful anti-corrosion performances based upon low frequency impedance data.³¹ This inhibition was observed only when the electrolyte was in contact with the metal substrate containing dissolved inhibitor (not when present in the coating), indicating that free volume/inhibitor properties do not directly affect anti-corrosion performance. For this inhibition mechanism, there is a likely advantage to materials which minimize water uptake (silicate) and the resulting impact on coating properties.

Free Volume Cavity Concentration (Dry)

The o-Ps intensity values for the dry epoxy specimens, as a function of the Epoxide B:G ratio, are shown in Figures 6-8. Epoxy G had a lower concentration of free volume cavities (15% versus 23%) with a larger free volume cavity size (τ_3) with respect to epoxide B. Because epoxy G absorbed a larger volume fraction of water than epoxy B (measurements were made at room temperature, above the T_g of epoxy G), the epoxy G might be expected to have a larger concentration of free volume cavities than epoxy B and thus offer poorer corrosion protection than epoxy B, as reported.5 However, epoxy G has a lower concentration of free volume cavities with larger cavities than epoxy B due to the structural differences between the epoxide molecules. The epoxide B molecules contain phenyl rings while the epoxide G molecules are comprised of aliphatic chains. The aliphatic chain allows more rotational degrees of freedom, resulting in an epoxide molecule that has more possible configurations than the epoxide B molecule. Upon crosslinking, epoxies containing the epoxide B contribute to a rigid structure while those containing the epoxide G contribute to a less rigid, coiled structure (capable of flexible packing or swelling). The o-Ps intensity (Figure 6) increases linearly with the ratio of epoxide B while the lifetime decreases (Figure 7). The free volume fraction increases linearly as a function of the epoxide B:G ratio for dry specimens (Figure 8). The consistency between the T_o (Figure 1) and I₃ (Figure 6, dry) is shown. There is a correspondence between the T_g (Figure 1) and I₃ (Figure 6, number of free volume sites per unit volume) as the number of free volume cavities increased, the T_g of the epoxy increased. Gupta and Brahatheeswaran³² report that those epoxies with a high crosslink density have relatively higher free volumes at and below T_o because the crosslink sites do not provide a suitable environment for close packing of the rigid molecules. They also postulate that in highly crosslinked specimens the free volume distribution is made up of a large number of relative smaller free volume cavities. Their conclusion is consistent with the PALS results that show increasing the ratio of the rigid epoxy (epoxide B) increases the number of free volume cavities (Figure 6) and decreases the cavity size. A possible explanation of this linearity is that epoxide B and epoxide G react with the polyamide forming rigid and flexible groups, respectively, which are primarily responsible for the size of the cavities. The size determined by Positronfit is the average of these two cavity size fractions (sum of all cavity volumes divided by the total number of cavities). The distribution of the cavity sites is random throughout the polymer bulk and a linear function of epoxide ratio for the systems studied.

Free Volume Cavity Concentration (Wet)

Two phenomena occurred in the epoxy mixture as water molecules saturated the epoxy matrix. First, as shown in *Figure* 6, the intensity (free volume cavity concentration) decreases for wet versus dry specimens, except for the higher ratios of epoxide G (30/70 and 100%). Lifetime values show an opposite effect (Figure 7). The intensity for dry specimens was a linear function of the epoxide B ratio. In wet specimens the intensity was almost linear above 50% epoxide B, with a discontinuity below 50% (Figure 6). This suggests that water molecules have two types of interactions with the epoxy matrix. As discussed in the preceding section, epoxy B created a rigid, coiled structure and did not allow water molecules to break bonds or interact with hydrophilic groups. First, water fills the available free volume cavities of the epoxy B matrix. The concentration of the free volume decreases due to inhibition of o-Ps formation reducing



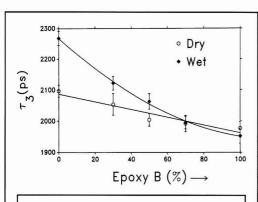
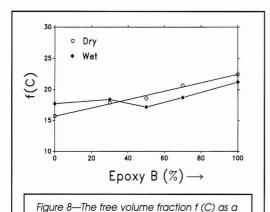


Figure 7—The third lifetime of PALS measurements as a function of epoxy B concentration.



the intensity of o-Ps. Second, water molecules can have polar interactions with aliphatic chains and free volume cavities causing swelling and expansion of the epoxy matrix. As a result of such interactions, the lifetime of the o-Ps increased due to the expansion of free volume. In the 50/50 specimen, both phenomena occurred causing a reduction in the intensity and an expansion in the free volume cavities. The discontinuity in the line (Figure 6, wet) below 50% epoxy B is consistent with water molecules having a polar interaction, rather than with water molecules filling the cavities. The lifetime in 100% epoxy B did not change significantly, which indicates that upon absorbing water, the free volume cavities fill with water molecules thus inhibiting o-Ps formation in the large cavities. It has been shown that Ps forms solely within cavities.⁹⁻¹¹ At percentages of 50% and higher epoxy B, water caused decreases in intensity (Figure 6) by filling the cavities.

The free volume size can be calculated from 9-11

$$\tau = 0.5 \times [1 - R/R_0 + 1/2\pi \sin(2\pi R/R_0)]^{-1}$$
 (2)

where $R_0 = R + \Delta R$, and $\Delta R = 1.656 \text{Å}$ is a layer of an electron cloud around the o-Ps in the spherical cavities. The free volume cavity will be found from $V_c = 4/3 \pi R^3$. From V_c, two to four water molecules can be accommodated in each of the free volume cavities reducing the o-Ps intensity.⁵ After water equilibrium occurred in the specimens having more than 30% epoxide G, a significantly longer lifetime (τ_3) was observed. This result indicated that the presence of water molecules increased the free volume cavity size as evidenced by the macroscopic swelling of these specimens.⁵ The nonlinear curve of τ_3 versus epoxide B ratio (Figure 7, filled diamond) and the direct measurement of the water uptake in the specimen (Figure 3, filled circle) are consistent. The presence of water molecules in epoxy G stretch the coiled polymer and plasticize the epoxy, increasing the free volume cavity size.5 This larger free volume allows more than two to four water molecules to fit into the cavity. The polar interaction between water molecules and polymer cavities expanded the size of the polymer cavities. The net volume is the difference between the polymer cavity volume and the volume occupied by the water molecules. This net volume is the free volume measured by PALS. The value of I₃ in epoxy G remained constant in both wet and dry measurements while τ₃ increased significantly (11.6%). After the specimen dried out, physical cracks appeared throughout the whole specimen indicating stress release in the molecules due to the polymer rearrangement.

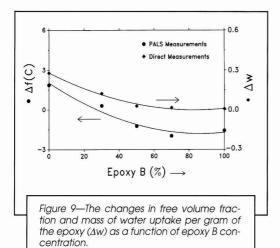
Free Volume and Water Uptake

The free volume of an intact polymer is defined as:

$$f = V_f / (V_o + V_f) = V_c \times C_f$$
 (3)

where V_f is the free volume, V_o is the volume occupied by polymer molecules, and $V_o + V_f$ is the specific volume of the polymer. V_c is free volume cavity as calculated from equation (2) and C_f is concentration of free volume. For a bulk polymer (all positrons annihilate within the polymer), the concentration of free volume cavities per

function of epoxy B concentration.



unit volume, C_t , is proportional to the o-Ps intensity as given by:

$$C_f = A \times I_3$$
 (4)

where A is a proportionality constant, and it is assumed that $\rm I_3$ = 0 when $\rm C_f$ = 0 (perfect crystalline structure). The relative free volume fraction as determined by PALS is given by,

$$f_p = V_c \times I_3 \tag{5}$$

The relationship between free volume fraction and absolute free volume fraction is:

$$f = A f_n$$
 (6)

Substituting equation (5) into equation (6) then we have:

$$f = A \times V_c \times I_3 = V_c \times C_f \tag{7}$$

For simplicity we assume the constant, A = 1.

For specimens whose free volume fraction f decreases after water equilibrium, the decrease is caused by the presence of water in the free volume which increases the pickoff annihilation rate and inhibits the formation of o-Ps. The free volume fraction after water saturation, where no swelling occurs, can be expressed as:

$$f_{e} = f - f_{w} \tag{8}$$

where f_e is free volume fraction after water equilibrium, f is free volume fraction of the dry specimen and f_w is the free volume fraction occupied by water molecules. Since swelling occurs in epoxy G and its mixtures, the free volume fraction after water equilibrium can be written as:

$$f'_e = f' + f_s \tag{9}$$

where f_e' is the free volume fraction after water equilibrium, f' is the free volume fraction of dry specimens and f_s is the free volume fraction created by the swelling of the epoxy. From equations (8) and (9), one can derive an equation for the free volume fraction, f(C), dependent on epoxide B ratio as:

$$f(C) = C \times f_e + (1 - C) \times f'_e$$
 (10)

where C is the mass fraction of epoxy B ratio between 0 to 1.0. The free volume cavity size was calculated for each epoxy mixture, and results are shown in *Table* 4. The values in *Table* 4 were used to plot Δf (C) in *Figure* 9 (note in the case of dry polymer, $f'_e = f' = f$). However, the relationship for the epoxy mixture is not linear after water uptake, as observed from the rate of water gained in the specimens and the change in free volume fraction. The change in free volume fraction, ΔF (C) = f_{wet} (C) - f_{dry} (C) and the normalized mass of the water uptake were fitted to an empirical quadratic equation (the solid line in *Figure* 9), as:

$$\Delta F(C) = 2.035 - 0.088 \times C + 5.084 \times 10^{-4} \times C^{2}$$
 (11)

where C is concentration of epoxy B in the mixture. The mass of water sorbed per gram of the epoxy (Δw) was fitted to:

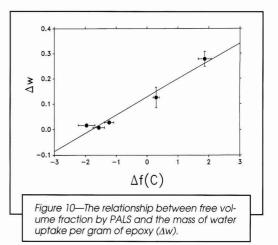
$$\Delta w$$
 (C) = 0.279 - 0.00671 × C + 4.018 × 10⁻⁵ × C² (12)

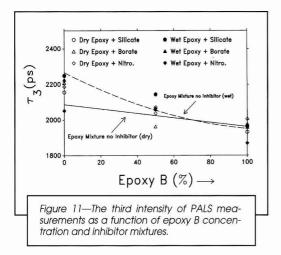
From equations (11) and (12), one can derive an approximate relationship between free volume fraction and mass of water uptake per gram of epoxy:

$$\Delta w (C) = k + D \times \Delta f (C)$$
 (13)

where k is the maximum amount of the water that can be absorbed in one gram of polymer without changing the volume of the polymer, and D is a proportionality factor. Equation (13) expresses the a relationship between the PALS and direct mass measurements. Figure 10 shows this correlation with $k=0.128\pm0.023$ and $D=(7.1\pm0.6)\times10^{-2}$.

The interactions of plasticizer and polymer have been reported 33,34 with each polymer and plasticizer having different interactions. Plasticization of epoxy polymer by water is known to lower $T_{\rm g}$. However, three theories have been proposed to describe plasticization 36 : (1) the gel and (2) lubricity theories describe the increase in flexibility of polymer upon plasticization while the (3) free volume theory of plasticization attempts to describe the mechanism of the plasticization process. The lower-





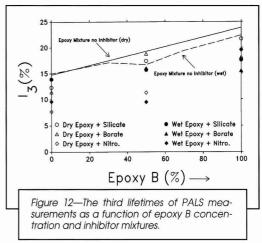
ing of a polymer T_g by a plasticizer is thought to be caused by an increase in free volume of the polymer at any temperature. The free volume fraction increases the degree of plasticization in the polymer³⁷; therefore, the plasticized polymer must be cooled to a lower temperature to reduce its free volume fraction to that observed at T_o. However, the PALS results show the fraction of free volume in epoxy B decreases after water uptake at room temperature, which is contradicted with free volume theory of plasticization. The PALS data are more consistent with the gel model.³⁴ When $\Delta f < 0$ the free volume fraction decreases as water is absorbed in the epoxy B and the 70/30 specimens resulting in antiplasticization. For ratios less than 50% epoxy B, Δf became positive and data showed more plasticization of the polymer (Figure 9). This statement is consistent with increasing the size of the free volume cavity. If Δf (C) < 0.0, the water molecules filled the epoxy free volume cavity (antiplasticization). If Δf (C) > 0.0, the free volume expanded (plasticization).

These results for mixed epoxy systems demonstrated a relationship between PALS parameters (free volume) and gravimetric measurements of water uptake.

Inhibitors and Epoxy Mixtures

PALS performed on dry corrosion inhibitors revealed three lifetimes in the silicate and borate inhibitors, but only two in the nitrophthalate inhibitor. The third lifetime and the corresponding intensity of the silicate inhibitor were 1.982 ± 0.125 ns and $2.1 \pm 0.2\%$, respectively. The third lifetime in the borate inhibitor was 2.596 ± 0.123 ns with an intensity of $2.0 \pm 0.1\%$. A longer lifetime (τ_3) is likely quenched by the nitro group in the nitrophthalate inhibitor.²⁷

Mayo et al. reported that positrons behave quite differently in dry pigment than in pigment wetted with solvents or oligomers, because some of the pigments are totally crystalline ($I_3 = 0$) materials with no amorphous phase. Accordingly, the lifetimes and intensities for the inhibitor's particles are not expected to be the same as when wetted by the epoxy resins in formulations. Three



lifetimes and intensities were found in all the epoxy and inhibitor specimens both dry and wet. The results are summarized in *Figures* 11-12. The relative inhibitor volumes with respect to the total specimen volumes were 6.6, 7.2 and 9.6% for the silicate, borate, and nitrophthalate inhibitors, respectively. The solid lines in *Figures* 11 and 12 are the PALS results for the epoxy mixtures (no inhibitor, dry). In each inhibitor/epoxy mixture, the intensities of the third lifetimes are below the solid lines (*Figure* 12). As the inhibitor surface is wetted by the epoxy resin, the adsorbed resin will have a lower free volume than the epoxy resin in the bulk and the inhibitor/epoxy mixture will have a lower intensity.³⁸

For the dry inhibitor/epoxy B (100%) systems, the I₃ values are larger relative to the equilibrated wet systems. However, the opposite was observed for the nitrophthalate-filled epoxy specimen due to quenching by the nitro group. The τ_3 values were not influenced by inhibitor type in epoxy B (*Figure* 11). In the case of epoxy G with different inhibitor mixtures (dry), the intensities increased slightly after the specimens were exposed to water (wet). This may be due to o-Ps interaction with the interfacial region (inhibitor particles/epoxy matrix) expanded by water uptake.³⁹ The τ_3 values in epoxy G with inhibitors were greater than the τ₃ values in epoxy G without inhibitors- which may also be due to the interfacial region.³⁸ In the case of 50% epoxy G mixtures, the wet specimens' I₃ values decreased versus dry specimens. This decrease is due a fraction of the voids being occupied by water molecules which inhibits the formation of o-Ps.

These relative changes suggest that significant free volume changes due to a specific inhibitor or filler may be determined relative to nitrophthalate. The nitrophthalate material in this case serves as a probe within the polymer matrix due to its o-Ps quenching property. Thus increases or decreases in free volume (see Figure 11) may be attributed to the inhibitor (filler) and correlated with barrier properties. Specifically, Figure 11 shows that nitrophthalate has a much smaller influence on $\rm I_3$ at 100% epoxy B than at lower percentages. An intriguing point is that the nitrophthalate can

no longer quench o-Ps in 100% epoxy B. This obervation suggests that the electronic properties of nitrophthalate have been altered by encapsulation in 100% epoxy B with respect to adjacent o-Ps/polymer interactions. Thus the polymer filler material can have no chemical role in the coating unless the encapsulation by the polymer is compromised.³¹ The effectiveness and mechanism of reactive fillers in coatings is important for corrosion inhibitors and other additives. Additional studies of this topic are warranted but are beyond the scope of this paper.

Models of Protective Coatings Systems

This work describes methods for improving the understanding of water interactions with polymer coatings used for protection from environmental degradation. Water exposure may have insignificant effects on some polymer systems or damaging effects on others. Anticorrosive pigments interact with water to perform their corrosion inhibition functions. Water is also critical to most low temperature corrosion processes influencing conductivity and participating in both reduction and oxidation half-reactions. From one perspective, water is the common factor for both degradation and repair processes involving protective polymer coatings.

Three types of water penetration occur in polymer systems¹: (1) polymer-diluent solution; (2) adsorption on hydrophilic sities; and (3) adsorption on free volume elements or cavities. The first type has a minor influence on systems containing hydrophilic groups (systems similar to those studied here). The second and third types have been used in our discussions which indicate that both occur. Polymer sensitivity to water can result in plasticization rationalized by models based on dilution (polymer/solvent), on entropy (configuration/energy change), and on intermolecular hydrogen bond rupture. Determining the models applicable to a specific polymer system is often complicated. Further, inclusion of filler materials (anti-corrosives) in the polymer matrix creates more difficulties in understanding the effects of water on a protective coating system. In solid polymer electrolyte systems, free volume and conductivity studies³³ have yielded information useful for refining the system toward optimal properties. PALS offers a useful tool for studying filled polymer systems for anti-corrosive protective coatings.

SUMMARY

The results are summarized as follows:

- (1) The long lifetime component (τ_3) in bisphenol A/glycol (B:G) diepoxide mixtures was independent of curing conditions;
- (2) Free volume cavities similar to those in the cured epoxies formed before 100% cure;
- (3) Linear relationships between the lifetime, intensity and free volume fraction versus B:G epoxide ratio have been observed for dry specimens;
- (4) Nonlinear relationships exist between the lifetime and free volume fraction versus the ratio of the two

Table 4—Free Volume Cavity Sizes and Intensities for Wet and Dry Specimens

Specimens	V _c (ų) Dry	Intensity I ₃ (%) Dry	V _c (ų) Wet	Intensity I ₃ (%) Wet
B (100%)	94.8	23.7	94.8	22.4
B & G (70/30%)	96.2	21.5	96.5	19.4
B & G (50/50%)	97.3	19.1	102.9	16.7
B & G (30/70%)	103.0	17.4	108.0	17.1
G (100%)		14.8	118.2	15

epoxies after water saturation and are likely related to polymer swelling;

- (5) An approximate linear correlation was found between the mass of water gained by the epoxies (direct measurement) and the PALS free volume fraction measurements;
- (6) A change in the free volume fraction, Δf (C) < 0.0, shows that the water molecules filled the free volume for ratios of bisphenol A epoxide above 50%. Conversely, below 50% bisphenol epoxide, Δf (C) > 0.0, the free volume expanded and water molecules possibly had a polar interaction with the glycol epoxide chains;
- (7) Inhibitors increase the size of free volume cavities in the 100% glycol epoxide G specimens, but not in mixtures containing bisphenol A epoxide B. Inhibitors silicate (calcium strontium phosphosilicate) and borate (barium metaborate monohydrate) have no major effect on bisphenol A epoxide B as a function of water uptake; and
- (8) The nitrophthalate (Zn salt) inhibitor or functionality similar (positron interactive) materials, may be useful as probe molecules (research tool) for reactive corrosion inhibitors in specific formulations.

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Interfacial Studies of Crosslinked Polyurethanes; Part I. Quantitative and Structural Aspects of Crosslinking Near Film-Air and Film-Substrate Interfaces in Solvent-Borne Polyurethanes

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INTRODUCTION

Ithough urethane-based polymers have been extensively studied, ^{1,4} there are various aspects of the urethane chemistry that are not fully understood. For example, kinetics and thermodynamics of the crosslinking reactions near surfaces and interfaces of urethane coatings have been rarely explored. This issue is particularly important because understanding of chemical processes near the interfaces may affect numerous film properties, including adhesion, friction coefficient, abrasion resistance, and others. On a similar note, quantitative analysis of the crosslinking reactions at various surface depths appears to have a strong effect on properties.⁵ Thus, understanding of the kinetics of urethane crosslinking reactions near the film-air (F-A) and film-substrate (F-S) interfaces is particularly important.

Urethane linkages are formed through the addition reaction of a polyisocyanate crosslinker and a hydroxylfunctional oligomer. The isocyanate reacts with the nucleophilic or active hydrogen of the oligomer to form the urethane linkage. *Scheme* 1 illustrates a generic example of this reaction, in which R or R' groups can range from aliphatic to aromatic species.

$$R-N=C=O + R'-OH \longrightarrow R-N' \xrightarrow{C} XR'$$
Scheme 1

Although this reaction illustrates only one functional group on each reactant, the presence of two or more reactive sites is required to form a urethane crosslinked network. For example, trimereization of isocyanates to isocyanurates in the presence of a strong base produces a crosslinker with three isocyanate functionalities.⁸ One of the commonly used species to form crosslinked networks are isocyanurates, such as 1,6-hexamethylene

One of the reactions leading to the formation of polyurethane (PU) crosslinked networks is the reaction of NCO and OH functionalities. In this study, we examined how crosslinking reactions of hexamethylene diisocyanate isocyanurate and polyacrylate near the film-air (F-A) and film-substrate (F-S) interfaces in urethane coatings may affect crosslink density as well as other network properties. While at the initial stages of the crosslinking reactions, solvent evaporation competes with the urethane network formation and isocyanate consumption changes at various depths from the F-A and F-S interfaces. Quantitative analysis of the NCO consumption as a function of depth showed that the NCO concentrations change from 2.35×10^{-5} to 2.09×10^{-5} M, while going from 0.27 to 1.14 µm. During reaction times not exceeding two to three hours, the NCO consumption at the F-A and F-S interfaces is consumed more rapidly. At low relative humidity conditions, excessive amounts of unreacted NCO exists at both the F-A and F-S interfaces. However, at the extended reaction times, NCO concentration levels at the F-S are greater than at the F-A interface, and the NCO concentration differences can be as high as 3×10^{-5} M. In this study we also examined how crosslinking reactions of hexamethylene diisocyanate (HDI) isocyanurate and polyacrylate near the F-S interfaces in urethane films may affect orientation and distribution of urethane functionalities.

