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A Review of Inverse
Gas Chromatography
Theory Used in the
Thermodynamic
Analysis of Pigment
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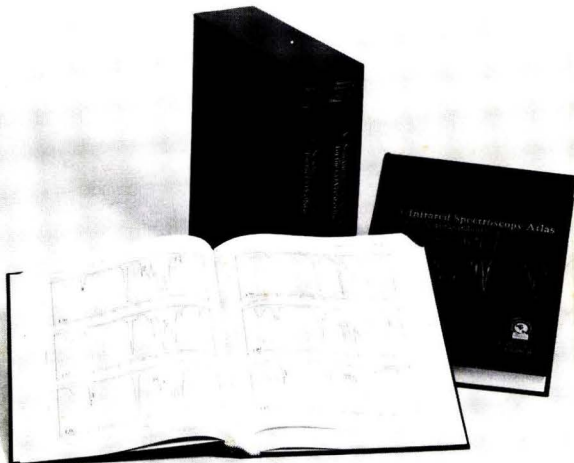
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GUIDE FOR AUTHORS

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 All manuscripts will be assumed to be previously unpublished writing of the authors, not under consideration for publication elsewhere.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

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

Papers in which proprietary products or processes are promoted for commercial purposes are specifically nonacceptable for publication.

SUBMISSION OF MANUSCRIPTS...

...for the Journal

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 *Back to Basics.*

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 the Editor at the address listed previously.

...for Roon Foundation Award Competition

Ten complete copies of the manuscript are required, and should be submitted to the Chairman of the 1993 Roon Awards Committee, Louis J. Sharp, Dexter Corp., 1 E. Water St., Waukegan, IL 60085. (For complete details, see "Roon Awards" section of the JOURNAL in January 1993 issue.)

MANUSCRIPT PREPARATION

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

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

Original Research Papers: The main technical content of the JOURNAL OF COATINGS TECHNOLOGY will continue to be original research papers. Editors support the trend in scientific writing to a direct, less formal style that permits limited use of personal pronouns to avoid repititious 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 solicted. Reviews that show how advances from other fields can beneficially be applied to coatings are also desired. Reviews that consist mainly of computer searches with little attempt to integrate or critically evaluate are not solicited.

Open Forum: Topics for this category may be nontechnical in nature, dealing with any aspect of the coatings industry. The subject may be approached informally. Editors encourage submission of manuscripts that constructively address industry prolbems and their solutions.

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

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

Manuscripts should be typed with double spacing on one side of $8^1/2$ 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.

Title

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

Authors' Biographies and Photographs

Give complete names, company or institutional affiliations, and brief biographical sketches of all authors. If available, submit a 5 x 7 inch (13 x 18 cm) black-and-white photograph with glossy or smooth high sheen surface, for each author. See later section on photographs for further details.

Abstracts

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

Text

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, EXPERIMENTAL, RESULTS AND DISCUSSION, and SUMMARY or CONCLUSIONS. Sub-headings will be specific to the subject.

Only as much review as is necessary should be given to provide 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 afterwords in parentheses. The ASTM Metric Practice Guide, E 380-72 (American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103) is a convenient reference.

Tables, Graphs, and Drawings

Tables, rather than descriptive text, should be used only when they are genuinely helpful. They should be proportioned in accordance with the height and width limitations of the JOURNAL'S pages. 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 blue-lined $8^1/2$ x 11 inch paper. Each graph should be drawn on a separate sheet, numbered, and the captions listed on a *copy* of the original graph. Graph captions and legends should also be typed on a separate sheet from original for typesetting.

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

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

Color prints and slides are unacceptable.

When illustrations are secured from an outside source, the source must be identified and the Editor assured that permission to reprint has been granted.

Nomenclature

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).

OTHER INFORMATION

Galley proofs will be sent to the author for checking about six weeks prior to publication.

Offprints may be purchased in quantities of 100 or more. Authors will receive price quotations. Each author will receive a complimentary copy of the JOURNAL issue in which his or her paper was published.

* * * *

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The Federation of Societies for Coatings Technology assumes no responsibility for the opinions by authors in this publication.

Comment

An Investment in the Future

Much has been written on the need to invest in our future through education. However, the time needed to make this investment pay off is sometimes viewed as too long by those who check the balance sheets and report the quarterly earnings. Thus, because it usually takes some time to recoup this investment and realize a gain, less and less money is being budgeted for employee education.

Therefore, it is very important to maximize the funds available and make the best use of the limited resources. One way to do this is to fully participate in the activities of the Federation of Societies for Coatings Technology and its 26 Constituent Societies. These activities are reasonably priced, offer a wide variety of educational opportunities, and cover all levels from beginner to advanced.

For starters, take a look at the offerings of your local Society. It has nine or ten technical meetings each year, and probably an additional seminar or a short course at a local college. Also, the Societies which offer special seminars also use extended mailing lists to promote the events, which means you are contacted by more than your own Society about such programs. These activities are in addition to the FSCT Annual Meeting and Paint Industries' Show and the yearly Spring Week Technical Seminar.