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diisocyanate (HDI) with the structure shown in the following:

Most commonly employed multifunctional co-reactants are hydroxy-substituted acrylics and hydroxy-terminated polyesters.⁷

However, reactions leading to the formation of ure-thane crosslinked networks are also influenced by external conditions. For example, Scheme 2 illustrates an important linkage that forms when water is present during crosslinking reactions. The source of water is typically high relative humidity or water absorbed by the system. A condensation reaction between the isocyanate and water produces unstable carbamic acid. As a result $\rm CO_2$ and amine groups are produced. The latter will rapidly react with another isocyanate functionality to form urea linkages, such as that shown in Scheme 2.

In the presence of water, amines are more nucleophilic than alcohols, and the formation of urea linkages will result with relatively small amounts of the urethane linkages generated. This vigorous reaction between the amine and isocyanate causes a rapid increase of molecular weight and viscosity, thus reducing the lifetime of a mixture. The formation of CO_2 may also adversely affect reaction characteristics. It should be mentioned that the quantities of CO_2 diffusing out of the system are small, thus no film foaming occurs. As a matter of fact, recent studies showed that the rate of CO_2 production during the urethane network formation can be controlled by

crosslinking conditions, in particular relative humidity and temperature.² However, production of CO₂ inherently affects adhesion over the urethane coatings.² It should be also mentioned that because urethane and urea linkages still have an active hydrogen, they can further react with an isocyanate to form an allophonate and a biuret, respectively. The reactions leading to their formation are shown in *Scheme* 3. It should also be noted that these reactions are significantly slower than the reactions leading to the formation of urea and urethane linkages and not affect overall kinetics.

With this background in mind, let us focus on the kinetics of the isocyanate consumption occurring during the urethane formation near the F-A and F-S interfaces and attempt to correlate crosslinking reactions and other reactions that may compete with the urethane formation. A particular emphasis will be given to the quantitative analysis near the F-A and F-S interfaces. Because ATR FTIR spectroscopy has proven to be a useful tool in the analysis of surfaces and interfaces, 9-12 we will also take advantage of the recent developments 12 and utilize this approach in quantitative analysis of the F-A and F-S urethane interfaces.

EXPERIMENTAL

Sample Preparation

Hydroxy-substituted polyacrylate (Desmophen A450A) and an aliphatic polyisocyanate, 1,6-hexamethylene diisocyanate (Desmodur N3300) were supplied by Bayer Corp. Following manufacturer's recommendations, an NCO:OH equivalent ratio of 1.1:1.0 was maintained. The mixture was solubilized in a 50/50 mixture of xylene/nbutyl acetate in a 1:1 ratio. The polyacrylate and isocyanurate crosslinker were mixed for two minutes. The samples were drawn onto a 45° end $3 \times 20 \times 5$ mm parallelogram KRS-5 and Ge crystals to yield a wet film thickness of 200 µm \pm 25 µm. The films were allowed to crosslink on an ATR crystal at 25°C \pm 5°C under various

relative humidity (RH) conditions in an environmentally controlled hood. Unless otherwise stated, all spectra were recorded every hour for the first 72 hr and every 24 hr thereafter.

Analytical Methods

Transmission and attenuated total reflectance (ATR) FTIR spectra were collected on a Mattson Cygnus 100 equipped with a single beam spectrometer (Sirius 100). The instrument was continuously purged with dry air supplied from a Balston Type 75-60 air purification system. The spectra were collected at a 4 cm⁻¹ resolution using a 0.18 cm/s mirror speed. An attenuated total reflectance (ATR) variable-angle multiple reflection attachment (Spectra Tech, Inc.) with 45° end parallelogram KRS-5 and Ge crys-

$$R-N=C=O+H_2O \longrightarrow \begin{bmatrix} & & & & \\ & R-N & & \\ & & &$$

tals were utilized. In order to vary depth of penetration into the films, the ATR attachment was aligned at 40°, 45°, and 60° angles of incidence. These angles correspond to approximate depths of penetrations of 1.14, 0.92, and 0.65 µm with KRS-5 and 0.30, 0.29, and 0.27 μm with Ge. Concentrations of isocyanate and butyl acetate were determined by constructing calibration curves that allowed us to relate the specific band intensities at 2,273 cm⁻¹ and 1,243 cm⁻¹ due to NCO and butyl acetate concentrations. All spectra were collected using parallel transverse electric (TE) and perpendicular transverse magnetic (TM) polarizations using a Graseby Specac 12000 polarizer. Each spectrum represents 200 coadded scans collected and ratioed against a background of 200 co-added scans of an empty ATR cell equipped with a KRS-5 crystal. In an effort to minimize the effects of the dispersive nature of the ATR band intensities and the sample crystal coverage effects on band intensity, all spectra were corrected with Q-ATR software (obtained from Quan Specialties). Such corrected spectra were normalized to the CH2 stretching vibrations at 2,952 cm⁻¹.

Dynamic Mechanical Thermal Analysis Measurements (DMTA)

Dynamic mechanical measurements were conducted on a Rheometrics Mark III Analyzer at a frequency of 1 Hz and a force of 0.133N. Plastic mesh was used as a support for initially liquid specimens. Immediately after depositing the polyurethane (PU) specimens, the mesh was mounted horizontally in the instrument clamps. Log modulus (log E') and log tan delta (tan δ) of all samples were recorded isothermally at 28.5°C as a function of time.

Solvent Specific Evaporation Rate Measurements

Weight loss measurements were obtained by applying the coating to a KRS-5 crystal and weighing the sample at appropriate time intervals. Specific evaporation rate (SER) was calculated using the following equation:

$$SER = \frac{dW / dt(soln) - dW / dt(solv)}{dW / dt(solv)}$$

Where dW/dt(soln) is the slope from the weight loss curve of the urethane and dW/dt(solv) is the slope weight loss curve of pure solvent (a 50:50 mixture of xylene and n-butyl acetate).¹³

RESULTS AND DISCUSSION

As indicated in the Introduction Section as well as reported by others, ¹⁴ the formation of urethane linkages and isocyanate crosslinking reactions may have multiple character. Often, there are several parallel and competing reactions, and their kinetics may depend upon

$$R-N=C=O + R-N' \xrightarrow{C} OR' \longrightarrow R-N' \xrightarrow{C} OR' OR' O' \xrightarrow{C} N'H$$

$$R-N=C=O + R-N' \xrightarrow{C} N-R' \longrightarrow R-N' \xrightarrow{C} NH-R' O' \xrightarrow{C} N'H$$

$$R-N=C=O + R-N' \xrightarrow{C} N-R' \longrightarrow R-N' \xrightarrow{C} NH-R'$$

$$R-N=C=O + R-N' \xrightarrow{C} N-R' \longrightarrow R-N' \xrightarrow{C} NH-R'$$

$$R-N=C=O + R-N'$$

$$R-N=C=O + R-N' \xrightarrow{C} NH-R'$$

$$R-N=C=O + R-N'$$

numerous parameters. For that reason, in our attempt to understand kinetics and crosslinking reactions near the F-A and F-S interfaces, we will first assign infrared bands to vibrational modes of the individual components. While Figure 1, traces A and B, show transmission FTIR spectra of the acrylic polyol and the HDI crosslinker, respectively, *Table* 1 lists their tentative band assignments. Of particular interest are the bands responsible for the crosslinking reactions. For example, the band at 1,731 cm⁻¹ in Figure 1, trace A, is due to non-H-bonded C=O stretching modes of acrylic ester functionalities, and the band at 1,150 cm⁻¹ due to the C–C–O stretching vibrations in acrylic polyols. The CH₂ stretching and bending deformation modes are detected at 2,952 cm⁻¹

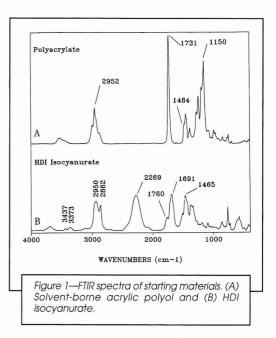


Table 1—Tentative IR Band Assignments for Acrylate Polyol, HDI Isocyanurate, Polyurethane, and Polyurea

Band	Wavenumber (cm ⁻¹)					
Assignment	Acrylic Polyol	Isocyanurate	Polyurethane	Polyurea		
H-bond. OH str	3543	_	3542	3542		
H-bond. OH str	3454	<u> </u>	_	_		
H-bond. N-H str	—	<u> </u>	3394	3394		
va CH3	2999	_	2989	2989		
v _a CH ₂		2939	2952	2952		
v _s CH ₂		2862	2873	2873		
NCO out-of-phase		2269	_	_		
free C=O stretch		1760	1733	1691		
H-bond C=O stretch	—	-	1720	-		
δ N-H & v _o C-N		1511	1525	1525		
5 CH ₂		1465	1463	1463		
S-C(ČH ₃)		1376	1384	1384		
SN-H & v. C-N	—	1368	1367	1367		
NCO in-phase		1338	—	_		
(O=)C-O-C str			1241	1241		
v _o C-N-C		1189	1189	1189		
(Ö=)C-O-C str	1192	_	1170	1170		
O=)C-O-C str		· —	1150	1150		
v C-N		1090	_	_		
n-butyl		1	1068	1068		
C-C skel. vib		1033	l -i			
C-C skel. vib.	990	_	990	990		
C-C skel. vib	967	_	967	967		
ν _s C-N-C		846	845	845		
C–N skel. str		768	767	767		
CH ₂ in-phase rock		730	753	753		

and 1,484 cm⁻¹, respectively. *Figure* 1, trace B, also illustrates a pronounced band at 2,273 cm⁻¹ due to the NCO asymmetric stretching modes along with the band at 1,691 cm⁻¹, the latter being attributed to the H-bonded C=O stretching modes in isocyanurate. ¹²⁻¹⁵ Amide II and Amide III bands are detected at 1,511 and 1,376

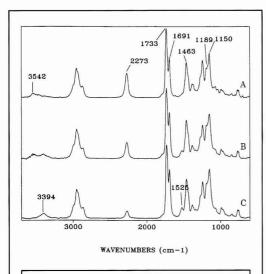
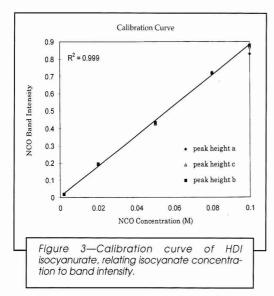


Figure 2—ATR FTIR spectra of solvent-borne polyurethanes recorded 0.92 µm from the F-S interface at 5-10% RH and 25°C for reaction times of: (A) 0 hr; (B) 35 hr; and (C) 144 hr.

cm⁻¹, respectively, and free and H-bonded NH stretching modes are detected at 3,437 and 3,373 cm⁻¹, respectively.¹⁵ The bands at 2,950, 2,862, and 1,465 cm⁻¹ are attributed to the CH₂ stretching and bending deformation modes, respectively.^{12,15,16} *Table* 1 lists IR bands and their tentative assignments for HDI isocyanurate and polyols, along with the bands due to the urethane and urea formation.

According to the reactions presented in the Introduction Section, the presence of water may play a significant role in the reactions leading to the urethane formation. For that reason we designed experiments in which urethane crosslinking was conducted under controlled humidity conditions. Figure 2, traces A, B, and C, represent ATR FTIR TE polarized spectra collected from the F-S interface crosslinked under 5-10% RH at 25°C, and recorded after 0, 35, 144 hr, respectively. A comparison of the spectra indicates that, as the crosslinking reactions progress, the band at 2,273 cm⁻¹, due to NCO, is reduced. On the other hand, the Amide II and the (O=)C-O bending modes, at 1,525 and 1,150 cm⁻¹, respectively, and the N-H stretching mode at 3,394 cm⁻¹, all due to the urethane linkages, increase. Similar trends are exhibited by the 1,189 cm⁻¹ band due to the C-N-C asymmetric stretching modes. In contrast, the 3,542 cm⁻¹ due to the C-OH stretching bands of polyacrylate and the 1,243 cm⁻¹ band due to (O=)C-O-C asymmetric stretching vibrations of the ester linkages of the solvent decrease. 17-19

Following the band assignments provided in *Table* 1, the 2,273 cm⁻¹ band can serve as a measure of isocyanate consumption during crosslinking reactions. However,

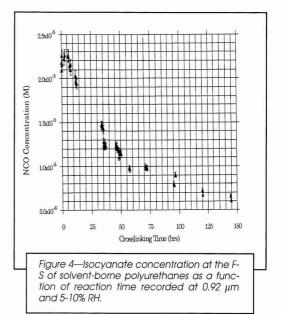


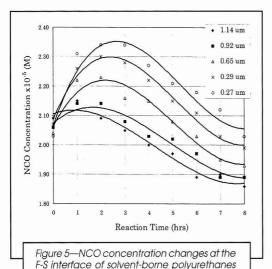
one of the drawbacks with quantitative IR spectroscopy is the necessity of generating a calibration curve. Typically, such a curve is generated by measuring the band intensity as a function of known concentrations. Once such a curve is created, one can use it to determine an unknown concentration. Although for transmission measurements such calibration curves represent a simple task, the situation becomes more complex when one wants to quantify ATR data. This is primarily because the spectra need to be corrected for optical effects, and previous studies identified numerous reasons necessitating such corrections. 12 Furthermore, determination of an absorption coefficient is essential, which for the 2,273 cm⁻¹ band, can be obtained by measuring the NCO intensity of isocyanurate mixture based on 50:50 butyl acetate:xylene as a function of known concentration. The choice of this mixture is justified by the fact that it approximates the environment of the isocyanate near the actual crosslinking conditions. Such a calibration curve is shown in *Figure 3*. According to the Beer-Lambert's law, the slope of the calibration curve is the product of the absolute molar absorptivity and the transmission cell path length. The second requirement, a correction of the ATR spectra, has been established in the previous studies.¹¹ This allows corrections of the ATR band intensities, so that they are equivalent to the product of the molar absorptivity and concentration of the NCO groups. This relationship and the absolute molar absorptivity determined from the calibration curve allow the quantification of unknown isocyanate concentrations at the F-S interface.

Now that the process for quantification of the NCO consumption is established, let us analyze crosslinking of PU exposed to 5-10% RH. *Figure* 4 shows a plot of the isocyanate concentration as a function of crosslinking time. As seen at the early stages of the reaction, there is approximately 2.09×10^{-5} M of isocyanate functionality of $0.92\,\mu m$ from the F-S interface. This value corresponds to approximately 0.002% of the original isocyanate con-

tent. Although it should be noted that the initial data point was taken after the surface of the film was dry to the touch (two hours), to our surprise, as the reaction progresses, the isocyanate concentration increases during the first one to two hours. A closeup of this time frame is shown in *Figure* 5 for the reaction conditions at 5-10% RH, and at 0.27, 0.29, 0.65, 0.92, and 1.14 μm depths from the F-S interface. After the first one to two hours, a continuous decrease is observed, and within 24 hr the NCO concentration at all depths becomes equal to $1.5 \times 10^{-5}\, M.$

While the diminishing intensity of the 2,273 cm⁻¹ band at the later stages results from the reactions shown in Scheme 1, its initial increase is somewhat surprising. In an effort to understand this behavior, dynamic mechanical thermal analysis (DMTA) was used to assess the changes of mechanical behavior as solvent evaporation and crosslinking reactions occur. For that reason, log E' and log E" moduli were measured isothermally as a function of time. Figure 6 illustrates the log E' and tan δ (tan $\delta = \log E' / \log E''$) results. As shown, tan δ increases to a maximum between one and two hours of the reaction time, followed by a gradual decrease after approximately 24 hr and levels off. It appears that the log E' values increase at much slower rates than the corresponding tan δ values at extended reaction times. The rapid increase of the log E' values detected at initial stages is likely attributed to the decrease of log E" values, resulting from solvent evaporation. As evaporation rates become diffusion controlled, the rate of log E' increase will be slow, as the mobility of the crosslinking functionalities decreases at these stages of crosslinking. It should be noted that no other driving forces, such as elevated temperatures or a catalyst concentration, were utilized. Therefore, crosslinking reactions are facilitated by mobility of the crosslinking functionalities. During the first hours, the tan δ increase correlates well with the





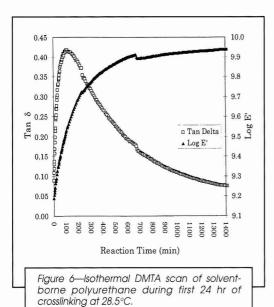
increase of the NCO concentration observed at the surface of the PU film in ATR FTIR spectroscopic analysis.

during first eight hours of reaction time at 5-

10% RH and depths of: (a) 1.14 μm; (b) 0.92

μm; (c) 0.65 μm; (d) 0.29 μm; and (e) 0.27 μm.

Although it appears that there is a relationship between the DMTA and spectroscopic data which may be related to solvent evaporation, in an effort to verify this hypothesis, weight loss experiments were conducted. The primary objective of these experiments was to determine the specific evaporation rates of the solvent system. Figure 7 compares the weight loss as a function of time for the pure solvent system (50:50 butyl acetate/xylene (BA/XY)) and the solvent system in the PU environment. It appears that the solvent evaporation rates

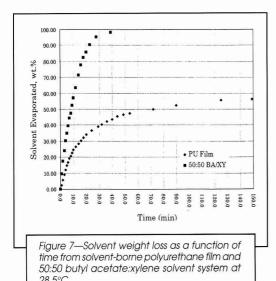


for the pure solvent mixture and the PU system vary only at the later evaporation stages. Although it is not surprising that the polymer film has an effect on the rate of evaporation of 50:50 BA/XY, a transition stage is detected between 55-75 wt% solids. The end point of this transition, at 75 wt%, solids corresponds to approximately 85 min of the reaction time.

The specific evaporation rate (SER) of 50:50 BA/XY plotted as a function of percent solids is shown in *Figure* 8. A comparison of the solvent evaporation data indicates a good correlation with the increase in the tan δ and log E' values as well as the isocyanate concentration changes. Furthermore, it is apparent from *Figure* 7 that approximately 40% of the solvent remains in the film after 150 min of reaction.

At this point it is appropriate to correlate solvent evaporation analysis and the tan δ changes with spectroscopic analysis. Therefore, let us examine the 1,243 cm⁻¹ band due to the (O=)C-O-C stretching vibrations of the solvent ester linkages. It appears that the decrease with the time is most pronounced during the first eight hours of reaction. Since a change in this band is not due to crosslinking reactions of the polymer system, this change is attributed to the evaporation of solvents. Using a calibration curve, these changes were converted to butyl acetate concentrations, which are plotted in Figure 9, indicating the sharp decrease during the first one to two hours. As we recall, these results agree with the DMTA (Figure 6) and SER (Figure 9) data, thus substantiating our hypothesis that solvent evaporation plays a significant role during the early stages of urethane film forma-

As mentioned in the Experimental Section, for this particular PU system, the film is dry to touch (two hours) before the first spectrum is collected. However, based on spectroscopic analysis of the subsequent spectra recorded at later stages, it became apparent that there is a residual solvent present below the F-A interface. As the crosslinking reactions proceed, and solvent molecules evaporate, the relative isocyanate concentration increases throughout the film. Because the F-S interface is hidden from the external conditions, the mobility of the species at this interface is significantly enhanced due to solvent plasticization effect. Therefore, the NCO containing reactant will diffuse to this interface, which is facilitated by slowly evaporating solvent molecules. After the NCO concentration reaches a maximum, the isocyanate concentration decreases as the reactions of isocyanates with the OH-terminated polyols and water dominate the crosslinking reactions. Thus, the initial increase of the isocyanate concentration results from two competing processes, relatively slower evaporation rates of solvent molecules and kinetics of isocyanate-polyol crosslinking reactions. As reactions progress further, initial reduction in the NCO concentration is rapid, followed by its leveling off. The initial decrease of the NCO results from the NCO and OH reactions, which become immobilized as diffusion becomes the rate determining step during relatively early stages of crosslinking. The decreased mobility of the NCO also suggests that these species have less chance of encountering an active hydrogen and becoming available for further reactions. These results agree



with the recent studies on isocyanate consumption rates in pigmented $\mbox{PUs.}^{1,2}$

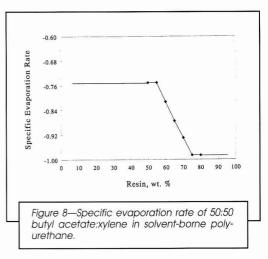
As mentioned in the Introduction Section, the presence of water vapor may have a significant effect on PU reactions. For that reason, Figure 10 was constructed and shows the consumption of NCO at the F-S interface in PU films crosslinked under four different relative humidities; 5-10, 35-40, 75-80, and 90-95%. As the RH increases, the NCO is consumed more rapidly, which again agrees quite well with the recent studies on isocyanate consumption rates in pigmented PUs.1 After one hour under 90-95% RH reaction conditions, the maximum isocvanate concentration, 2.60×10^{-5} M, is detected at the F-S interface. This correlates to 0.0024% of the original 1.10 M isocyanate concentration, indicating that the remaining 99.9976% of isocyanate resides in the bulk of the film or has already reacted. Subsequently, at relative humidities lower than 90-95%, the isocyanate concentration at the F-S interface is initially small, but increases as a result of the enhanced likelihood of reactions with water at 90-95% RH.

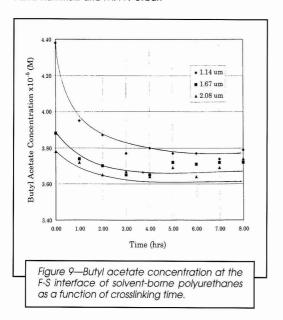
Figure 11 shows that the NCO concentration at the F-A interface decreases at extended reaction times and the NCO concentration is the lowest at the highest relative humidity. Although there are quantitative differences between the F-A and F-S interfaces results, similar trends are detected. A comparison of NCO concentrations as the F-A and F-S interfaces of the films crosslinked under the same relative humidity conditions indicates that after two hours, the NCO concentration is approximately the same at both interfaces. However, as the reaction progresses, the NCO concentration at the F-A interface is higher, and the difference between both interfaces becomes more pronounced at extended reaction times. Similar trends were observed in the previous studies,¹ and this behavior was attributed to solvent concentration gradients at both interfaces resulting from evaporation rate differences.

It is also appropriate to consider the volume changes resulting from solvent evaporation and crosslinking reactions. For that reason, let us consider stratification of the isocyanate concentration as a function of depth, which was also discussed in Figure 5. The isocyanate concentration is highest at relatively shallow depths of penetration and increases at higher depths. However, at these depths, the NCO concentration decreases with the reaction time, which is attributed to the volume decreases with reaction time as a result of solvent evaporation. The depths of penetration range from 0.14 to 0.57%, respectively, of the original 200 µm film volume. Within 24 hr, PU film crosslinked under reported RH conditions and reached its final film thickness of 80 µm, which is approximately 40% of the original volume of the film. At this film thickness, stratification of isocyanate concentration at various depths is not detected for depths of 0.34 to 1.40% of the final film volume.

Although there have been numerous studies 19-24 concerning orientation and mobility of molecular segments in organic films, it appears that external forces, along with the chemical make-up of a film, also play a significant role on arrangements of surface and interfacial structures. 6,25,26 It is well documented that for the latex films, temperature, pressure, and a partial vapor pressure will influence film formation. 20-24 However, other factors, such as surface tension, mechanical deformations, and a chemical make-up of a film may also play an essential role. 17-22 While all these factors are interdependent and influence each other, orientation of surface species has a significant effect on such macroscopic properties as durability or abrasion resistance, surface tension, and inter-layer adhesion. These issues are particularly important for urethane polymers because they contain functional groups capable of H-bonding, thus they are able to rearrange.²⁴⁻²⁷ As a matter of fact, hydrogen bonding has long been considered as one of the important intermolecular interactions in PUs.6,29

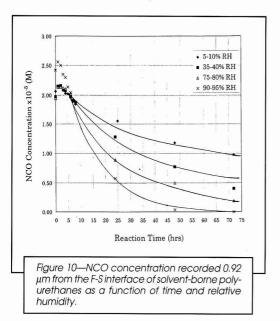
In this context, orientation of the functional groups resulting from interactions between urethane network components and environmental influences during the crosslinking reactions near surfaces and interfaces have

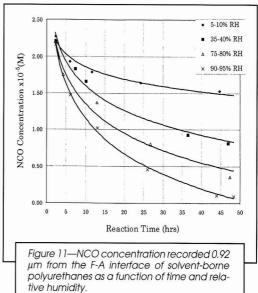




never been explored. Although quantitative analysis of the urethane crosslinking reactions exposed to controlled relative humidity conditions identified differences in the degree of crosslinking at the film-air (F-A) and film-substrate (F-S) interfaces, 1 studies that relate molecular structures near the interfaces and macroscopic properties are limited. As the studies presented in the earlier part showed, for the reaction times less than 24 hr, isocyanate concentration ranges between 2.35×10^{-5} to 2.09×10^{-5} as penetration depth into the F-S interface increases from 0.27 to 1.14 µm.

One of the features of ATR FTIR spectroscopy is that, by using polarized light, it is possible to determine ori-





entation of surface functional groups.³⁰ For this reason, as well as the requirements imposed by reflective and absorption index relationships,³¹ these studies will focus on polarization ATR FTIR experiments. Following the accepted convention,¹² the transverse electric (TE) wave is defined as having its electric vector parallel to the crystal plane (or perpendicular to the plane of incidence), whereas the transverse magnetic (TM) polarization has an electric vector perpendicular to the crystal plane. The spatial direction of the two polarizations thus allows us to distinguish between the dipole moment changes in the film plane and those oriented perpendicular to the plane.

With these considerations in mind, let us focus on the results of the polarization experiments of PU films prepared on a KRS-5 crystal with a surface tension of approximately 70 mN/m.35 These experiments were conducted as a function of penetration depth into the interface in the range of 0.5 to 2.0 µm. By superimposing the TE and TM spectra recorded from the same penetration depth, we will be able to examine orientation differences of the NCO and C=O functionalities. Figure 12 shows the 2,500-1,600 cm⁻¹ region of the TE and TM polarized spectra collected at the F-S interface. Within an experimental error, it appears that there are no intensity or band shape changes in either the NCO or C=O regions. However, if we examine the spectra recorded at 0.92 µm into the F-S interface, which are shown in Figure 13, there is a significant difference in the intensity of the bands in the C=O region recorded with TE (transverse electric) and TM (transverse magnetic) polarizations. Furthermore, the shape of the 1,733 cm⁻¹ band in the TM polarized spectrum becomes asymmetric. Examining the F-S interface at 1.14 µm reveals the same trends that were detected at 0.92 µm from the F-S interface, but the asymmetry of the band at 1,733 cm⁻¹ is enhanced. Figure 14 shows the TM polarized spectra recorded at 0.85, 1.21, and 1.51 µm depths. The intensity and band shape

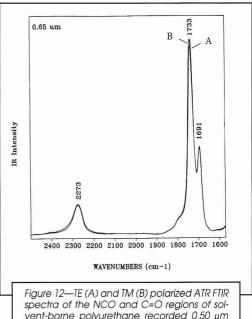
changes detected only in the TM polarized spectra indicate that the C=O groups have preferentially perpendicular orientation. At the same time, the absence of the intensity changes of the 2,273 cm⁻¹ band at either polarization indicates a random orientation of the NCO groups.

Let us further analyze the spectral changes detected at 1.21 µm from the F-S interface. Figure 15, traces A, B, C, and D, show ATR FTIR spectra recorded with TE and TM polarizations after 36 hr of crosslinking reactions. While traces A and B represent the TE and TM spectra, traces C and D show the TE and TM spectra after their spectral deconvolution using a maximum likelihood (ML) spectral analysis.¹⁹ The deconvoluted spectra were generated from the best estimation of a maximum probability approach using physical information about spectral features and statistical knowledge of the unknown number of overlapping bands. It appears that the 1,733 cm⁻¹ band recorded using TM polarization consists of two bands at 1,739 and 1,720 cm⁻¹. However, these bands are not observed in the TE polarization where the band at 1,733 cm⁻¹ due to the C=O stretching modes in polyol and the free C=O stretching in urethane are detected. These data indicate that at around 1.21 µm from the F-S interface, the C=O groups are preferentially perpendicular to the F-S interface.

At this point it is appropriate to focus on the assignment of the 1,691, 1,720, and 1,739 cm⁻¹ bands. The 1,720 cm⁻¹ band is attributed to the H-bonded C=O stretching mode resulting from the presence of PU linkages as well as one of the two carbonyl bands assigned to allophonates and biurets. The 1,690 cm⁻¹ band would be attributed then to the second of the two bands due to formation of an allophonate or a biuret. However, the 1,691 band can also also attributed to the C=O stretching modes in isocyanurate and due to free C=O stretching of a urea, or the disordered (non-crystalline) hydrogen bonded C=O stretching modes of urethane linkages. 14,32

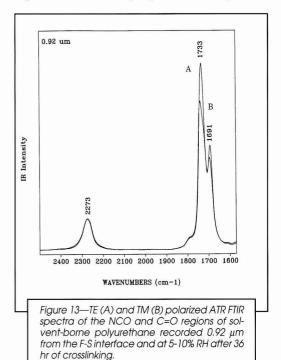
In determining the origin of the 1,691, 1,720, and 1,739 cm⁻¹ bands, it should be remembered that the kinetics of the reactions involved in the crosslinking should not be neglected. The reactions that lead to the formation of an allophonate or a biuret proceed at much slower rates because of the lower reactivity of the NCO functionalities, and slower diffusion, due to its bulky side groups. Therefore, the former are less likely to occur, and the assumption that the allophonate and biuret formation contributes very little, if any, to the bands at 1,691 and 1,720 cm⁻¹ seems reasonable. As a matter of fact, the films were reacted under low relative humidity conditions (5-10% RH), which further minimizes the formation of urea, thus practically eliminating the possibility of biuret formation. Keeping in mind that the 1,739, 1,720, and 1,691 cm-1 bands are detected at the initial stages of crosslinking, and the amount of water available for the formation of urea is minimal, the bands at 1,739 and 1,720 cm⁻¹ are primarily due to the formation of non-Hbonded and H-bonded urethane linkages, respectively, whereas the band at 1.691 cm⁻¹ results from the non Hbonded urea.

Having identified the origin of the carbonyl bands, let us go back to a main theme and focus on how orienta-



vent-borne polyurethane recorded 0.50 μm from the F-S interface and at 5-10% RH after 36 hr of crosslinking.

tion of the specific C=O species may vary at various depths from the F/S interface. Figure 15, trace D, shows that the H-bonded urethane C=O intensity at 1720 cm⁻¹ is enhanced in TM polarization, thus indicating that at a depth of 1.21 µm, C=O groups are oriented preferen-



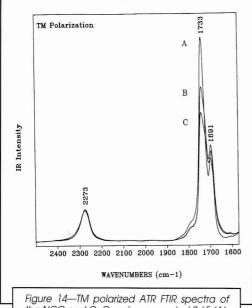


Figure 14—IM polarized AIR FIIR spectra of the NCO and C=O regions recorded 0.65 (A), 1.21 (B), and 1.51 (C) µm from the F-S interface and at 5-10% RH after 36 hr of crosslinking.

tially perpendicular to the film plane. Similar observations were made at 1.51 μm from the F-S interface. However, as was shown in Figure 12, at the shallower depths, around 0.85 μm , the H-bonded urethane C=O is not present, which is evidenced by the absence of the 1,720 cm $^{-1}$ band in both polarizations. In fact, the band at 1,733 cm $^{-1}$ shows no changes with polarization, thus indicating no preferential orientation at approximately 0.85 μm from the F-S interface.

Now let us focus on the shallower depths of penetration near the F-S interface. In this case due to close proximity of the substrate, it is important to consider the driving forces generated by surfaces and the resulting surface tension differences between the substrate and the urethane film. KRS-5 crystal, which was used as a substrate, has a critical surface tension of approximately 70 mN/m. In an effort to impose high surface energy on urethane films, ATR FTIR spectra were recorded of the specimens applied to a Ge substrate, which has a surface tension of approximately 706.8 mN/m.33 In addition to a higher surface tension, Ge has a higher refractive index, making it possible to penetrate shallower depths into the interface. Figure 16, traces A and B, show ATR FTIR spectra recorded from 0.39 μm into the F-S interface using TM polarization. While trace A is the original spectrum, trace B is its deconvoluted counterpart. The three bands at 1,739, 1,720, and 1,691 cm⁻¹ trace B of *Figure* 16 are attributed to the C=O stretching vibrations, with the 1,720 cm⁻¹ band being attributed to the Hbonded C=O groups. The same bands were detected in spectra obtained of the Ge/PU F-S interface recorded at 0.37 µm (not shown). As we recall, the band due to Hbonded C=O groups was not detected at depths above

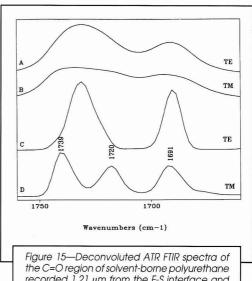
 $1.21~\mu m$ from the KRS-5/PU F-S interface. Therefore, as one would anticipate, effects of the substrate surface tension on the urethane network formation occur at depths closest to the F-S interface, which indicates that the surface tension of a substrate has a significant effect on orientation of the C=O groups. Surface tension was identified earlier as the driving force for orientation and migration effects in the recent studies of latex films. $^{9\cdot11}$

As we recall surface tension is a surface energy between a condensed phase (either a solid or a liquid) and a vapor, while interfacial surface tension refers to a surface energy difference between two condensed phases. 34,35 In view of the experiments conducted in this study, critical surface tensions of KRS-5 and Ge will significantly affect interfacial surface tensions when a urethane film is deposited on these crystals. The surface tension of urethane polymer is approximately 36 mN/m. 34 Using the relationship for interfacial surface tension:

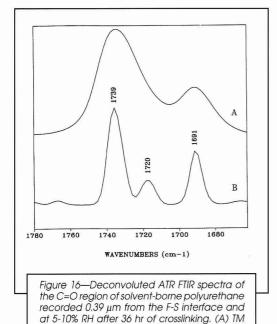
$$\gamma_{int} = \gamma_{sub} + \gamma_{film} - 2(\gamma_{sub}^{d}\gamma_{film}^{d})^{1/2}$$
 (1)

one can determine γ_{int} values for Ge/PU and KRS-5/PU systems (where: γ_{int} is the interfacial surface tension, γ_{sub} and γ_{film} are the critical surface tensions of the substrate and the polymer, respectively, and γ_{sub}^d and γ_{film}^d are the dispersion components of the substrate and the polymer, respectively). Using equation (1), estimated interfacial tensions between a PU film and KRS-5 and Ge substrates are 0.02 mN/m and 541.04 mN/m, respectively.

In order for each system to reach its lowest energy state, KRS-5/PU and Ge/PU interfaces will attempt to minimize the interfacial surface tension, and a reduction of the interfacial surface tension can be achieved through molecular rearrangements and migration of specific



the C=O region of solvent-borne polyurethane recorded 1.21 µm from the F-S interface and at 5-10% RH after 36 hr of crosslinking. (A) TE Pol.–original; (B) TM Pol.–original; (C) TE Pol.–resolved; and (D) TM Pol.–resolved.



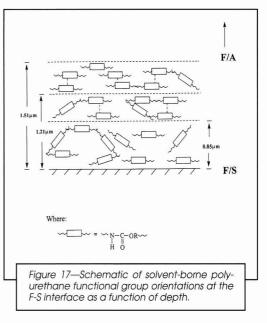
functionalities to the interface. Comparing interfacial energy values for KRS-5/PU and Ge/PU, it appears that the interfacial surface tension for KRS-5/PU is significantly smaller. Therefore, the driving force for molecular rearrangements at the KRS-5/PU interface is lower. ATR FTIR spectra recorded at 0.85 µm shown in Figure 12 indeed indicated that the orientation of the urethanes carbonyl groups at the F-S interface is random. It should be noted that, although not shown, random orientation of the C=O groups was detected when crosslinking times did not exceed 30 min. This observation indicates that at this early stage, solvent molecules plasticize (known fact) the system and its glass transition (Tg) is low enough to provide a significant amount of segmental motion. Thus, the C=O groups are random. In contrast, the interfacial surface tension of the Ge/PU system is above 540 mN/m, thus providing a significant driving force for inducing preferred orientation and migration of the Hbonded C=O of the urethane linkages to the F-S inter-

Pol.-original and (B) TM Pol.-resolved.

At this point it is appropriate to consider the effect of interfacial surface tension for the KRS-5/PU and Ge/PU systems as a function of depth into the F-S interface. Figure 17 summarizes the spectrally detected changes in H-bonding character and orientation of the C=O of ure-thanes cast on KRS-5 as a function of penetration depth. Based on the spectral changes discussed in Figures 12-16, it is possible to distinguish three zones of orientation of PU linkages. It appears that between 0.85 and 1.21 μm there is a transition zone in the intermolecular forces experienced by the bulk and interfacial regions. Furthermore, as shown in Figure 14, increasing asymmetry of the C=O region in the spectra recorded from 1.21 to 1.51 μm , demonstrates that the intermolecular forces acting

on urethane films become enhanced at greater depths. As the effective depth increases, the surface induced effects decrease into the bulk, and the tendency for the urethane network to form intermolecular H-bonding becomes predominant. This is manifested by the detection of the H-bonded C=O functionalities at 1.21 and 1.51 μm from the F-S interface in the KRS-5/PU system. In addition, the C=O groups are preferentially oriented perpendicular to the substrate, thus allowing more efficient H-bonding. The point at which H-bonding dominates may define the length of interfacial forces, and thus the depth of the urethane interface. In contrast, molecular orientations do not change as a function of depth for the Ge/PU system. Although penetration depths are in the range of 0.35 and 0.39 μm when Ge is used as an ATR element, it is anticipated that the intermolecular interactions in the bulk will be similar to those detected for the KRS-5/PU system. Therefore, in the Ge/PU system, the urethane C=O would be H-bonded and oriented perpendicular to the F-S interface, and a transitional region, as shown in Figure 17 between 0.85 and 1.21 µm depths, would not be observed.

Let us now consider preferential orientation of H-bonded C=O groups. There are three possible orientations that the urethane functionalities can assume: in-plane, out-of-plane (end-on), and random. Because the 1,720 cm⁻¹ band is detected only in the spectra collected with the TM polarization (*Figures* 15 and 16, trace D and B, respectively), the urethane H-bonded C=O is preferentially oriented out-of-plane. This effect may result from the fact that the urethane segments at a F-S interface will orient themselves in such a way as to accommodate the lowest energy state.³⁶ Therefore, preferential out-of-plane orientation of the C=O groups results from close packing, which increases H-bonding by lying out-of-plane, in the direction perpendicular to the film plane. This is schematically represented in *Figure* 17. The non-H-



bonded C=O groups of urethanes as well as the urea appear to be randomly oriented.

In view of the orientation of the C=O groups as a function of depth, it is necessary to identify processes that compete with the isocyanate crosslinking reactions. There are three processes that occur concurrently: solvent evaporation, reorientation of molecular segments, and isocyanate groups reacting with OH functionalities of the polyacrylate. While at the early stages of crosslinking, solvent evaporation competes with the isocyanate crosslinking reactions, reorientation of urethane linkages and H-bonding of the C=O urethane groups at the F-S interface may have an effect on the isocyanate crosslinking reactions at various depths from the F-S interface. The fact that the NCO groups are randomly oriented at the F-S interface indicates that the reaction kinetics are not affected by the formation of H-bonded C=O groups. The latter appear to form at the later stages, when solvent molecules are unable to provide a low glass transition environment allowing a significant amount of mobility of the system.

CONCLUSIONS

Isocyanate consumption has been investigated as a function of depth from the F-S and F-A interfaces as well as relative humidity. During initial stages, the isocyanate concentration increases throughout the film due to the solvent evaporation, followed by a rapid decrease. After approximately 24 hr, the isocyanate consumption levels off, as the diffusion effects become the limiting factor. The isocyanate concentration is highest at 0.27 µm from the F-S interface. As the reaction progresses and becomes diffusion controlled, the isocyanate concentration diminishes at significantly slower rates and levels off at all depths. The initial increase of isocyanate is attributed to solvent evaporation rates and corresponds well with the DMTA data. Relative humidity causes an increase of the isocyanate consumption as a result of the increased likelihood of reactions with water. Isocyanate functionalities are consumed more rapidly at the F-S interface than at the F-A interface.

The orientation of the isocyanate functionalities is random at all depths. In contrast, urethane H-bonded C=O groups exhibit preferential orientation perpendicular to the F-S interface at depths of 1.21 and 1.51 μm . At shallower depths, below 0.85 μm from the F-S interface, H-bonded urethane C=O groups are not present. When Ge is used as a substrate, the H-bonded C=O of the urethane is present as low as 0.35 and 0.39 μm from the F-S interface. These studies show that high interfacial tensions may induce molecular migration and rearrangement at the F-S urethane interfaces to alleviate excess energy at the surface.

ACKNOWLEDGMENTS

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



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

Environmental Protection Agency July 15, 1997—FR 62 37720 OMB Approval Number Under the Paperwork Reduction Act; National Emission Standards for Hazardous Air Pollutant Emissions; Group I Polymers and Resins; National Emission Standards for Hazardous Air Pollutant Emissions: Group IV Polymers and Resins

Action: Final rule, correcting amendments

This action corrects errors and clarifies regulatory text in two final rules establishing national emission standards for hazardous air pollutants for Group I polymers and resins (originally published September 5, 1996—61 FR 46906) and for Group IV polymers and resins (September 12, 1996—61 FR 48208). The correcting amendments are effective July 15, 1997.

For additional information, contact Robert Rosensteel, Environmental Protection Agency, (919) 541-5608.

Environmental Protection Agency July 18, 1997—62 FR 38652 National Ambient Air Quality Standards for Ozone Action: Final rule

The Environmental Protection Agency (EPA) has issued its final rule revising the national ambient air quality standards (NAAQS) for ozone. The rule is being promulgated in conjunction with a new NAAQS for particulate matter and goes into effect September 16, 1997.

The amendments lower the current concentration limit for ozone from 0.12 parts per million to 0.08, measured over the course of an eight-hour time period. In addition, the averaging time for this limit, as well as the method the agency uses to determine

compliance, has been revised. In addition, under the final rule on ozone, an area will be allowed to exceed the standard four times, rather than the proposal's three-time limit, before it is considered to be out of compliance.

Copies of the final rule can be downloaded from the EPA's Technology Transfer Network bulletin board under the "Recently Signed Rules" section at (919) 541-5742. Electronic copies of the final regulation and additional fact sheets can be obtained over the Internet at http://www.epa.gov/airlinks/.

For additional information, contact David McKee, EPA, (919) 541-5288.

Environmental Protection Agency July 18, 1997—62 FR 38652 National Ambient Air Quality Standards for Particulate Matter Action: Final rule

In this final regulation, the EPA has adopted a national ambient air quality standard (NAAQS) for particulate matter. This rule is being issued in conjunction with a NAAQS for ozone; the requirements will go into effect Sept. 16, 1997.

Under the new standards, an annual 2.5-micron particulate matter level is established. In addition, EPA's original proposal would have established a 24-hour standard of 50 micrograms per cubic meter; the final rule will set a standard of 65.

Copies of the final rule can be downloaded from the EPA's Technology Transfer Network bulletin board under the "Recently Signed Rules" section at (919) 541-5742. Electronic copies of the final regulation and additional fact sheets can be obtained over the Internet at http://www.epa.gov/airlinks/.

For more information, contact John Haines, EPA, (919) 541-5533.

Department of Transportation Research and Special Programs Administration

July 22, 1997—62 FR 39398 Improvements to Hazardous Materials Identification Systems; Corrections and Responses to Petitions for Consideration

Action: Final rule; editorial revisions and response to petitions for reconsideration

In this notice the Research and Special Programs Administration (RSPA) is making changes to a final rule (published on January 8, 1997) in which the Hazardous Materials Regulations were amended to better identify and communicate the hazards associated with hazardous materials in transportation in commerce. This rule is intended to assist emergency response personnel in responding to and mitigating the effects of incidents involving the transportation of hazardous materials, and to improve safety to transportation workers and the

Among other things, this notice corrects certain editorial errors and responds to petitions for reconsideration of the final rule. In addition, the effective date of the final rule has been postponed until October 1, 1998; the date for compliance with a requirement for new labels on packaging containing materials poisonous by inhalation has also been extended until October 1, 1999.

For more information, contact Helen Engrum or Paul Polydores, RSPA, (202) 3668553. EPA Sponsors Pollution Prevention Conference—EPA Region 4 is sponsoring a Pollution Prevention/Green Manufacturing Conference for industry located in the southeastern United States. The conference is scheduled for November 17-19 at the Marriott Marquis Hotel in Atlanta, GA.

Highlights of the conference include presentations on reducing and eliminating hazardous waste generation, solid waste, wastewater discharges and air emissions, focusing on the potential benefits of such reductions. The conference is free, but advance registration is required.

For conference information, call EPA, Region 4, at (404) 562-9362.

Industry Initiative Seeks Mutual Recognition of New Substance Notification Requirements under the **Canadian Environmental Protection** Act (CEPA) and the U.S. Toxic Substances Control Act (TSCA)-Chemical industry representatives have been concerned for some time that Canada and the U.S. have no mutual recognition program for new chemical substances manufactured in each country. Development of such a program is widely viewed as having the potential to improve trade, as envisioned under the North American Free Trade Agreement as well as foster consistent environmental impact and control information.

A new Industry Coordinating Group is seeking to develop support for a mutual recognition program. A recent briefing given (by representatives of this group) to the Council of Ministers of the North American Commission on **Environmental Cooperation** identified key considerations, including the fact that additional testing and assessment requirements in Canada, under CEPA regulations, often lead to more animal testing. The industry advocated approach would allow one jurisdiction to offer manufacturing approval for new substances, accepting the data developed (for the approval) as reasonable and representative, and provide a mechanism for the second jurisdiction to offer its conditional approval, and vice versa. Such an approach is deemed preferable to lengthy and unproductive approaches that would

seek to "harmonize" different regulatory systems. Numerous industry case studies have demonstrated the burdens associated with the current system.

In general, implementation of the mutual recognition effort would go a long way towards removing regulatory, non-tariff barriers to trade, while at the same time maintaining public confidence in chemical safety and protection of the environment.

The National Paint and Coatings Association has voiced its support for the initiative in a letter to EPA Administrator Carol Browner. Over time, a successful effort to breach gaps in reciprocity between the U.S. and Canada may lead to additional international recognition of chemical control regulations.

Congress has been in recess since August 1 and will remain so until Sept. 2, 1997. The following are various issues of interest:

Canada's TDG Clear Language Amendments—The Transportation of Dangerous Goods (TDG) Directorate is in the process of rewriting, in clearer language, the entire Transportation of Dangerous Goods Regulations (TDG Regulations). As part of this process, the directorate has made available draft #4 of this document for review and comment. The directorate has also made the draft document as well as submitted comments available on the Internet. This web site operates like a public meeting where everything submitted is available for viewing as well as the ability to submit comments over the Internet.

Some of the changes of concern include: limits for "not-regulated" and "partially-regulated" materials; and the handling of combustible liquids with flash points greater than 100 °F.

The comment deadline for draft #4 was Sept. 15, 1997. For additional information, contact Ray Clark (613) 998-0509; fax: (613) 990-2917. The web site address is www.tc.gc.ca.

Fifth Circuit Vacates OSHA Standards on Roof Coatings, Sealants in Construction—On July 24, the U.S. Court of Appeals for the Fifth Circuit vacated the Occupational Safety and Health Administration's (OSHA) construction and shipyard standards on asbestos containing asphalt roof coatings and sealants. (Asbestos Information Association/North America v. Secretary of Labor, CA 5, No. 94-41907) The court determined the standards were not supported by substantial evidence.

According to Circuit Judge Robert M. Parker, "there is no evidence in the record that asbestos fibers can ever escape from roofing sealants and become airborne; in fact, the evidence in the record indicates that they cannot."

The decision is the last of the litigation over the asbestos rules, which were first issued in 1986. This particular suit was a result of the asbestos association's challenge to a final OSHA rule published in 1994.

Library of Congress has Comprehensive On-line Resources—The Library of Congress provides several resources that can be accessed through its World Wide Web home page, including:

- Images and text from many of the library's major exhibitions.
- THOMAS, an on-line resource that provides congressional information, including the full text of bills, the *Congressional Record*, and the *Bill Digest*.
- Tens of thousands of items from the library's collections.
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The Library of Congress web site can be accessed at http://www.loc.gov.

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Information from the U.S. House of Representatives (legislative calendar, proposed and passed bills, current summaries and full text bills, as well as those in the archives) can be found at http://www.house.gov. Information from the U.S. Senate can be found at http://www.senate.gov.

States Proposed Legislation and Regulations

California

Recycling-CA A. 705 (Strom-Martin) reenacts, until January 1, 2001, provisions of existing law requiring all state agencies to purchase re-refined automotive lubricants, recycled antifreeze, recycled solvents, and recycled paints, except that the bill would require the purchase of the listed materials only when they are available, of a fitness and quality equal to their nonrecycled counterparts, and not more costly than the lowest price quoted by suppliers. The bill passed the Assembly in June and in July was referred to the Senate Committee on Appro-

Controlled Substances—CA. A. 1173 (Olberg) requires manufacturers, wholesalers, retailers who purchase for sale or who sell to any person chemical reagents and/or solvents, where the value exceeds \$100, to follow certain procedures including retaining bills of sale (for three years) for inspection on demand by the Attorney General's Office. The bill has been approved by both houses and awaits the Governor's signature.

Florida

Air Quality (Regulation)—The Florida Department of Environmental Protection (DEP) has adopted a final rule (23 FLAR 3252; 6/27/97) which broadens the eligibility requirements for Title V general permits for area sources and reduces the permit reporting criteria. The regulation was effective July 7. Contact Beth Hardin, DEP, (904) 488-0114.

Illinois

Graffiti—IL H. 661 (Scott and Feigenholtz) provides that a municipality may remove graffiti from private property but may not recover the cost from the owner. The bill was signed by the Governor on August 1, 1997. Public Act No. 90-292.

Community Right-to-Know (Proposed Regulation)—A proposed regulation (21 ILR 7789; 6/27/97) of the Illinois Emergency Management Agency (EMA) would amend emergency community right-to-know requirements,

including standards for reporting incidents, chemical inventory forms, and material safety data sheets. Contact Dean Schlee, Illinois EPA, (217) 782-4694.

Indiana

Air Quality (Regulation)—A final rule (20 INR 2761; 7/1/97) adopted by the Indiana Department of Environmental Management (DEM) incorporates by reference national emission standards for hazardous air pollutants for polymers and resins. The regulation became effective June 11. Contact Joe Salignoe, DEM, (317) 233-1179.

Automotive Refinishing (Notice)—The Indiana DEM has issued a guidance document (20 INR 2918; 7/1/97) concerning specific requirements for gun cleaners used in automotive refinishing operations. Contact DEM, (317) 232-8223.

Kentucky

Lead (Regulation)—The Kentucky Department for Health Services has adopted new regulations (902 KAR 47:080,:090, and :100) regarding procedures for performing lead-hazard activities. The rule adopts training and certification standard for people who perform lead-hazard detection and abatement, including application for certification procedures, renewal and recertification procedures, and related fees. The rule establishes procedures for conducting such activities in target housing and child-occupied facilities. For additional information, contact Mae Lewis, Cabinet for Health Services, (502) 564-7900.

Louisiana

Air Quality (Notice)—The Louisiana Department of Environmental Quality (DEQ) has published the state air quality report for 1996 (23 LAR 833; 6/20/97), including information on toxic air pollutant emissions and ozone standard attainment. Contact Joyce Coleman, DEQ, (504) 765-0902.

Lead—LA S. 616 (Hainkel) provides for licensing, certification, accreditation, and notification fees for lead abatement activities.

The governor signed the measure on July 15.

Massachusetts

Toxics-in-Packaging—MA H. 2901 (S. Angelo) reduces the presence of heavy metals in consumer packaging. On July 21, the bill was reported favorably from the Joint Committee on Natural Resources and Agriculture and sent to the Joint Committee on Science and Technology.

Michigan

Air Quality—MI S. 672 (A. Smith) reduces the clean air emission and facility fees. Introduced on July 9, the legislation was referred to the Senate Committee on Natural Resources and Environmental Affairs.

New Jersey

Lead (Regulation)—The Department of Community Affairs, Division of Codes and Standards has adopted regulations under a new Section NJAC 5:17-11.1 to revise lead hazard evaluation and abatement standards and incorporate by reference the Industrial Lead Paint Removal Handbook. The rule defines "super-structure" and "commercial building"; states that contractors wishing to be certified to perform abatement work in both buildings and steel structures need make only one application and pay only one fee; provides that lead abatement contractors wishing to add steel structures to the work that they are permitted to perform must supply a list of employees who have New Jersey Department of Health and Senior Services certification in this discipline, together with such other documentation as may be required. The rule also specifies that lead abatement from steel structures or commercial buildings is not subject to the issuance of a permit or a lead abatement clearance certification under the uniform construction code and exempts lead abatement work on steel structures or other superstructures in commercial buildings from permit and lead abatement clearance certificate requirements. The rule went into effect on July 21 and becomes operative on September 24, 1997. For additional

information, contact Michael Ticktin, DCA, Division of Codes and Standards, (609) 530-8803.

New York

Lead—NY A. 7099 (Eve) establishes a certification program to reduce the health risks of residential lead-based paint hazards; provides for the discontinuance of lead poisoning conditions; provides for training and education in lead-based paint activities; provides for certification of contractors, recordkeeping, information outreach, and monitoring children's blood levels; establishes maintenance standards for painted surfaces; provides for promulgation of rules and regulations. The bill passed the Senate earlier this year and on August 3 was sent to the Senate Committee on Rules.

NY A. 4735 (Clark) increases the penalty for the failure to comply with a notice and demand for the discontinuance of a paint condition conducive to lead poisoning. On July 22, the legislation was reported out of the Assembly Rules Committee.

Air Quality (Proposed Regulation)—A proposed rule issued by the New York Department of Environmental Conservation (DEC) would establish the 1997 operating permit program and fee procedures for air pollutant sources subject to federal Title V permit requirements. Contact Doris McCarthy, DEC, (518) 457-2823.

North Carolina

Lead (Proposed Regulation)—The North Carolina Department of Environment, Health and Natural Resources (DEHNR) plans to adopt regulations (12 NCR 31; 7/1/97) which would establish certification requirements for individuals involved in lead abatement work. This proposal is currently in effect as a temporary rule, effective July 7. Contact Pat Curran, DEHNR, (919) 733-0820.

Toluene Diisocyanate (Temporary Rule)—The North Carolina Department of Environment, Health, and Natural Resources, Environmental Management Commission has issued a temporary rule amending regulations that will establish an acceptable ambient air level for toluene diisocyanate, 2,4- and 2,6-isomers. The rule remains temporary while the commission collects and reviews comments. Comments are requested, and as of this date, there is no comment deadline. For additional information, contact Thomas Allen, DEHNR, Division of Air Quality, (919) 733-1489.

Ohio

Air Quality (Regulation)—A final regulation of the Ohio Environmental Protection Agency (EPA) eliminates certain reporting requirements that exceed the scope of federal standards relating to air quality regulations for ozone. The rule went into effect July 31. Contact Steve Feldmann, EPA, (614) 644-2115.

Pennsylvania

Hazardous Materials Transportation (Proposed Regulation)—A regulation (27 PAB 3231; 715197) proposed by the Pennsylvania Department of Transportation (DOT) would incorporate by reference amendments to the federal hazardous materials standards and would eliminate the requirements for hazmat transporters to register annually with the state. Contact Daniel Smyser, DOT, (717) 787-7445.

Rhode Island

Graffiti—RI S. 106 (Palazzo) Sets a reporting date for the study commission on graffiti proof aerosol spray paint containers. The bill was filed with the Secretary of State on June 27.

South Carolina

Hazardous Waste (Regulation)— The South Carolina Department of Health and Environmental Control (DHEC) adopted a final rule (21 SCSR 165; 6/27/97) which amends hazardous waste management regulations by (1) modifying recordkeeping and reporting requirements; (2) adopts 10-year permit renewal periods; (3) revising provisions for marking and labeling containers; and (4) reducing land disposal restriction notification for small quantity generators of hazardous waste. The rule went into effect June 27. Contact Suzanne Rhodes, DHEC, (803) 896-4174.

Tennessee

Air Quality (Regulation)—A final regulation of the Tennessee Department of Environment and Conservation (DEC) updates air quality requirements concerning leaks from synthetic organic chemical and resin manufacturing equipment; the manufacture of certain types of resins; and other operations that emit more than 100 tons per year of VOCs. The rule became effective July 30. Contact Malcolm Butler, DEC, (615) 532-0600.

Washington

Occupational Safety and Health (Proposed Regulation)—A proposed rule (97-13 WASR 69; 7/2/97) issued by the Washington Department of Labor and Industries (DLI) would adopt federal occupational safety and health standards concerning occupational exposure to methylene chloride. Contact Tracy Spencer, DLI, (360) 902-5530.

Wisconsin

Hazardous Waste (Proposed Regulation)—The Wisconsin Department of Natural Resources (DNR) proposed a regulation (498 WIAR 37; 6/30/97) which would establish fugitive organic air emissions limits for hazardous waste treatment, storage and disposal facilities and for certain hazardous waste generators who are accumulating waste on-site in tanks and containers. Contact Al Matano, DNR, (608) 267-3531.

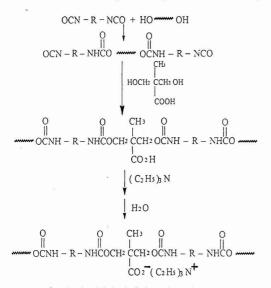
Lead (Proposed Regulation)—A proposed regulation (498 WIAR 34; 6/30/97) of the Wisconsin Department of Health and Family Services (DHFS) would adopt certification requirements for lead management workers; establish lead examination and certification fees; and would require two days notice of abatement activity before the work could start. Contact Gail Boushon, DHFS, (608) 266-5280.

Study of Anionic Polyurethane Ionomer Dispersant

Y.S. Huang, S.L. Ding, K.H. Yang, C.P. Chwang, and D.Y. Chaot—Chinese Culture University*

INTRODUCTION

he compatibility of powders^{1,2} dispersed in water is highly dependent on how close they are in po larity. Addition of a surfactant may enable the powder to be successfully dispersed as a result of changing the powder's polarity. In the last two decades, 3-6 a great deal of industrial and academic attention has been given to polymer-like surfactants. A number of investigators3-6 developed such surfactants with unique properties that have found application as dispersants, emulsifiers, and the like. In this study we describe the synthesis and evaluation of anionic polyurethanes for use as dispersants in water-based coatings. The goals of the investigation were to determine the effect of anionic polyurethanes on the environment and on the dispersion properties and the stability of titanium dioxide (TiO₂) in the water-based acrylic paint. The reaction scheme and possible structure of an anionic polyurethane ionomer for use as a dispersant is given in the following:



Synthesis of Anionic Polyurethane Ionomer

Anionic polyurethane ionomers designed for use as dispersants in water-soluble acrylic baking enamels were synthesized, and their structures were confirmed by infrared analysis. In an aqueous media, the surface tension of the polyurethane ionomers with different ionic or mixed ionic groups was found to increase with increased concentration of ionic groups, because of the orientation of hydrophobic groups at the air-water interface. It was also found that viscosity in water is higher for a polyurethane ionomer with L-2,5-diaminovaleric acid hydrochloride than for a polyurethane ionomer with dimethylol propionic acid.

Polyurethane ionomers with dimethylol propionic acid, p-amino benzoic acid, and mixed dimethylol propionic acid/p-amino benzoic acid were better dispersants for the wettability and dispersion of titanium dioxide (TiO_2) in the water-soluble acrylic enamels. The degree of dispersion of TiO_2 was found to improve with increased agitation time and anionic polyurethane ionomer concentration. In addition, polyurethanes with dimethylol propionic acid or p-amino benzoic acid or mixtures of these acids improved TiO_2 dispersion stability in the baking enamel.

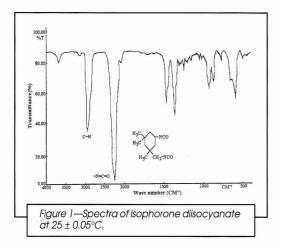
EXPERIMENTAL

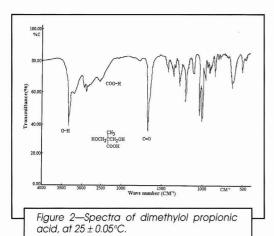
Materials

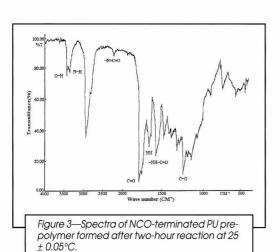
A polyester with a number-average molecular weight of 2,800 (hydroxyl number \sim 40.1) and a poly(ethylene glycol) with a number average molecular weight of \sim 6,000

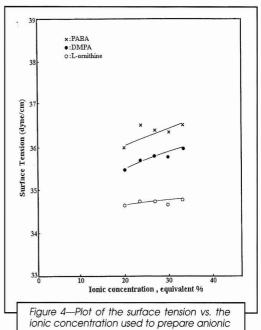
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Y.S. Huang et al.









PU ionomer, at 25±0.05°C.

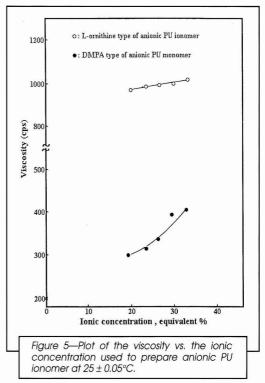


Table 1—Ingredients for Preparation of Anionic Polyurethane Ionomers

			Polyurethane Iono	mer	
Ingredients PUA	-1 PUA-2	PUA-3	PUA-4	PUA-5	PUA-6
Polyester, eq 0.02	26 0.026	0.026	0.026	0.026	0.026
Poly(ethylene glycol), eq 0.00	0.004	0.004	0.004	0.004	0.004
Dimethylol propionic acid, eq 0.0	o6 —	_	0.03	0.03	-
L-2,5-diaminovaleric acid					
hydrochloride, eq –	- 0.06	_	0.03		0.03
p-Aminobenzoic acid, eq—	_	0.06	_	0.03	0.03

were obtained from Tai Chin Chemical Industry Co., Ltd., and Sino-Japan Chemical Co., respectively. Isophorone diisocyanate (IPDI) was obtained from Merck Co. Dimethylol propionic acid and L-2,5-diaminovaleric acid hydrochloride (L-ornithine) were obtained from Alcolac Industrial Chemical and Sigma Chemical Co., respectively. N,N-dimethyl acetamide and p-aminobenzoic acid (PABA) were obtained from Jassen Co. and Ferak berlin Co., respectively. The water-reducible acrylic polymer (Eterac™ 1125-50) and TiO₂ (Ti-Pure® R-900) were obtained from Eternal Chemical Co., Ltd. and DuPont Co., respectively. Zinc sulfide-barium pigment (lithopone) was obtained from China Metallic Chemical Co., Ltd. Isopropyl alcohol (IPA) and 2-butoxy ethanol (BCS) were obtained from China Petroleum Oil Co. Melamine formaldehyde resin (Cymel® 325) was obtained from Cytec Co. All chemicals were used in an asreceived condition.

Method of Ionomer Preparation

The polyester (made by the reaction of adipic acid with 1,6 hexanediol and diethylene glycol; difunctional), polyethylene glycol, and dimethylol propionic acid or P-aminobenzoic acid or L-2,5-diaminovaleric acid chloride described in Table 1 were charged to a 1,000 mL, four-neck reaction kettle that contained 16 g of N,Ndimethyl acetamide and was equipped with a stirrer, thermometer, and condenser. The mixture was agitated at a speed of 400 rpm, and this agitation was maintained throughout the reaction time. Then, 0.108 equivalent of isophorone diisocyanate was slowly added. To the reaction mixture, 0.2% by weight dibutyltin dilaurate was added, and the temperature was increased to 105°C. The reaction was allowed to continue at this temperature for 2.5 hr to form the isocyanate-terminated prepolymer.

The prepolymer was then cooled and 60 g of acetone was added. To the solution, 0.03 equivalents of triethylamine were added to form the amine salt of the prepolymer. The amine neutralized prepolymer was further reacted with approximately 300 g of water in the presence of a small amount of diethylene triamine to form the final anionic polyurethane ionomer. The acetone was completely removed from the polymer by an evaporation technique. The final anionic polyurethane ionomer product had an approximately 38% solids content. When used as a dispersant in the paint formulations, the anionic ionomer was used at a one percent by weight level.

Paint Preparation

The water-soluble acrylic baking enamels were prepared from the ingredients shown in Table 2. The grind materials were placed in an aluminum can containing 2 mm glass beads and the can was sealed. The can was placed on a paint shaker (Red Devil Co., Model 5400) and agitated for 0.25, 0.50, 1.0, 1.5, and 2.0 hr to determine the degree of dispersion. Then the glass beads were removed from the grind mixture, and the Cymel 325 and water were added while agitating the formulation. Triethylamine was used to adjust the pH of the mixture and the quality of grind was determined⁷⁻¹⁰ with a fineness gauge (BYK Germany, Model 1512). The formulations containing the various anionic polyurethane ionomer dispersants were placed in aluminum cans and sealed. They were stored at room temperature, under 60% RH for 5, 10, 15, 20, 25, and 30 days. To study the stability of these samples, the change in degree of dispersion was determined as a function of time. The experimental error for these measurements was estimated to be within ± 0.5 unit.

RESULTS AND DISCUSSION

The infrared spectra for IPDI and DMPA are given in *Figures* 1 and 2. The isocyanate functionality of IPDI will rapidly react with DMPA or the other acid functional compounds, polyester, and poly(ethylene glycol) in the presence of the other additives to form the isocyanate-terminated polyurethane prepolymers. The formation of the prepolymer is illustrated by the formation of the

Table 2—Formulation of Water-Soluble Acrylic Baking Enamel

ngredients	Parts by Weight
Grind Ingredients	
terac™ 1125 - 50 acrylic polyme	r 19.9
iO,	
ithopone	
Anionic polyurethane ionomer dis	spersant 1.0
so-propyl alcohol	6.2
2-Butoxy ethanol	12.4
et-Down Ingredients	
Cymel® 325	38.0
Vater	
riethylamine	*

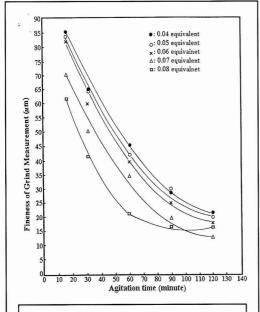


Figure 6—Plot of the fineness of ${\rm TiO_2}$ in the presence of anionic PU ionomer with 0.04-0.08 equivalent of DMPA as a dispersant in water-soluble paint vs. agitation time at 25 \pm 0.05°C.

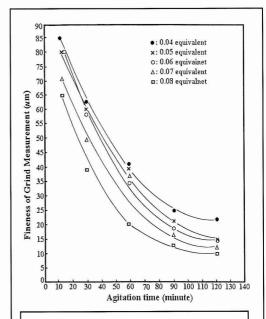


Figure 8—Plot of the fineness of ${\rm TiO_2}$ in the presence of anionic PU ionomer with 0.04-0.08 equivalent of PABA as a dispersant in water-soluble paint vs. agitation time, at 25 \pm 0.05°C.

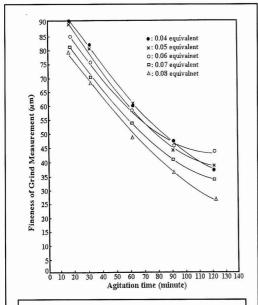


Figure 7—Plot of the fineness of ${\rm TiO_2}$ in the presence of anionic PU ionomer with 0.04-0.08 equivalent of ι -ornithine as a dispersant in water-soluble paint vs. agitation time, at 25 \pm 0.05°C.

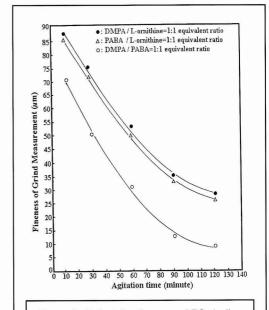
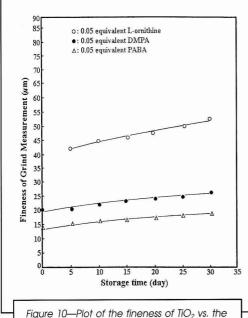
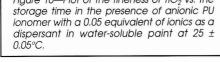


Figure 9—Plot of the fineness of TiO_2 in the presence of anionic PU ionomer with mixed ionics (1:1 equivalent ratio) as a dispersant, in water-soluble paint vs. agitation time, at $25\pm0.05^{\circ}\text{C}$.

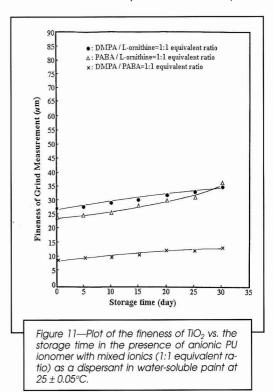




absorbance peaks at about 1,250 cm $^{-1}$ (C—O—), 1540 cm $^{-1}$ (NHCO), 1,620 cm $^{-1}$ (NH), 1,720 cm $^{-1}$ (C=O), 3,400 cm $^{-1}$ (NH), and by the substantially decreased absorbance peaks at approximately 2,275 cm $^{-1}$ (NCO) and 3,500 cm $^{-1}$ (OH) as given in the spectra of *Figure* 3 and in comparison to *Figures* 1 and 2. These results demonstrate that the reaction of the various compounds proceeds to form the anionic polyurethane ionomer. $^{11-15}$

As described in Figure 4, at room temperature the surface tension increases as the ionic concentration increases for the various carboxylic acids investigated. The electrostatic interaction between the charged ionomer molecules becomes stronger as the amount of ionic material attached to the backbone of the polyurethane molecules increases. This interaction will increase orientation of the hydrophobes at the air-water interface. As a result, the surface tension increases as the ionic content of the anionic polyurethane ionomer molecules increases. However, the surface tension is slightly greater for the PABA-containing molecules than for the DMPA- and Lornithine-containing anionic polyurethane molecules. This is due to the fact that the PABA-containing molecules have their hydrophobes more oriented at the airwater interface than do the other types of anionic molecules. The viscosity data presented in *Figure* 5 indicates that the one-ornithine-containing molecules have a significantly higher viscosity than the DMPA-containing molecules. This is believed to be due to the large phase volume formed with the L-ornithine hydrophobes.

The degree of dispersion for TiO_2 in the water soluble baking enamels when ionomers with 0.04 to 0.08 equiva-



lents of the various carboxylic acids are used as the dispersants is described in *Figures* 6-8. These data illustrated that the degree of dispersion rapidly decreases with increasing agitation time and with increasing ionic content. In general when the anionic polyurethane ionomers are used, the degree of titanium dioxide dispersion due to the collisions between glass beads and pigment particles becomes smaller as agitation time increases.

Increasing the amount of ionic material attached to the polyurethane backbone provides good wettability and improves dispersion of the pigment in the paint as indicated by the improvement in fineness of grind. It is readily apparent from the data in *Figures* 6-8 that the dispersion is poorer in the formulations containing ionomer based on L-ornithine than for those based on DMPA or PABA. This is related to the hydrogen bonding between L-ornithine and water molecules and resulting increase in phase volume of L-ornithine which causes poor wettability and dispersion of TiO₂ relative to the effect of the DMPA or PABA based ionomers.

The effect of an ionomer based on a 1:1 equivalent ratio of mixed ionic compounds on the degree of dispersion under the same experimental conditions is given in *Figure* 9. Here the data indicate that the degree of dispersion is markedly improved with increasing agitation time and with increasing amounts of ionomer. Although explanations similar to those mentioned earlier can be evoked for these systems, it seems that the confirmation of the anionic polyurethane molecules also plays an im-

portant role. Factual data to provide this latter supposition is not presently available.

The stability of the TiO₂ dispersions as a function of storage time for paints containing 0.05 equivalents of the various carboxylic acid compounds alone and in a 1:1 equivalent ratio mixtures is given in *Figures* 10 and 11. From the data in *Figure* 10, it is apparent that the degree of pigment dispersion decreases as a function of storage time. The change takes place because of hydrogen bonding forming between anionic polyurethane ionomer molecules and between ionomer molecules and water that contains other ingredients. There is less decrease in pigment dispersion when DMPA and PABA are used instead of L-ornithine, and therefore, these compounds will form most stable dispersions. The data in Figure 11 indicate that there is a decrease in dispersion as a function of storage time when the mixed acid-containing ionomers are used. Of these systems, the mixed DMPA/ PABA- based ionomers provide the most stable dispersions of pigment in the paint.

CONCLUSIONS

The reaction of IPDI with polyester, poly(ethylene glycol), and various carboxylic acids and other ingredients results in the formation of anionic polyurethane ionomers. Formation of the ionomer was established by infrared analysis. The surface tension of the ionomers in aqueous solution increases at the ionic concentration of ionomer increases due to hydrophobe alignment in the air-water interface. The viscosity of the L-ornithine-containing ionomers is markedly greater than that for the ionomers based on DMPA. The high viscosity may be due to the large phase volume formed with the L-ornithine ionomers. The degree of pigment dispersion in the water-soluble acrylic enamels increases as the agitation time increases and as the concentration of carboxylic acid compound used to prepare the polyurethane

ionomer increases. The data indicate that systems based on ionomers prepared from PMPA, PABA, and mixtures of these compounds have the best titanium dioxide dispersion and form the most stable formulated paints.

ACKNOWLEDGMENT

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Influence of Crosslink Density, Glass Transition Temperature and Addition of Pigment and Wax on the Scratch Resistance of an Epoxy Coating

Jakob Lange, Alexandre Luisier and Anders Hult[†]—Royal Institute of Technology*

INTRODUCTION

cratch resistance is a highly desired but little understood property of coatings. It depends on the intrinsic properties of the coating material in a way that still is largely unknown. Many workers have tried to relate mechanical properties, e.g., from tensile testing to scratch resistance. In most cases it has been possible to find relations valid for single materials, but much more difficult to identify any general relationships. Some studies have shown that the scratch resistance increases with increasing hardness and crosslink density, 1 whereas others have shown that less crosslinked, softer materials are better.^{2,3} Correlations between scratch resistance and toughness, i.e., fracture energy, have been found for certain systems such as urethanes.4 One reason for these opposing results and difficulties in establishing general correlations is the multitude of mechanisms by which scratching may occur. Depending on the type and rate of deformation imposed, scratches might occur through brittle or ductile failure, plastic flow, or fatigue.^{3,5} The response of a coating will thus be largely determined by the mechanism favored by the selected scratch test.

There is no generally accepted test method for quantitative analysis of scratch resistance, although several different test methods have been developed. ^{2,6,7} The tests use either loose or bonded abrasives, and the result is evaluated, in terms of weight loss or decrease in gloss. Tests using loose abrasives give a constant energy input irrespective of the sample characteristics, whereas tests with bonded abrasives have varying energy input, supplying more energy to softer materials. The Taber Abraser, which employs abrasives bonded to a pair of rubber wheels weighted against a spinning test panel, is widely used to evaluate scratch resistance, although it often is argued to be too severe. ² More specialized tests simulating scratching, e.g., under wet conditions in a car wash, have also been developed. ⁸

Little has been reported in the literature about the effects of pigment on scratch resistance. The influence of friction-reducing additives such as silicones and wax has been discussed to some extent. One study has shown

 \hat{T} he scratch resistance of a series of cationically cured epoxy coatings with varying crosslink densities has been investigated using five different scratch tests. The tests simulate a variety of scratch modes, ranging from single scratches to repeated surface wear. The relative influences of crosslink density and glass transition temperature (T_{\circ}) on scratch resistance, as well as the effects of adding pigment and wax to the coatings, were examined. The results show the scratch resistance to increase both with increasing crosslink density and increasing T_{α} in all tests. It is also shown that adding pigment or wax either increased or decreased the scratch resistance of the coating, depending on which type of scratching was performed.

that silicone increased the scratch resistance only when it was chemically bonded to the coating, e.g., through co-polymerization.⁸ Addition of wax has been shown to improve the scratch resistance in sliding scratch modes, but has no effect when scratches were caused by impact.⁹

In the present work, the scratch resistance of a series of cationically cured epoxy coatings with different crosslink densities is examined, and the relative influences of glass transition temperature (T_g) and crosslink density on scratch resistance are investigated. Several scratch tests, some previously presented and some newly developed, are used. The tests simulate a variety of different scratch modes, ranging from single scratches to repeated surface wear. Using the different tests, the effect on the scratch resistance of adding pigment and wax to the coating is also examined.

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caprolactone triol (2) monomers.

EXPERIMENTAL

The monomers used were a cycloaliphatic epoxy; UV-6110, 3,4-epoxycyclohexylmethyl-3,4-epoxycyclohexane carboxylate (1) with an epoxide equivalent weight of 350-450, and a polyol; TONE 0301, an ε -caprolactone triol (2) with a hydroxide equivalent weight of 98-103. Both monomers were obtained from Union Carbide and are presented in *Figure* 1. The surfactant, Silwet L-7604, was also obtained from Union Carbide. A thermal onium salt was used as a cationic initiator. 10 Five sample compositions, presented in *Table 1*, were prepared. To some mixtures, 40% by weight of pigment (titanium dioxide, Tipaque cr-50, <1 μm particle size, Ishimara Sangyo Kaisha Ltd.) or one percent by weight of polyethylene wax was added. The mixtures were stirred and applied to glass substrates as films with 100 µm thickness. The films were cured at 140°C for 30 min, followed by a postcure at 200°C for another 30 min for samples D and E. Free films for the mechanical testing were obtained by immersion in water followed by drying at 120°C for 30 min.

Dynamic mechanical analysis was performed using a Polymer Laboratories Mark II DMTA, at 1Hz and a scanning rate of 5°C/min. The strain was about 0.1%. Tensile tests were run in an Instron 5566 tensile tester at 0.1 mm/min on rectangular films of 10 mm width and a distance of 10 mm between the grips. Five different scratch tests were used. The tests are depicted in Figure

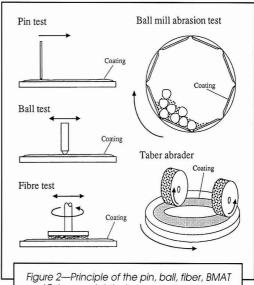
Table 1—Composition of the Different Coatings by Weight

Coating	Epoxy (%)	Polyol (%)	Surfactant (%)	Initiator (%)
Α	56.8	38.7	0.5	4.0
В	60.0	35.5	0.5	4.0
C	63.7	31.8	0.5	4.0
D	79.6	15.9	0.5	4.0
E	95.5	0	0.5	4.0

2. The test conditions, i.e., load, time, etc., were selected to produce scratches deep enough to be detected but not so severe as to penetrate the coatings entirely. The "pin" test consisted of sliding a steel pin (end diameter 2 mm) at 20 mm/min for 50 mm with a weight of 2 or 2.5 kg one time over the sample surface. To perform tests above room temperature, the samples were preheated on a hot-plate and then rapidly transferred to the test set-up. The temperature decreased at the most about five degrees as a result of the transfer, and then continued to decrease during the test. It was therefore not possible to perform tests at 180°C, the temperature required for sample E.

For the "ball" test, a carbide ball of 3 mm diameter was moved back and forth over the sample at an average speed of 15 mm/min, creating a groove of 7 mm length and 0.5 mm width. The weight on the carbide ball was 110 g and the test time 83 min. Tests at higher temperatures were performed with the scratching device in an oven. The "fiber" test used a circular disc (diameter 40 mm) of scouring cloth composed of nylon fibers (3M Scotch Brite), which was rotated at 60 rpm and moved back and forth over the sample at 15 mm/min, thereby creating a marred surface. The weight on the cloth was 80 g and the testing time was five minutes.

The ball mill abrasion test⁴ (BMAT) was performed by attaching the rectangular samples (20×50 mm) to the inner surface of a drum (diameter 200 mm), in which 50 g of sand (Askavia silversand 55, KEBO, Sweden) and 200 g of ceramic pellets (average diameter 18 mm) was placed. The drum was then rotated at a speed of 30 rpm for 30 min. A Taber abraser was also used (according to ASTM D 1044), with a load of 25 g and run for 200 cycles. The samples scratched in the pin and ball tests were evaluated using a Mitutoyo Surftest 301 profilometer, which permitted the depth of the scratch (peak-to-valley) to be measured. The samples subjected to the fiber,



and Taber scratch tests.

Table 2—Properties of the Different Coatings

Coating	Crosslink Density ^a (moles/m³)	T _g (°C) Neat/Pigmented	Youngs Modulus (MPa), Neat/Pigmented	Strength (MPa), Neat/Pigmented	Elongation at Break (%), Neat/Pigmented
Α	170	45/50	402/520	7/17	84/68
В	270	55/65	570/840	26/31	33/30
C	680	70/80	608/800	43/51	25/14
D	1300	150/155	669/829	69/78	15/9
E	2750	200/210	905/935	70/60	10/9

BMAT, and Taber tests were evaluated in terms of gloss reduction, i.e., relative change in gloss on scratching. The gloss was measured according to ISO 2813 at an angle of 60° .

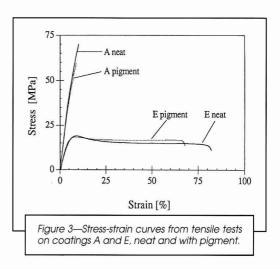
RESULTS AND DISCUSSION

The T_g , crosslink densities, and ultimate tensile properties of the different coatings are presented in *Table 2*. Typical stress-strain curves for the hardest and softest coatings are shown in *Figure 3*. The crosslink density in terms of moles of elastically effective network chains, $V_{\rm e}$, was estimated from the elastic modulus, E, using the following expression derived from the theory of rubber elasticity¹¹:

$$v_e = \frac{E}{3RT}, T \gg T_g$$
 (1)

Here R is the gas constant and T the temperature. Equation (1) is strictly only valid for very lightly crosslinked polymers, but is still helpful in providing a common scale for comparison. Table 2 and Figure 3 show that the coatings have quite different properties, ranging from brittle to highly ductile. As the crosslink density increases, the $T_{\rm g}$ goes up and the ductility decreases. It may be noted that a series of UV-cured coatings, prepared from the same epoxy and polyol, showed very similar tensile properties. However, the UV-cured coatings were slightly more brittle (showed higher strengths and lower elongations at break) than the coatings in the present work. 12 When 40% by weight of pigment is added to the coatings, the stiffness, strength, and Tos increase and the elongation at break decreases (see Table 2 and Figure 3). No measurable difference in the properties was observed when wax was added to the coatings.

Five different scratch tests (see *Figure* 2) were used to test the coatings. The pin and ball tests produce single scratches, and the damage induced is due to fracture and/or plastic deformation (pin test) or a mixture of wear and fatigue (ball test). The fiber, Taber and ball mill abrasion (BMAT) tests create marred surfaces, and the damage is largely due to wear. All the tests except the BMAT involve bonded abrasives, and thus give a variable energy input to the sample, the amount depending on the properties of the coating. On the other hand, BMAT uses loose abrasives and therefore gives the same energy input to all samples, irrespective of their properties.



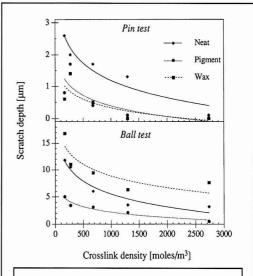


Figure 4—Scratch depths obtained with the pin and ball tests for coatings A-E with and without additives vs. crosslink density as calculated from equation (1). The lines are shown to indicate the trend of the experimental data points. The load in the pin test was 2 kg.

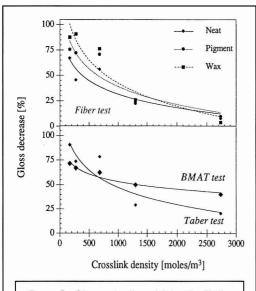


Figure 5—Gloss reduction obtained with the fiber, BMAT and Taber tests for coatings A-E versus crosslink density as calculated from equation (1). Fibre tests were done on coatings with and without additives, whereas BMAT and Taber tests were only performed on neat coatings.

Tests on Neat Coatings

The results of the five scratch tests on the different neat coatings (diamonds/solids lines) are presented in Figures 4 and 5. As can be seen, in all cases the scratch depth or gloss reduction decreases as the crosslink density is increased. In other words, the scratch resistance increases with increasing crosslink density. Many workers have found that hardness promotes scratch resistance, as long as all other material properties remain unchanged.1,9 This seems to agree with the present results, where the most crosslinked and hardest epoxy always is the best. In spite of some scatter in the data, all tests give remarkably similar results. It is important to remember that the test involves different types of deformation, ranging from single scratching under a high load to repeated wear under low loads. The rates of deformation (speed and load combined) vary considerably between the tests. Previous work, e.g., by Betz and Bartelt on acrylate/melamine coatings,8 has shown that the scratch resistance of a given material may vary greatly depending on which test it is subjected to. In particular the BMAT test is expected to give a different result than the other tests. A previous study where a series of epoxy coatings were tested with Taber and BMAT found that the two tests graded the materials differently.³ In spite of this, for the present coating series, all tests including BMAT give the same trend. The consistency of the tests may be broken if the load is changed. Results of pin tests at 2 kg (the same data as in Figure 4) and 2.5 kg are shown in Figure 6. As can be seen, when the load is increased to 2.5 kg, brittle fracture through the whole

coating thickness is observed in the most densely crosslinked sample. This shows that a harder, brittle material is better, but only up to a certain limit.

If only the data in Figures 4 and 5 are considered, there seems to be a simple relationship between coating properties and scratch resistance. It is worth mentioning that work on a series of polyester coatings with similar crosslink densities and tensile properties, using the same scratch tests, shows the scratch resistance to decrease with increasing crosslink density.¹³ Again all tests give the same results. Inspection of the scratches with light microscopy and SEM reveals that they occur mainly through plastic flow for these polyester coatings, whereas they appear to be the result of brittle fracture for the epoxies in the present work (data not shown). It thus seems as if the scratch tests induce quite different behaviors in the two coatings systems. The key to these discrepancies could lie in the rate-dependence of the mechanical properties. In tensile tests at higher rates (1 and 10 mm/min), all epoxy coatings behave in a brittle manner. As opposed to this, the polyesters exhibit the full range of ductile to brittle behavior also at higher rates of testing. 13 Further work is required to understand these differences in behavior and how they relate to the scratch resistance.

For the materials in the present study, it appears that the more crosslinked the coating is, the better the resistance towards scratches. It should be noted that when the crosslink density increases, there is a concomitant increase in $T_{\rm g}.$ The question is now which one of the factors has the strongest influence on the scratch resistance. In order to separate the effects of the two factors, tests have to be performed at different temperatures. To investigate the influence of $T_{\rm g},$ tests on one epoxy composition were performed at different temperatures. This simulates a series of materials with different $T_{\rm g}s$ but the same crosslink density. The results of tests at different temperatures are shown in Figure 7. As can be seen, the scratch resistance increases with decreasing temperature in both the pin and ball tests. In other words, a high

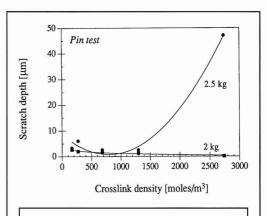
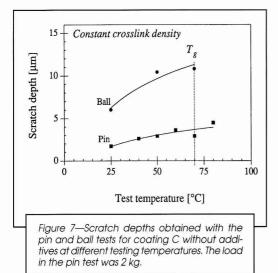


Figure 6—Scratch depths obtained with the pin test at different loads for coatings A-E without additives vs. crosslink density as calculated from equation (1).



T_o improves the scratch resistance. To determine the influence of the crosslink density itself, i.e., to simulate a series of materials with different crosslink densities but the same T_g, another technique has to be employed. To eliminate the effect of the increasing T_g in the series of coatings in the present study, tests were run at a constant temperature with respect to the T_g ; $T_{test} = T_g - 20$. This approximately results in testing the materials as if they were to have the same T_g. The results of such tests are depicted in Figure 8. It should be noted that it was not possible to reach 180°C, the temperature required for coating E, in the pin test. It appears that the influence of crosslink density is somewhat less pronounced than the influence of T_g. The ball test exhibits a clear trend towards increasing scratch resistance with increasing crosslink density, but no trend is visible in the pin test. In general, ultimate mechanical properties do not improve as the crosslink density is increased. Work by Nusselder and Bos on amine-cured epoxies has shown the fracture behavior to be largely unaffected by the crosslink density,14 whereas Fischer et al., also studying amine-cured epoxies, found that fatigue crack growth rates increased with increasing crosslink density.¹⁵ As regarding the influence of Tg, Espuche et al. have shown that the fracture toughness of epoxy-amine networks decreases markedly when the T_g increases. ¹⁶ It thus appears that scratch resistance, as measured in the present work, does not at all show the same dependence on T_g and crosslink density as other ultimate properties.

Influence of Pigment and Wax

In many applications, coatings contain substantial amounts of pigment. To the authors' knowledge, no study of the influence of pigment on the scratch resistance has been published. The results of scratch tests on pigmented epoxy coatings are shown in *Figures* 3 and 4. Comparing with the results on neat coatings, it may be seen that the curves for the pigmented coatings have the same general trend and shape as the curves for the neat

materials. The difference is that the curves are shifted with respect to each other. Thus the addition of pigment increases the scratch resistance in the pin and ball tests, but slightly reduces the resistance in the fiber test. The reason for this is believed to be as follows: In the pin and ball tests, it is the increase in hardness brought about by the pigment that gives the increased scratch resistance. All the material that is removed from the coating in the scratching process is transported away from the scratch and does not interfere with the test. In the fiber test, the material removed from the coating remains between the cloth and the substrate, and acts as an abrasive. Therefore, the increase in hardness on addition of pigment not only increases the scratch resistance of the coating but also its effectiveness as an abrasive once it has been removed from the surface. This is why a slight decrease in scratch resistance on addition of pigment is observed in the fiber test.

Regarding the effect of adding wax to a coating, previous work has shown that some beneficial effects on scratch resistance may be achieved.9 However, the benefits were limited to gliding rather than impacting modes of scratching, since the main effect of the wax was to reduce the friction coefficient. In addition to reducing friction, the wax will also reduce the hardness of the coating somewhat. The results of scratch tests on coatings with wax added are shown in Figures 3 and 4. As can be seen, the general shape and trend of the curves are similar to what was observed for neat and pigmented coatings. Furthermore, a comparison of the curves shows that the addition of wax increases the scratch resistance in the pin test, but decreases it in the ball and fiber tests. The decrease is small in the fiber test, but quite pronounced in the ball test. The results thus show that wax may be beneficial when a scratch is produced by a single draw, but not when it occurs as the result of repeated

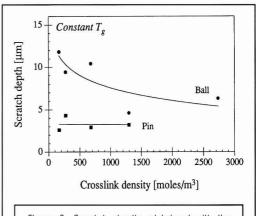


Figure 8—Scratch depths obtained with the pin and ball tests on coatings A-E (ball) and A-D (pin) without additives vs. crosslink density as calculated from equation (1). The tests were performed at a temperature 20° below the T_g of each resin, i.e., at 25, 35, 50, 130 and 180°C for coatings, A-E, respectively. The load in the pin test was 2 kg.

movement across the surface. The reason for this is presumably that during a single draw, the reduction in friction is the most important effect, whereas during repeated movement, it is the reduction in hardness caused by the wax that dominates. Why adding wax works better for the fiber test than for the ball test is believed to be related to the scratching mechanism. In the ball test, no material is retained and the reduction in hardness translates directly into reduced scratch resistance, whereas in the fiber tests the wax renders the removed material less abrasive, which somewhat counteracts the reduction in hardness.

CONCLUSIONS

The scratch resistance of a series of cationically cured epoxy coatings was examined with five different scratch tests. All tests showed the scratch resistance to increase both with increasing crosslink density and increasing glass transition temperature. Adding pigment to the coatings decreased the scratch resistance in tests where the material removed in the scratch process remained between the substrate and the scratching device, but increased the scratch resistance in other tests. Wax was found to increase the scratch resistance in tests where a scratch was created in a single draw, but to decrease the scratch resistance when scratching occurred as the result of repeated movement.

When selecting a test for screening of coating scratch resistance, any of the scratch tests could be used, since they all indicate the same trend with respect to mechanical properties. Such a rough evaluation could also be expected to give the same ranking as field tests, although this has yet to be confirmed. If more detailed information is desired, or if the effects of additives is to be evaluated, the choice of test becomes important. In this case at least a couple of tests covering a range of scratching situations should be used, and the agreement with behavior in the field thoroughly checked.

ACKNOWLEDGMENT

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Fax: (516) 694-9177

M. Blum, V.P.: M. Samuels, Product Mgr. Cellulose Derivatives

B My Supply, Inc. 635 N. Hickory Ave. Arlington Heights, IL 60004 Phone: (847) 398-6468 Fax: (847) 398-2897

David D. McCarthy, General Mgr., Rolf Grupp, Marketing Mgr.

Hydrocarbon Resins Natural Resins

Borden Inc., Forest Prods, Div. 114 Industrial Blvd. Morganton, NC 28655

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Dave Blough, National Account Mgr.; Peter Wallace, National Sales Mgr.

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Cardolite Corp. 500 Doremus Ave. Newark, NJ 07105 Phone: (201) 344-5015 Fax: (201) 344-1197 Deirdre Igo, Marketing Communications;

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919 E. 14th Ave. P.O. Box 419389 Kansas City, MO 64116 Phone: (816) 391-6000 Fax: (816) 391-6337

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West Paterson, NJ 07424
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Fax: (708) 496-7367
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CCP
The Degen Co.
Dock Resins Corp.
Engineered Polymer Solutions, Inc.
McWhorter Technologies
Ohio Polychemical Co.
Schenectady International, Inc.
Syntech, Inc.
Texas Resin Co.
U.S. Polymers, Inc.

Nondrying & Semi-Oxidizing, Oil-Modified

Accurez Corp.
Akzo Nobel Resins, Inc.
CCP
The Degen Co.
Engineered Polymer Solutions, Inc.
McWhorter Technologies
Schenectady International, Inc.
Syntech, Inc.
Texas Resin Co.
U.S. Polymers, Inc.

Thixotropic

Accurez Corp.
Akzo Nobel Resins, Inc.
CCP
McWhorter Technologies
U.S. Polymers, Inc.

Water-Reducible

Accurez Corp.
Akzo Nobel Resins, Inc.
CCP
Dock Resins Corp.
Engineered Polymer Solutions, Inc.
McWhorter Technologies
Ohio Polychemical Co.
Schenectady International, Inc.
Syntech, Inc.
Texas Resin Co.
U.S. Polymers, Inc.
Zeneca Resins

Misc. Alkyd Resins

Accurez Corp.
Akzo Nobel Resins, Inc.
CCP
The Chemical Co.
Dock Resins Corp.
Engineered Polymer Solutions, Inc.
McWhorter Technologies
Ohio Polychemical Co.
Schenectady International, Inc.
Texas Resin Co.
U.S. Polymers, Inc.

*Advertisers in this issue appear in color.

AMINO RESINS

Akzo Nobel Resins, Inc.
American Casein Co., Div. of Protein Polymer
Group
Borden Inc., Forest Prods. Div.
Cytec Industries, Inc.
Dock Resins Corp.
DSM Resins U.S., Inc.
McWhorter Technologies
Mitsubishi Chemical America

Reichhold Chemicals, Inc. Syntech, Inc.

CELLULOSE DERIVATIVES

Aceto Corporation
Atomergic Chemetals Corp.
HERCULES INCORPORATED, AQUALON DIV.
Union Carbide Corp.

EPOXY HARDENERS-CHEMICALS

Cardolite Corp.
CCP
Dock Resins Corp.
Estron Chemical, Inc.
Hardman, Div. of Harcross Chemical, Inc.
HENKEL CORP., COATINGS & INKS DIV.
HÜLS AMERICA INC.
Lonza, Inc. Specialty Anhydride
PolySat, Inc.
Royce Associates, ALP
SKW Chemicals, Inc.
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CPS Chemical Company, Inc.
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Liquid

Daubert Chemical Co., Inc. Shell Chemical Co. Union Carbide Corp.

1

Solid

Phenoxy Specialties Shell Chemical Co.

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Shell Chemical Co.
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Water-Based

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Misc. Epoxy Resins

CPS Chemical Company, Inc. Cytec Industries, Inc. Daubert Chemical Co., Inc. Lonza, Inc. Specialty Anhydride McWhorter Technologies Royce Associates, ALP Shell Chemical Co.

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Arakawa Chemical (USA) Inc. International Paper Arizona Chemical Resinall Corp.

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Arakawa Chemical (USA) Inc. Resinall Corp.

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HYBRID RESINS

CCP
Dock Resins Corp.
Raven Lining Systems-Tulsa
Reichhold Chemicals, Inc.
Resinall Corp.

HYDROCARBON RESINS

American Gilsonite Co. Arakawa Chemical (USA) Inc. B My Supply, Inc. Kinetics Corp. PolySat, Inc. Resinall Corp. Rit-Chem Co., Inc. SARTOMER CO.

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Arakawa Chemical (USA) Inc. PolySat, Inc.

Liquid/Solution

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Arakawa Chemical (USA) Inc.

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International Paper, Arizona Chemical Div.
Kinetics Corp.
Lonza, Inc. Specialty Anhydride
Mitsubishi Chemical America
Petrolite Corp., Polymers Div.
Resinall Corp.

Solid

Arakawa Chemical (USA) Inc. International Paper, Arizona Chemical Div. Kinetics Corp. Petrolite Corp., Polymers Div. Resinall Corp.

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International Paper, Arizona Chemical Div. Kinetics Corp. Petrolite Corp., Polymers Div. Resinall Corp.

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Akzo Nobel Resins, Inc. Borden Inc., Forest Prods. Div. The Chemical Co.
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McWhorter Technologies
Mitsubishi Chemical America
Monsanto Co., Coatings Performance Materials
Div.
Syntech, Inc.

METHYACRLIC MONOMERS & POLYMERS

Elf ATOCHEM North America, Inc.

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Dock Resins Corp.
DSM Resins U.S., Inc.
Engineered Polymer Solutions, Inc.
BFGoodrich Coatings Div.
Lonza, Inc. Specialty Anhydride
Lorama Chemicals, Inc.
McWhorter Technologies
Ohio Polychemical Co.
Phenoxy Specialties
PolySat, Inc.
Syntech, Inc.
Union Carbide Corp.
Zaclon, Inc.

Solid

DSM Resins U.S., Inc. Phenoxy Specialties Syntech, Inc.

Liquid & Solution

DSM Resins U.S., Inc.
Engineered Polymer Solutions, Inc.
Lorama Chemicals, Inc.
McWhorter Technologies
Phenoxy Specialties
PolySat, Inc.
Syntech, Inc.
Union Carbiide Corp.

Emulsion

Chemcor DSM Resins U.S., Inc. Engineered Polymer Solutions, Inc. BFGoodrich Coatings Div. McWhorter Technologies Phenoxy Specialties Union Carbide Corp.

Water-Reducible

DSM Resins U.S., Inc.
Engineered Polymer Solutions, Inc.
Lorama Chemicals, Inc.
McWhorter Technologies
Ohio Polychemical Co.
Syntech, Inc.
Union Carbide Corp.
Zaclon, Inc.

Waterborne Vinyl Chloride Acetate Copolymer Dispersion

Union Carbide Corp.

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Engineered Polymer Solutions, Inc.
BFGoodrich Coatings Div.
McWhorter Technologies
Royce Associates, ALP
U.S. Polymers, Inc.

Modified Phenolic

Accurez Corp.
Alvar, Inc.
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CCP
The Chemical Co.
The Degen Co.
Engineered Polymer Solutions, Inc.
McWhorter Technologies
Royce Associates, ALP
U.S. Polymers, Inc.

Phenolic

Accurez Corp.
Alvar, Inc.
Arakawa Chemical (USA) Inc.
The Chemical Co.
The Degen Co.
Engineered Polymer Solutions, Inc.
Royce Associates, ALP
U.S. Polymers, Inc.

Phenolic Free

Accurez Corp.
Alvar, Inc.
CCP
The Degen Co.
Royce Associates, ALP
U.S. Polymers, Inc.

PHENOLIC RESINS

Arakawa Chemical (USA) Inc.
The Chemical Co.
Cytec Industries, Inc.
DSM Resins U.S., Inc.
Elf ATOCHEM North America, Inc.
Enzymol International, Inc.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
International Paper, Arizona Chemical Div.
Lonza, Inc. Specialty Anhydride
Mitsubishi Chemical America

Monsanto Co., Coatings Performance Materials Div. Occidental Chemical Corp. Resinall Corp. Schenectady International, Inc. Vanguard Chemical International Inc.

Modified for Powder Coatings

The Chemical Co. Lonza, Inc. Specialty Anhydride Occidental Chemical Corp.

Polyamide

Lonza, Inc. Specialty Anhydride

Solid

Arakawa Chemical (USA) Inc.
The Chemical Co.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
International Paper Arizona Chemical
Occidental Chemical Corp.
Schenectady International, Inc.

Solution

The Chemical Co.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
International Paper Arizona Chemical
Occidental Chemical Corp.
Schenectady International, Inc.

Pure

Arakawa Chemical (USA) Inc.
The Chemical Co.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
Occidental Chemical Corp.
Schenectady International, Inc.

Heat Hardening-Solid

Arakawa Chemical (USA) Inc.
The Chemical Co.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
Occidental Chemical Corp.
Vanquard Chemical International Inc.

Heat Hardening-Solution & Dispersion

The Chemical Co.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
Occidental Chemical Corp.
Schenectady International, Inc.

Heat Reactive-Solid

Arakawa Chemical (USA) Inc.
The Chemical Co.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
Occidental Chemical Corp.
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Nonheat Hardening-Solid

The Chemical Co.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
Occidental Chemical Corp.

Nonheat Hardening-Solution

The Chemical Co.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
Occidental Chemical Corp.

Misc. Phenolic Resins

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The Chemical Co.
Cytec Industries, Inc.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
Occidental Chemical Corp.
Resinall Corp.
Schenectady International, Inc.
Vanguard Chemical International Inc.

PHENOXY RESINS

Elf ATOCHEM North America, Inc. Phenoxy Specialties

POLYAMIDE RESINS

Cytec Industries, Inc.
Elf ATOCHEM North America, Inc.
HENKEL CORP., COATINGS & INKS DIV.
Lonza, Inc. Specialty Anhydride
Mitsubishi Chemical America

POLYBUTADIENES

Elf ATOCHEM North America, Inc.

POLYBUTENES

Mitsubishi Chemical America

POLYESTER RESINS

Accurez Corp.
Akzo Nobel Resins, Inc.
CCP
The Chemical Co.
DSM Resins U.S., Inc.
Engineered Polymer Solutions, Inc.
Estron Chemical, Inc.
HÜLS AMERICA INC.
McWhorter Technologies
Mitsubishi Chemical America
Ohio Polychemical Co.
Reichhold Chemicals, Inc.
Ruco Polymer Corp.
Schenectady International, Inc.
Shell Chemical Co.
Syntech, Inc.
U.S. Polymers, Inc.
Zeneca Resins

Drying

CCP Engineered Polymer Solutions, Inc. Texas Resin Co.

Non-Drying

Akzo Nobel Resins, Inc. Engineered Polymer Solutions, Inc. Schenectady International, Inc. Syntech, Inc.

Oil-Free

Akzo Nobel Resins, Inc. CCP DSM Resins U.S., Inc. Engineered Polymer Solutions, Inc. McWhorter Technologies Ohio Polychemical Co. Schenectady International, Inc. Syntech, Inc.

Saturated

Accurez Corp.
Akzo Nobel Resins, Inc.
CCP
DSM Resins U.S., Inc.
Engineered Polymer Solutions, Inc.
HÜLS AMERICA INC.
McWhorter Technologies
Ruco Polymer Corp.
Schenectady International, Inc.
Shell Chemical Co.
Syntech, Inc.
Texas Resin Co.
U.S. Polymers, Inc.

Solid

DSM Resins U.S., Inc.

Unsaturated

CCP
The Chemical Co.
HÜLS AMERICA INC.
McWhorter Technologies

Water-Reducible

Akzo Nobel Resins, Inc. CCP DSM Resins U.S., Inc. Engineered Polymer Solutions, Inc. McWhorter Technologies Ruco Polymer Corp. Schenectady International, Inc. Syntech, Inc. Texas Resin Co.

Misc. Polyester Resins

The Chemical Co.
DSM Resins U.S., Inc.
Engineered Polymer Solutions, Inc.
Elf ATOCHEM North America, Inc.
Estron Chemical, Inc.
HÜLS AMERICA INC.
McWhorter Technologies
Ruco Polymer Corp.
Schenectady International, Inc.
Shell Chemical Co.
Syntech, Inc.
Texas Resin Co.

POLYETHYLENE RESINS

Mitsubishi Chemical America R.I.T.A. Corp. Union Carbide Corp.

POLYPROPYLENE RESINS

Mitsubishi Chemical America Union Carbide Corp.

POLYSACCHARIDE RESINS

Lorama Chemicals, Inc.

ROSIN ESTERS

Arakawa Chemical (USA) Inc.
The Chemical Co.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
International Paper, Arizona Chemical Div.
Kinetics Corp.
Resinall Corp.

ROSIN SALTS

Arakawa Chemical (USA) Inc. Georgia-Pacific Resins, Inc., Chemical Div. of Georgia-Pacific Corp. Resinall Corp.

SILANES

Dow Corning Corp. McWhorter Technologies

SILICONE POLYESTERS

McWhorter Technologies

SILICONE RESINS

CCP
Dow Corning Corp.
McWhorter Technologies
Rhône-Poulenc, Inc.
Tego Chemie Service USA
Wacker Silicones Corp.
Witco Corp., OLEO-Surf. Group

STYRENE RESINS

Akzo Nobel Resins, Inc. CCP The Chemical Co. Dock Resins Corp. The Dow Chemical Co. Elf ATOCHEM North America, Inc. Engineered Polymer Solutions, Inc. H.B. Fuller Co. BFGoodrich Coatings Div. Hampshire Chemical Corp. S.C. Johnson Polymer Mitsubishi Chemical America Morton International Ohio Polychemical Co PolySat, Inc. Reichhold Chemicals, Inc. Scott Bader, Inc.

Styrene & Vinyl Toluene Polymer & Copolymer—Solid

S.C. Johnson Polymer

Styrene & Vinyl Toluene Polymer & Copolymer—Solution

Akzo Nobel Resins, Inc. CCP Dock Resins Corp. Engineered Polymer Solutions, Inc. S.C. Johnson Polymer McWhorter Technologies Ohio Polychemical Co. PolySat, Inc. Texas Resin Co.

Styrene-Butadiene Emulsion

The Dow Chemical Co. H.B. Fuller Co. BFGoodrich Coatings Div. Hampshire Chemical Corp.

Urea Resins

The Chemical Co. McWhorter Technologies

Misc. Styrene Resins

Elf ATOCHEM North America, Inc. Engineered Polymer Solutions, Inc. H.B. Fuller Co.

RESINS: product listing

S.C. Johnson Polymer Morton International PolySat, Inc. Scott Bader, Inc.

TERPENES

Aerocote Corp. Arakawa Chemical (USA) Inc.

TGIC RESINS

Aceto Corporation DSM Resins U.S., Inc. Royce Associates, ALP Ruco Polymer Corp. Syntech. Inc.

ULTRAVIOLET CURING RESINS

CPS Chemical Company, Inc.
DSM Resins U.S., Inc.
Echo Resins and Laboratory
HENKEL CORP., COATINGS & INKS DIV.
McWhorter Technologies
San Esters Corp.
SARTOMER CO.
Syntech, Inc.
UCB Chemicals Corp.
Union Carbide Corp.

URETHANE CROSSLINKERS

McWhorter Technologies Mitsubishi Chemical America

URETHANE LACQUERS

BFGoodrich Coatings Div.

URETHANE & POLYURETHANE RESINS

Akzo Nobel Resins, Inc. CasChem, Inc. CCP Cytec Industries, Inc. Dock Resins Corp. BFGoodrich Coatings Div. Hardman, Div. of Harcross Chemical, Inc. HENKEL CORP., COATINGS & INKS DIV. ICI Polyurethanes International Specialty Products (ISP) Kane International Corp. McWhorter Technologies Mitsubishi Chemical America Ohio Polychemical Co. K.J. Quinn & Co., Inc. Reichhold Chemicals, Inc. Ruco Polymer Corp. Schenectady International, Inc. Syntech, Inc. 21st Century Coatings Inc. Texas Resin Co. Witco Corp., OLEO-Surf. Group Zeneca Resins

ASTM Type I-One-Package Precured

Akzo Nobel Resins, Inc. McWhorter Technologies Ohio Polychemical Co. K.J. Quinn & Co., Inc. Ruco Polymer Corp.

ASTM Type II—One-Package Moisture Cured

ICI Polyurethanes K.J. Quinn & Co., Inc. Ruco Polymer Corp. Texas Resin Co.

ASTM Type III—One-Package Heat Cured

Kane International Corp. McWhorter Technologies K.J. Quinn & Co., Inc. Ruco Polymer Corp. 21st Century Coatings Inc.

ASTM Type IV-Two-Package Catalyst

CasChem, Inc.
HENKEL CORP., COATINGS & INKS DIV.
K.J. Quinn & Co., Inc.
Syntech. Inc.

ASTM Type V-Two-Package Polyol

CasChem, Inc.
HENKEL CORP., COATINGS & INKS DIV.
McWhorter Technologies
K.J. Quinn & Co., Inc.
Ruco Polymer Corp.
Syntech, Inc.
21st Century Coatings Inc.

Waterborne

Cytec Industries, Inc.
Dock Resins Corp.
BFGoodrich Coatings Div.
Kane International Corp.
McWhorter Technologies
K.J. Quinn & Co., Inc.
Ruco Polymer Corp.
Syntech, Inc.
Witco Corp., OLEO—Surf. Group

Misc. Urethane and Polyurethane Resins

K.J. Quinn & Co., Inc. Ruco Polymer Corp. Witco Corp., OLEO-Surf. Group

VINYL CHLORIDE & VINYLIDENE CHLORIDE POLYMER EMULSIONS

BFGoodrich Coatings Div. Hampshire Chemical Corp. Mitsubishi Chemical America Scott Bader, Inc. Union Carbide Corp. Zeneca Resins

VINYL RESINS

Elf ATOCHEM North America, Inc. Engineered Polymer Solutions, Inc. H.B. Fuller Co. BFGoodrich Coatings Div. Hampshire Chemical Corp. International Specialty Products (ISP) Mitsubishi Chemical America Nacan Products Ltd., Resins Div. Reichhold Chemicals, Inc. Union Carbide Corp.

Polyvinyl Acetate

H.B. Fuller Co. McWhorter Technologies Union Carbide Corp.

Polyvinyl Chloride & Polyvinylidene Chloride

BFGoodrich Coatings Div.

Solution

McWhorter Technologies Union Carbide Corp.

Vinyl Acetate Polymer Emulsions

Engineered Polymer Solutions, Inc. H.B. Fuller Co. BFGoodrich Coatings Div. Hampshire Chemical Corp. Nacan Products Ltd., Resins Div. Union Carbide Corp.

Vinyl Copolymers

H.B. Fuller Co. Hampshire Chemical Corp. International Specialty Products (ISP) McWhorter Technologies Nacan Products Ltd., Resins Div. Texas Resin Co. Union Carbide Corp.

Misc. Vinyl Resins

Elf ATOCHEM North America, Inc. BFGoodrich Coatings Div. Hampshire Chemical Corp. Nacan Products Ltd., Resins Div.

WATER-THINNED RESINS

Akzo Nobel Resins, Inc.
BFGoodrich Coatings Div.
DSM Resins U.S., Inc.
Engineered Polymer Solutions, Inc.
Georgia-Pacific Resins, Inc., Chemical Div. of
Georgia-Pacific Corp.
McWhorter Technologies
Occidental Chemical Corp.
Ohio Polychemical Co.
Reichhold Chemicals, Inc.
Ruco Polymer Corp.
S.C. Johnson Polymer
Union Carbide Corp.
Zadon. Inc.

Lab Apparatus

FTIR Sampling Accessory ASI Sensir TECHNOLOGIES



ASI SensIR Technologies has announced the availability of DuraSampIIR™-EL. The entry-level SiComp (silicon based) product provides throughput and rapid sample analysis and can be upgraded to the DiComp (diamond based) DuraSampIIR. In addition, DuraSampIIR™-EL enables the ATR analysis of corrosive liquids and abrasive powders.

Circle No. 47 on Reader Service Card

PosiTector 6000-FHS DEFELSKO CORP.



Thick non-magnetic coatings on steel, such as epoxy, rubber, or fire-proofing can be measured using the PosiTector 6000-FHS. This DeFelsko Corp. product has a measuring range of up to 750 mils, with a resolution of 1 mil in the 0-400 mil range, and 5 mils in the 400-750 mil range. The FHS probe connects to the gage using a four-foot cable.

Circle No. 51 on Reader Service Card

Color Measurement Guide X-RITE, INCORPORATED



A guide to color measurement and quality control in paint formulation and finishing has been published by X-Rite, Incorporated. This publication provides a basic definition of color and explains how color is measured and interpreted as a precise color "fingerprint." Descriptions and application guidelines are also included in this guide.

Circle No. 48 on Reader Service Card

AA Spectrophotometers VARIAN ANALYTICAL INSTRUMENTS

A brochure available from Varian describes new atomic absorption spectrometers, including the stand-alone SpectrAA-50/55, the PC-controlled SpectrAA-110/220, and the industry's first sequential (FS), the SpectrAA-220 FS. The Windows $^{\rm TM}$ 95-based worksheet software is fully illustrated and discussed in this publication. Also featured is the company's Mark 7 flame atomization system.

Circle No. 49 on Reader Service Card

FISCHER TECHNOLOGY

The Fischerscope MMS has been developed to perform non-destructive testing of coating thickness using either the electromagnetic, eddy current, or beta backscatter method from a single desktop unit. The MMS, introduced by Fischer Technology, can also document, manage and evaluate measurement data using modern SPC/SQC methods, and organize data.

Circle No. 52 on Reader Service Card

Precision Thermometers BROOKLYN THERMOMETER CO., INC.



The Brooklyn Thermometer Co. has introduced a line of precision grade liquid-in-glass thermometers engraved in degrees Fahrenheit or centigrade. These thermometers are individually graduated in 0.2, 0.1, or 0.05 degree subdivisions and are available in more than 80 temperature ranges from –50 to 310°C and –35 to 465°F. They are filled with triple distilled mercury or red liquid spirits.

Circle No. 53 on Reader Service Card

Ultraviolet Reflectance Analyzer LABSPHERE, INC.



The UV-1000R ultraviolet reflectance analyzer is a benchtop spectroreflectometer designed for the measurement of diffuse ultraviolet reflectance over the wavelength region of 250 to 450 nm. Introduced by Labsphere, Inc., the UV-1000R analyzes the loss of sunscreen protection on the skin. The instrument's Windows™based software features a report generation function that calculates measurement results and displays critical information.

Circle No. 50 on Reader Service Card

CCD Spectrophotometer CVI SPECTRAL INSTRUMENTS



CVI Spectral Instruments highlights the SpectraMatch® SM-210 PC-based spectrophotometer for both spectral and color analysis in a laboratory, production, on-line, or QC environment. It can be used as a hand-held unit to measure color and color difference or it can be used with fiber optics to measure color in remote areas. It comes with a Windows™-based software that displays spectral information and absolute and color difference data

Circle No. 54 on Reader Service Card

People in the News



D. Rice

Kronos, Inc., Houston, TX, has appointed David Rice as Account Manager. Based in Cleveland, OH, Mr. Rice will cover the company's coatings, ink, and plastics accounts in Northern Ohio, Michigan, and Northeast Indiana.

He is a member of the Cleveland Society.

BYK-Gardner USA, Columbia, MD, has appointed **Mike Gogoel** to the position of General Manager. In this capacity, Mr. Gogoel will oversee day-to-day business of the company's operations in North and South America. He is a member of the Baltimore Society.

The Dow Chemical Co., Midland, MI, has restructured its Coatings Group. Accepting new positions in the sales organization are the following: Joel Cohen will serve both Architectural Coatings customers and Plastic Pigments customers; Irving Gerlach will serve customers in the Industrial Coatings area; Terry Christie will provide service in the Traffic Paint market; Both Julie Groen and Bob Harris will serve Dow Distributors in Canada; and Dennis Harrison will serve Dow Distributors in the United States. Ms. Groen is a member of the Toronto Society and Mr. Harris is a member of the Montreal Society.

Witco Corp., Greenwich, CT, has named Chuck Costas Director of Sales and Sales Services for its Industrial Surfactants and Petroleum Additives Strategic Business Units (SBUs). Mr. Costas will manage the North American sales and sales services personnel in these areas as well as provide guidance for functional excellence for all of the selling units of the global Performance Chemicals Group.

Brian Hagaman has accepted the posi-

tion of C.A.S.E.
Polyol Market
Manager for
Stepan Co.,
Northfield, IL. A
member of the
New England Society, Mr. Hagaman brings 11
years of experience in the chemical industry to this
position.



B. Hagaman

Chicago Society Honors Evans Angelos and William Fotis with Outstanding Service Awards

The Chicago Society for Coatings Technology presented both Evans Angelos, OMYA, Inc., and Bill Fotis, of The Valspar Corp. with The Outstanding Service Award. C. David Stromberg presented the Award to Evans Angelos and Ed Croco gave the Award to Bill Fotis. Both Messrs. Angelos and Fotis are Past-Presidents of the CSCT. Mr. Angelos currently serves as Society Representative and Mr. Fotis is Chair of the CPCA Educational Committee. In addition, \$2,000 Scholarship Certificates were presented to Mihir Shah, daughter of Barti Shah of Tru-Test Manufacturing Co. and Kristen Schmid, daughter of Steven Schmid of Engineered Polymer Solutions.

Michael M. Patterson has been named President of R.B. Patterson and Co., Inc., Houston, TX. A member of the Houston Society, Mr. Patterson previously held the position of Vice President of Sales.

Raymond J. Ehrlich has accepted the position of Director of Environmental Affairs for the Polystyrene Packaging Council, a business unit of The Society of the Plastics Industry, Inc., Washington, D.C.

Roman Adhesives, Inc., Calumet City, IL, has announced the appointment of **Kenneth Brown** to the newly created position of Technical Director. In this capacity, Mr. Brown will refine and develop the company's product lines.

Columbian Chemicals Co., Atlanta, GA, has announced the promotion of **Dietmar Voss** to Senior Vice President Market and Sell Worldwide. In addition, Mr. Voss will continue as a member of the Columbian Leadership Team.

COATEGRITY AFFORDABILITY FRADELITY

COATING PROBLEMS?

Eliminate unwanted coating problems before they occur. Gamry Instruments' EIS900 Impedance System can determine a coating's performance characteristics by measuring the properties of the coating itself. Use that information to detect application problems, specification

deviations, or potential coating failure before it is visable. Unlike salt spray tests, EIS is repeatable and can be performed under varying conditions to allow you to chose the optimal coating. Think how a longer and more predictable service life can lower your coating costs.





COATEGRITY PORTABILITY SIMPLICITY

Gamry Instruments, Inc.

734 Louis Drive Warminster, PA 18974 www.gamry.com Tel: 215-682-9330 Fax: 215-682-9331 sales@gamry.com

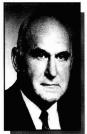
FLEXIBILITY PORTABILITY HOOPSPITALITY

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H@PSPITALITY OCTOPLICITY FRADELITY

Obituaries

FSCT Past-President Carlton H. Rose Passes Away in March



Carlton H. Rose, FSCT Past-President, passed away on March 31, 1997.

Mr. Rose received a B.S. Degree in Chemistry from the University of California in 1924. Upon graduation, he served National Lead Co., Oakland, CA, as a Chemist. Mr. Rose was later named Technical Representative of the firm.

In 1932, Mr. Rose joined the staff of the National Lead Co. Research Laboratories, Brooklyn, NY. He later became head of the company's Paint and Pigment Research Department.

In 1947, Mr. Rose was elected President of the Federation of Societies for Coatings Technology. In addition, he was an Honorary and 50-year member of FSCT.

C.H. Rose Mr. Rose served both the Golden Gate Society and the New York Society as President. Also, he was a member of the American Chemical Society, Gallows Birds Society, the Board of Directors of the American Society for Testing and Materials (ASTM) and Secretary of ASTM's Committee D-1. Mr. Rose also served on the editorial staff of ACS' Chemical Abstracts.



Royal A. Brown, former Director of the Technical Division of the National Paint and Coatings Association, died on July 8, 1997. He was 83-years old.

Mr. Brown received a B.S. Degree in Organic Chemistry from Marietta College and graduated from the Executive Development Program at the Graduate School of Commerce of Ohio State University.

Mr. Brown worked for many years in the paint industry. Among the positions he held were Plant Manager of the Chemical Division of The Stanley Works, Technical Director of The Egyptian Lacquer Manufacturing Co., and Vice President of Research and Manufacturing for the Southern Varnish Corp.

K.A. Brown In 1966, Mr. Brown was named Director of the Technical Division of the National Paint and Coatings Association. There he supervised the work of NPCA's Technical Division, advised several association, technical, manufacturing, and ecological committees, and supervised the preparation and production of technical publications.

Mr. Brown served as a consulting Technical Advisor for the FSCT, coordinating technical programming for FSCT activities. Mr. Brown was a member of the New York, Southern, and Baltimore Societies for Coatings Technology. In addition, he served as President of the Washington Paint Technical Group, and was voted their "Man of the Year" in 1974.

An author of many published articles relating to technical aspects of the paint and coatings industry, Mr. Brown had also given many presentations to various industry and academic groups.

Mr. Brown was an active member of the American Chemical Society, the American Association for the Advancement of Science, as well as the American Society for Testing and Materials. He served on the Executive Committee of ASTM Committee D-1. In addition, Mr. Brown was a Trustee of the Paint Research Institute and was active in the Gallows Birds Society, where he served as High Gallows Bird.

Merle D. Held, a retired member of the Cleveland Society, passed away recently. He was 72 years old.

Mr. Held was born in Cleveland, OH. He graduated from University School and attended the University of Wisconsin. In addition, he earned a degree in Chemical Engineering at Case Institute of Technology.

Mr. Held served as Marketing Manager for Cyprus Mines Corp.

John Russell Griswold, a member of the Kansas City Society, died on July 14, 1997. Mr. Griswold was the owner of Charles Paint Research Inc., Kansas City, MO.

A Past-President of the Kansas City Paint and Coatings Association, Mr. Griswold also served on the Executive Committee, and held the position of Executive Secretary and Past Assistant Treasurer.

Fusion UV Systems, Inc., Gaithersburg, MD, has appointed Michael L. Feren as Direct Sales Representative for the Canadian territory. In his new position, Mr. Feren will be responsible for the development of new business and servicing existing customers in his region and territory.

Hüls America Inc., Somerset, NJ, has named **John Rosato** as Director, Logistics. In this position, Mr. Rosato will provide cost-effective management for the company's electronic commerce, distribution, transportation, and inventory management network.

Karen Bilinski has accepted the position of Sales Account Manager for Magnus Equipment Group, Willoughby, OH. Ms. Bilinski will be responsible for developing and increasing standard equipment sales.

John A. Beahm has accepted the position of Marketing Communications Manager for Watson-Marlow, Inc., Wilmington, MA. Mr. Beahm will manage the company's U.S.-based corporate communications and will work with the inside and outside sales forces.

Mark E. Peden, Head of the Chemistry Division, Illinois State Water Survey, Champaign, IL, has received a 1997 American Society for Testing and Materials Award of Merit from ASTM standards writing Committee D-22 on Sampling and Analysis of Atmospheres. Mr. Peden was cited for outstanding service in developing standards and test methods in Committee D-22.

Anthony G. Fernandes, Executive Vice President of ARCO Chemical Co., Newtown Square, PA, has been elected Chairman of the Board. Mr. Fernandes, an ARCO Chemical board member since 1996, replaces Mike R. Bowlin, Chairman and Chief Executive Officer.

James A. Vysoky has been promoted to the newly created position of Manager,

Product Development/Industrial Additives for AN-GUS Chemical Co., Buffalo Grove, IL. Mr. Vysoky will be responsible for new product development in tertiary amines for the paint and coatings market and new amines for refinery corrosion.



J.A. Vysoky



Water-Based Epoxies

The Sherwin-Williams Chemical Coatings group has unveiled the Kem Aqua® Hyrdalon line of water-based epoxies. The new line includes Hydralon P, a water-reducible primer and Hydralon B and Hydralon T, water-reducible enamels with high-gloss and low-gloss finishes, respectively. The new waterborne epoxies are free of lead, chromates, and isocyanates and compliant with 2.8 VOC air quality regulations.

Circle No. 30 on Reader Service Card

Wood Coating

Van Technologies, Inc. has developed an advanced line of environmentally compliant wood coatings. The product line consists of washcoats, stains, sealers, and topcoats.

Circle No. 31 on Reader Service Card

Elastomeric Lining

An elastomeric polyurethane coating made by Futura Coatings, Inc. has been formulated with Bayer Corporation's Desmodur® aliphatic polyisocyanates. The polyurethane lining, part of a three-coat system, is expected to help minimize the migration of water and sulfite/sulfate salts through cracks in concrete.

Circle No. 32 on Reader Service Card



5.0 Class-VP

Shimadzu's new 32-bit Class-VP Version 5 software (Chromatography Laboratory Automated Software System, Validation and Productivity) is now available. The advanced log file is now instrument and method specific and is stored with the data. Data validation features method, results, instrument configuration and log file stored with the data ensuring compliance with regulatory requirements.

Circle No. 33 on Reader Service Card

CMMS Technology

Summit Software's new M-Pet version 4.0 incorporates the features of advanced windows/client server technology, an interactive on-screen scheduler, and concepts of TPM, overall equipment effec-

tiveness, and reliability and maintainability. New features include a suite of TPM related reports, equipment downtime module, and an equipment hierarchy interface that displays multiple levels of equipment, while allowing users to drill-down into specific equipment job lists.

Circle No. 34 on Reader Service Card

Color Quality Control

Datacolor International has developed a software package that reportedly controls the quality of color at every step of manufacturing and assembly. ColorTools QC® measures the color and appearance on all types of materials, components, and finished products to assure that they are correct at incoming, in-processing, and outgoing inspections. This software features analytical and data management tools as well.

Circle No. 35 on Reader Service Card

Spectrum for Windows Software

Perkin-Elmer Spectrum[™] for Windows[®] software now supports the company's

Model 16PC and 1600 Series FTIR systems. Users of the 16PC/1600 Series will benefit from the validated FTIR software available for instrument control, data collection and transfer, and instrument validation. Users can choose from two packages: the Spectrum for windows Validation and the Product Validation for Regulatory Compliance Package.

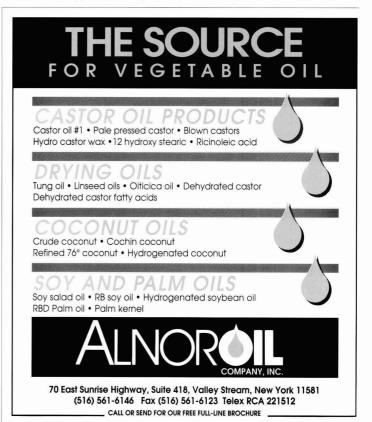
Circle No. 36 on Reader Service Card



Xenon Arc Test Instruments

Atlas Electric Devices Co. has introduced the XenoCal sensor designed to measure irradiance and radiant exposure for Xenotest brand of equipment, including the Suntest, the Alpha and Bea series, and 150S. Two sensors, one for measurement of UV irradiation (300-400 nm) and one for global solar irradiation (300-800 nm) are available.

Circle No. 37 on Reader Service Card



Circle No. 129 on Reader Service Card



Mass Flow Computer

The Schlumberger NexGen® SFT 100™ flow transmitter interfaces with the company's line of Coriolis mass flowmeter transducers. The SFT 100 uses digital signal processing technology to provide direct measurement of mass, density, and temperature and calculated measurements of volume, standard volume, percent concentration/solids and concentration/solids total. This product offers three design levels: blind transmitter, digital communications, and local interface.

Circle No. 38 on Reader Service Card

Cold-Glue Labeler

Pioneer Packaging Machinery has introduced a cold glue labeler as an alternative to traditional horizontal roll-thru design. The PR-90 features start-up times as low as 10 min and change-overs (from gallons to quarts) accomplished in one minute. The gluing method applies a thin film of water-soluble glue to the label while the labels feed out of a label track and then to the bypassing containers.

Circle No. 39 on Reader Service Card

DCF Filters

Ronningen-Petter's DCF Series Clean-in-Place Filters now feature permanent perforated media for strainer applications. Permanent media reduces downtime, waster disposal, and media replacement costs versus disposable media. Three sizes of performations are available for media retentions of 1,550, 3,125, or 6,250 microns.

Circle No. 40 on Reader Service Card

Portable Air Cleaning Systems

A new, full line of portable air cleaning systems is available from Aercology, Inc. The WA Series includes three models designed for applications from weld reworking and brazing to production welding. Each unit has a standard 10-foot arm with pick-up hood. A one-horse-power blower motor provides up to 1,100 CFM of air flow.

Circle No. 41 on Reader Service Card



High Performance Acrylics

CCP produces high performance acrylics. Featured acrylics include quick drying thermoplastics, weather resistant two-components, and high-gloss thermosets.

Circle No. 42 on Reader Service Card

Carbonyl Iron Powders

International Specialty Products (ISP) has announced the availability of carbonyl iron powders used for a variety of precision, industrial, aerospace, food supplement, and pharmaceutical applications. The fine powders are marketed under the Micropowder® and Ferronyl® trademark names.

Circle No. 43 on Reader Service Card

Pigment Dispersions

Tint-Ayd "ST" 8516 and Tint-Ayd "AL" 516 Van Dyke Brown pigment dispersions have been added to Daniel Products' standard solvent acrylic and modified alkyd lines, respectively. The pigment dispersions are for use in solvent-borne high-performance coatings based on alkyd, acrylic, urethane, polyester, vinyl, and cellulosic vehicle systems. They are reportedly free-flowing, easy-to-use pigment concentrates that provide stability and compatibility with solvent-borne trade sales, maintenance, and industrial coatings.

Circle No. 44 on Reader Service Card

Elastomeric Potting Compound

Insulcast has announced the availability of Insulcast 42, a self-priming, elastomeric potting compound. Designed to replace conventional RTV silicones, this product is black in color and has a usable temperature range of -55° to 155°C and meets UL94V-0 for flame retardancy.

Circle No. 45 on Reader Service Card

Plastic Additive

A slip additive that achieves combinations of properties needed for printing on thermoplastics with water-based inks that cannot be obtain with conventional slip agents has been introduced by Witco Corporation's Polymer Chemicals Group. Hydroslip 921 fatty acid is useful as an internal mold release agent for polyolefins. Derived from erucic acid, the N-hydroxyethyl additive is available in bead form.

Circle No. 46 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.Web site: http://www.coatingstech.org

1997

(Nov. 3-5)—ICE '97—FSCT Annual Meeting and International Coatings Expo and Technology Conference (Formerly Annual Meeting and Paint Industries' Show). Georgia World Congress Center, Atlanta, GA.

1998

(Oct. 14-16)—ICE '98—FSCT Annual Meeting and International Coatings Expo and Technology Conference (Formerly Annual Meeting and Paint Industries' Show). Ernest N. Morial Convention Center, New Orleans, LA.

SPECIAL SOCIETY MEETINGS

1998

(Feb. 18-20)—25th Annual International Waterborne, High-Solids, and Powder Coatings Symposium. Sponsored by the Southern Society and The University of Southern Mississippi (USM). New Orleans, LA. (Robson F. Storey or Shelby F. Thames, Co-Organizers, WHS&PC Symposium, Dept. of Polymer Science, USM, Box 10076, Hattiesburg, MS 39406-0076).

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(April 22)—"Manufacturing Symposium." Co-sponsored by the Cleveland Society for Coatings Technology and the Pittsburgh Society for Coatings Technology. (James Currie, Jamestown Paint Co., 108 Main St., P.O. Box 157, Jamestown, PA 16134; 412-932-3101).

(April 23-24)—"Waterborne Coatings: Sink or Swim II." 41st Annual Technical Symposium. Co-sponsored by the Cleveland Society for Coatings Technology and the Pittsburgh Society for Coatings Technology. (Vicki Fisher, Jamestown Paint Co., 108 Main St., P.O. Box 157, Jamestown, PA 16134; 412-932-3101).

(May 11-14)—Eastern Training Conference II. Sponsored by the Philadelphia Society. Valley Forge Convention Center, King of Prussia, PA. (Wayne Kraus, Hercules Incorporated, Research Center, 500 Hercules Rd., Wilmington, DE 19808).

1999

(Feb. 16-18)—24th Biennial Western Coatings Societies's Symposium and Show. Sponsored by the Golden Gate, Los Angeles, Pacific Northwest, and Rocky Mountain Societies. John Ascuaga's Nugget, Sparks, NV.

OTHER ORGANIZATIONS 1997-North America



(Sept.14-15)—Inter-Society Color Council (ISCC) Annual Meeting, Mar-

riott Inner Harbor, Baltimore, MD. (Robert T. Marcus, ISCC Publicity Chairman, D&S Plastics International, 100 S. Mitchell Rd., Mansfield, TX 76063).

(Sept. 14-18)—Third Annual ASM Surface Engineering Symposium. Sponsored by The Materials Information Society. Indianapolis, IN. (ASM Member Services Center, Materials Park, OH 44073-0002).

(Sept. 14-19)—"A Systems Approach to Service Life Prediction of Organic Coatings." The Village at Breckenridge, Breckenridge, CO. (Jonathan W. Martin, NIST, Bldg. 226, Rm. B350, Gaithersburg, MD 20899).

(Sept. 15-17)—"Fine Powder Processing." Sponsored by The Particulate Materials Center of The Pennsylvania State University in cooperation with Hosokawa Micron Powder Systems. Penn State Scanticon Hotel and Conference Center, State College, PA. (Particulate Materials Center, 147 Research Bldg. W., University Park, PA 16802-6809).

(Sept. 17-19)—"Molecular Modeling of Polymers." Sponsored by the American Chemical Society. University of Akron, Akron, OH. (Amercian Chemical Society, Dept. of Continuing Education, Meeting Code AKR9709, 1155 16th St., N.W., Washington, D.C. 20036).

(Sept. 22-25)—"Safety in Ammonia Plants and Related Facilities." Sponsored by the American Institute of Chemical Engineers (AIChE). Fairmont Hotel, San Francisco, CA. (AIChE, 345 E. 47th St., New York, NY 10017).

(Sept. 22-26)—"Introduction to Paint Formulation." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (UMR Coatings Institute, 1870 Miner Circle, Rolla, MO 65409).

(Sept. 23-25)—"Greensboro '97 Advanced Productivity Exposition." Sponsored by Society of Manufacturing Engineers (SME). Greensboro Coliseum Complex, Greensboro, NC. (SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121-0930).

(Sept. 29-Oct. 1)—"Fundamentals of Corrosion and Its Control." Sponsored by LaQue Corrosion Services, Wrightsville Beach, NC. (Sherree Darden, LaQue Center for Corrosion Technology, Inc., P.O. Box 656, Wrightsville Beach, NC 28480).

(Oct. 7-9)—"ISA Tech/97." Sponsored by International Society for Measurement and Control. Anaheim, CA. (ISA, 67 Alexander Dr., P.O. Box 12277, Research Triangle Park, NC 27709).

(Oct. 7-9)—Powder Coating '97. Sponsored by The Powder Coating Institute. Charlotte Convention Center, Charlotte, NC. (Vicki



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Materials CC Manufacturers of Equipment and Containers

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Thatcher, Goyer Management Int'l, Inc., P.O. Box 54464, Cincinnati, OH 45254).

(Oct. 13-15)—ASTM Committee D-22 on Sampling and Analysis of Atmospheres. Sponsored by the American Society for Testing and Materials (ASTM). Sheraton San Diego Hotel and Marina, San Diego, CA. (George Luciw, ASTM, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959).

(Oct. 14-16)—17th Aerospace Testing Seminar. Sponsored by the Institute of Environmental Sciences and The Aerospace Corp. Los Angeles, CA. (Institute of Environmental Sciences, 940 E. Northwest Hwy., Mount Prospect, IL 60056).

(Oct. 16-17)—"Spectroscopy Instrumentation and Application Seminars." Sponsored by Labsphere, Inc. North Sutton, NH. (Labsphere, Inc., P.O. Box 70, Shaker St., North Sutton, NH 03260).

(Oct. 21)—"Engineered Polymer Blends: VII Theory and Practice." Symposium sponsored by Polymer Research Institute, Polytechnic University. AlliedSignal Learning Center, Morristown, NJ. (Eli Pearce, Polymer Research Institute, Polytechnic University, 6 Metrotech Center, Brooklyn, NY 11201).

(Oct. 22-24)—"Spray Finishing Technology Workshop." Sponsored by Bowling Green State University and ITW DeVilbiss. ITW DeVilbis Training Center, Maumee, OH. (Richard A. Kruppa, Dept. of Technology Systems, College of Technology, Bowling Green, OH 43403-0301).

(Oct. 24-25)—"Fall Decor '97: Paint & Decorating Show." Sponsored by the National Decorating Products Association (NDPA). Cervantes Convention Center, St. Louis, MO. (Teri Flotron, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132-2994).

(Oct. 26-29)—Fall Convention. Sponsored by The Adhesive and Sealant Council, Inc. Hyatt Regency Dearborn, Dearborn, MI. (The Adhesive and Sealant Council, Inc., 1627 K St., N.W., Ste. 1000, Washington, D.C. 20006).

(Oct. 26-29)—"Adhesive Short Course II." Sponsored by The Adhesive and Sealant Council, Inc. Hyatt Regency Dearborn, Dearborn, MI. (The Adhesive and Sealant Council, Inc., 1627 K St., N.W., Ste. 1000, Washington, D.C. 20006).

(Oct. 26-29)—"End Use Applications Short Course." Sponsored by The Adhesive and Sealant Council, Inc. Hyatt Regency Dearborn, Dearborn, MI. (The Adhesive and Sealant Council, Inc., 1627 K St., N.W., Ste. 1000, Washington, D.C. 20006).

(Oct. 29-30)—"VOCs in Paints and Other Products." Sourse sponsored by the American Society for Testing and Materials (ASTM). Research Triangle Park, NC. (ASTM, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959).

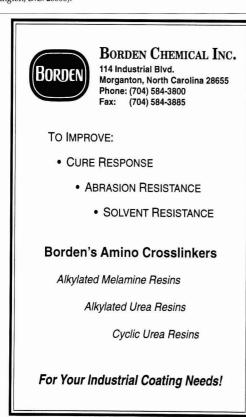
(Oct. 29-31)—Meeting of the International Biodeterioration Research Group. Meadowlands Hilton, Secaucus, NJ. (Marie Marabuto, Troy Corp., 8 Vreeland Rd., P.O. Box 955, Florham Park, NJ 07932).

(Nov. 2-4)—110th Annual Meeting and Industry Leadership Conference of the National Paint and Coatings Association (NPCA). Atlanta Hilton and Towers, Atlanta, GA. (NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005-5597; (202) 462-6272).

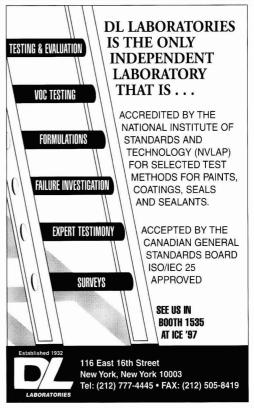
(Nov. 3-5)—ICE '97—FSCT Annual Meeting and International Coatings Expo and Technology Conference (Formerly Annual Meeting and Paint Industries' Show). Georgia World Congress Center, Atlanta, GA. (Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422 (610) 940-0777, FAX: (610) 940-0292. Web site: http://www.coatingstech.org).

(Nov. 3-5)—Second North American Research Conference on Emulsion Polymers/Polymer Colloids. Sponsored by the State University of New York. Hilton Head Island, SC. (Angelos V. Patsis, Institute of Materials Science, State University of New York, New Paltz, NY 12561).

(Nov. 10-13)—ASTM Committee B-08 on Metallic and Inorganic Coatings. Sheraton San Diego Hotel and Marina, San Diego, CA. (Dan







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Smith, ASTM, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959).

(Nov. 12)—"Effects of Surface Finish on Corrosion Testing." Sponsored by the American Society for Testing and Materials (ASTM). San Diego, CA. (ASTM, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959)

(Nov. 12-14)—ASTM Committee G-01 on Corrosion of Metals. Sheraton San Diego Hotel and Marina, San Diego, CA. (Bob Held, ASTM, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959).

(Nov. 13-14)—"Production Planning and Scheduling." Workshop sponsored by the National Paint and Coatings Association (NPCA). Rosemont Suites Hotel O'Hare, Chicago, IL. (Melina Jimenez, NPCA, 1500 Rhode Island Ave., N.W., Washington, D.C. 20005-5597)

(Nov. 15-17)-1997 Fluid Controls Institute Annual Meeting. LeMeridien, Boston, MA. (Fluid Controls Institute, Inc., 1300 Sumner Ave., Cleveland, OH 44115-2851).

(Nov. 17-20)—ICALEO '97. Sponsored by The Laser Institute of America. Catamaran Resort Hotel, San Diego, CA. (Laser Institute of America, 12424 Research Pkwy., Ste. 125, Orlando, FL 32826).

(Dec. 8-10)-Electroless Nickel '97. Sponsored by Products Finishing Magazine. Hyatt Regency, Cincinnati, OH. (Cindy Goodridge, Gardner Management Services, 6915 Valley Ave., Cincinnati, OH 45244-3029).

1998 – North America

(Jan 13)—PCI Technical Subcommittee on Test Methods and ASTM D01.51 on Powder Coatings. Sponsored by the American Society for Testing and Materials. Sheraton Harbor Island Hotel, San Diego, CA. (Jeffrey Hagerlin, O'Brien Powder Products, 9800 Genard Rd., Houston, TX 77041).

(Jan. 14-17)—"Winter Leadership Conference." Sponsored by The Adhesive and Sealant Council, Inc. The Registry, Naples, FL. (The Adhesive and Sealant Council, Inc., 1627 K St., N.W., Ste. 1000, Wash-

(Jan. 20-23)—"Environmentally Compliant Coatings." Course sponsored by The Department of Polymers and Coatings, North Dakota State University. Crowne Plaza Resort, Hilton Head Island, SC. (Debbie Shasky, Program Coordinator, North Dakota State University, Dept. of Polymers and Coatings, 54 Dunbar Hall, Fargo, ND 58105).

(Feb. 9-12)—"Composites '98 Manufacturing and Tooling Conference and Exhibits." Sponsored by Society for Manufacturing Engineers (SME). Hyatt Alicante, Anaheim, CA. (SME, One SME Dr., P.O. Box 930, Dearborn, MI 48121-0930).

(Feb. 18-20)—25th Annual International Waterborne, High-Solids, and Powder Coatings Symposium. Sponsored by the Southern Society and The University of Southern Mississippi (USM). New Orleans, LA. (Robson F. Storey or Shelby F. Thames, Co-Organizers, WHS&PC Symposium, Dept. of Polymer Science, USM, Box 10076, Hattiesburg, MS 39406-0076).

(Feb. 22-24)—"Color and Design: 21st Century Technology and Creativity." Williamsburg Conference sponsored by the Inter-Society Color Council. Williamsburg, VA. (Robert T. Marcus, ISCC Publicity Chairman, D&S Plastics International, 100 S. Mitchell Rd., Mansfield,

(Feb. 23-25)—"Basic Coatings for Sales, Marketing, and General Personnel." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (UMR Coatings Institute, 236 Schrenk Hall, 1870 Miner Circle, Rolla, MO 65409).

1998 - Africa

(Mar. 6-8)-"Coatings for Africa '98." Sponsored by The Oil & Colour Chemists'

Association's (OCCA) South African Division, the Natal Section, and Surfex Ltd. International Convention Centre, Durban, South Af-

rica. (Christopher Pacey-Day, OCCA, 967 Harrow Rd., Wembley, Middlesex, England HA0 2SF).



1997-Asia



(Oct. 22-24)—"New Developments in Colour Material Science and Technology." 70th

Anniversary Conference on Colour Materials Tokyo sponsored by the Japan Society of Colour Material. Arcadia Ichigaya (Shigaku

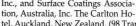


Kaikan), Tokyo, Japan. (Shuichi Hamada, Japan Society of Colour Material, Kitamura Bldg. 5F, 9-12, 2-chome, Iwamoto-cho, Chiyodaku, Tokyo 101, Japan).

(Nov. 3-5)—ChinaCoat '97. Sponsored by Paint & Coatings Chemical Industry Research Institute of the Ministry of Chemical Industry and Paint & Coatings Industry General Information Station of the Ministry of Chemical Industry. Shanghai International Exhibition Centre, Shanghai, PR. China. (Sinostar International Ltd., Unit 1501, Connaught Commercial Bldg., 185 Wanchai Rd., Wanchai, Hong

1998 – Australia









(Sept. 17-19)—"Crystal Growth of Organic Materials." Sponsored by University of Bremen. Bremen, Germany. (Dr. J. Ulrich, University of Bremen, Verfahrenstechnik/FB4, Postfach 330440, D-28334 Bremen, Germany).



(Sept. 23-24)—"Surcon '97." Sponsored by the Oil & Colour Chemists' Association (OCCA). Clarendon Suite, Birmingham, United Kingdom. (Yvonne Waterman, OCCA, Priory House, 967 Harrow Rd., Wembley, United Kingdom HA0 2SF).

(Sept. 23-25)—"Eurocoat '97." Organized by the Union of Associations. Parc des Expositions, Lyons-Eurexpo. (AFTPVA, 5, Rue Etex-75018 Paris, France).

(Sept. 23-25)—"High Temperature Surface Engineering." Conference sponsored by The Institute of Materials. Edinburgh Conference Centre, Edinburgh, Scotland. (Lisa Davies (C712), The Conference Dept., The Institute of Materials, 1 Carlton House Terrace, London, SW1Y 5DB, United Kingdom).

(Oct. 16-18)—"Polveri '97." Sponsored by ANVER (National Coating Association). Veronafiere, World Trade Center, Verona, Italy. (ANVER Palazzo Larice, Edifico M, Via Torri Bianche, 3, 20059 Vimercate MI).

(Oct. 27-29)—"Coating, Inks, and Adhesives for Plastics and Elastomers." Sponsored by The Paint Research Association. Milan, Italy. (Dip Dasgupta, Paint Research Association, 8 Waldegrave Rd., Teddington, Middlesex TW11 8LD United Kingdom).

(Dec. 4-5)—"Forum de la Connaissance." Sponsored by Association Française des Techniciens des Peintures, Vernis, Encres d'imprimerie, Colles et Adhésifs (AFTPVA). Maison de la Chimie, Paris, France. (AFTPVA, 5 rue Etex, F-75018, Paris, France).

1997 - South America

(Oct. 6-9)-"Latin-American Interfinish." Hotel Transamerica's Convention Center, São Paulo, Brazil. (Associação Brasileira de Tratamentos de Superfície, Av. Paulista, 1313, 9°a., conj. 913, CEP 01311-923 São Paulo, SP-Brazil).



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Advertisers' Index

Air Products & Chemicals Inc
Alnor Oil Co
Aqualon9
BASF Corp11
Borden Chemical Co
Burgess Pigment Co
Ciba Specialty Chemicals 12-13, Cover 4
Conn & Co
D/L Laboratories
Federation of Societies for Coatings TechnologyCover 2
FSCT Order Form99
Gamry Instruments, Inc

Halox Pigments
Henkel Corp29
Heubach17-18
Hüls America Inc20
The Lubrizol Corp1
Nissan Chemical Co14
Sartomer Co 15, 100
Specialty Minerals, Inc
Surface Coatings Association-New Zealand 75
Southwest Process Equipment 105
TeeMark Corp
Troy Corp 37
Western Equipment Co

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Humbug from Hillman

umbug welcomes Dr. Ray Dickie as Technical Editor of this esteemed Journal and congratulates him on his courage to meet the challenges posed by Humbug's backward philosophy which is based on a commitment to sluggish progress.

Humbug also wishes our recent past Technical Editor, Dr. Bob Brady, a good friend and faithful correspondent, much future success and happiness.

This month, I am pleased to welcome back to this page, Past-President Milt Glaser, who sent the following nonsense he found in the Chicago Bulletin of the ACS.

It notes: "It's truly astonishing what weird science our young scholars can create under the pressures of time and grades." (Right again-Ed.)

- —H₂O is hot water, and CO₂ is cold water.
- -To collect fumes of sulphur, hold a deacon over a
- -Water is composed of two gins. Oxygin and Hydrogin. Oxygin is pure gin. Hydrogin is water and gin.
- A super saturated solution is one that holds more than it can hold.
- -Magnet: something you find crawling over a dead cat.
 - –Vacuum: A large, empty space where the pope lives.
- -Artificial insemination is when the farmer does it to the cow instead of the bull.
- —A fossil is an extinct animal. The older it is, the more extinct it is.
- -To remove dust from the eye, pull the eye down over
- —For head colds: use an agonizer to spray the nose until it drops in your throat.
 - -Germinate: to become a naturalized German.

ick Kiefer says, "Here's one for the Northwestern Society that I received via E-mail": Four expectant fathers were in a Minneapolis hospital waiting room while their wives were in labor. The nurse comes in and tells the first man, "Congratulations! You're the father of twins."

"What a coincidence!" he replies. "I work for the Minnesota Twins baseball team.

The nurse returns a short time later and tells the second man, "You are the father of triplets." "Wow, what a coincidence, I work for the 3M Corporation," he replies.

At this point, the fourth guy faints. After he is revived, he says, "I work for Seven Up.

(Our humble apologies to all members of the Northwestern Society.)

s I promised last month, here is more from Joe Koleske's favorite columnist, Terry Marchal:

 An English teacher wrote the following sentence on the board:

"Woman without her man is a savage." She then instructed the class to punctuate the sentence. Most of the students wound up with:

"Woman, without her man, is a savage."

But one young woman wrote:

"Woman! Without her, man is a savage."

 A father pointed out an ant to his son and said, "There is a wonderful example for you in the life of an ant. Every day the ant goes to work and labors all day long. He never wastes time. He has a goal. He never wavers. That's

why he's successful."
"Yes sir," said the boy.
"Now with that example before you, what do you think happens to the person who applies himself with the same kind of drive."

"Someone steps on him?"

—Ted Brennan said he and his daughter were shopping in a department store and asked a clerk if a wristwatch on sale was electric.

"No," the clerk replied. "It works on a battery."

-Overheard in school: What was the reason for the Puritans coming to this country?

They came to this country so they could worship in their own way and make everyone else do the same.

-During the Civil War, a number of Confederate prisoners were held in a Western military post. It was new, clean and comfortable and probably the best place to be if you were a prisoner. Most rebel prisoners seemed to appreciate the place. But there was one fellow who was pretty unhappy with his captors. He constantly expressed his views to the Yankee soldiers. He railed about the battle of Chickamauga, where the Union forces had been

It got under the skin of the Union soldiers, and they complained to the Colonel who had the prisoner brought before him.

"All right," said the head of the post. "I want this constant belittling of the guards and the bantering about Chickamauga to stop. You've either got to take an oath of allegiance to the United States or we're going to ship you to one of those tough Northern prisons. What's it going to be?"

The Confederate prisoner thought for a minute and said, "I'll take the oath, Colonel."

The oath was administered. Then the repatriated citizen of the U.S. asked if he might say something. "Certainly," the Colonel said.

"Them Rebels sure did kick our butts at Chickamauga didn't they?"

> -Herb Hillman, Humbug's Nest, P.O. Box 135, Whitingham, VT 05361.



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