The Federation also offers a variety of written literature for your use. First and foremost of this literature is the JOURNAL OF COATINGS TECHNOLOGY, which offers a wide variety of data to assist your company's operation. The JCT is also a good source for learning about upcoming technical events. Also, the *Federation Series on Coatings Technology* is a must for every organization, because of its practical as well as theoretical approach to coatings. Right now there are 20 titles available, with additional ones being planned.

Just as important as the knowledge you gain from these offerings is the interaction you have with other attendees. The interchange among participants can usually bring about solutions to even the most stubborn problems, and these solutions will have a quick payback!

We send our children to school for at least 12 years with the hope of receiving a payoff in 10, 20, or 30 years. Don't we owe it to ourselves to invest in our future.

Michael G. Bell
Michael G. Bell
Director of Educational Services

Abstracts of Papers in This Issue

A Review of Inverse Gas Chromatography Theory Used in the Thermodynamic Analysis of Pigment and Polymer Surfaces—C.R. Hegedus and I.L. Kamel

JCT, 65, No. 820, 23 (May 1993)

Inverse gas chromatography offers the capability of analyzing filler particle and polymer surfaces, as well as the interactions which occur between these species in paints and other particulate-filled composites. This is accomplished by determining retention times of well characterized probe organic solvent molecules through columns packed with filler and polymer particles. Intermolecular interaction and gas chromatography theory are reviewed and equations are derived which allow the calculation of filler particle and polymer surface energy; free energy, enthalpy, and entropy of adsorption of polymers onto particles; and work of adhesion between these two constituents.

Thermodynamic Analysis of Pigment and Polymer Surfaces Using Inverse Gas Chromatography—C.R. Hegedus and I.L. Kamel

JCT, 65, No. 820, 31 (May 1993)

The thermodynamics of pigment particle surface-polymer interactions have been characterized using inverse gas chromatography (IGC). The use of IGC in this new application extends available analysis to the calculation of the thermodynamic properties of reversible particle-polymer interactions. This IGC technique was used to determine solid surface energies of particles and polymers; free energy, enthalpy, and entropy of adsorption; and work of adhesion. Available theories to calculate the nonpolar (dispersive) and polar contributions to these surface properties were reviewed and found applicable. The values obtained agree favorably with those obtained from alternate techniques. Nonpolar forces provided a large contribution to the free energy and enthalpy of adsorption. In addition, acid-base pairs were found to undergo favorable interactions while acid-acid and base-base pairs were less likely to undergo strong interactions. Amphoteric adsorbates underwent extensive interactions with both acids and bases due to their mutual compatibility and their tendency to self-associate. Entropies of adsorption for most interactions were found to be negative, indicating conformational changes from the gaseous state to a more restricted adsorbed configuration, similar to those in the condensation process. Entropy contributions to the total free en-

Une Revue de la Théorie de Chromatographie en Phase Gazeuse Inversée Utilisée pour l'Analyse Thermodynamique des Pigments et Surfaces Polymériques—C.R. Hegedus et I.L. Kamel

JCT, 65, No. 820, 23 (May 1993)

La chromatographie en phase gazeuse inversée offre la possibilité d'analyse des particules de remplissaage et des surfaces polymériques, en plus des interactions se produisant entre ces espèces dans les revêtements et autres composés. Ceci est accompli par la détermination des temps de rétention de sondes bien caractérisées où les molécules de solvants organiques passent à travers des colonnes contenant diverses particules polymériques. L'interaction intermoléculaire et la théorie de chromotographie en phase gazeuse sont étudiées. Des équations sont dérivées permettant le calcul d'énergie des particules de remplissage et des surfaces polymériques; énergie libre, enthalpie et entropie d'adsorption des polymères sur les particules; et l'adhésion entre ces composés.

Analyse Thermodynamique de Pigments et Surfaces Polymériques Utilisant la Chromotographie en Phase Gazeuse Inversée—C.R. Hegedus et I.L. Kamel

JCT, 65, No. 820, 31 (May 1993)

La thermodynamique des interactions entre les polymères et les surfaces de pigments a été caractérisée avec l'aide de la chromotographie en phase gazeuse inversée (IGC). L'utilisation de la technique IGC pour cette nouvelle application accroît l'analyse disponible par le calcul des propriétés thermodynamiques des interactions réversibles particule-polymère. Cette technique IGC a été utilisée pour déterminer l'énergie de surface de particules et polymères; énergie libre, enthalpie et entropie d'adsorption; et travail d'adhèsion. Les théories disponibles pour calculer les contributions polaires et non-polaires de ces propriétés de surface ont été revues et jugées applicables. Les valeurs obtenues s'accordent favorablement avec celles obtenues avec des techniques alternatives. Les forces non-polaires donnent une grande contribution à l'énergie libre et l'enthalpie d'adsorption. De plus, les paires acide-base ont donné des interactions favorables tandis que les paires acide-acide et base-base ont moins tendance à donner de fortes interactions. Les absorbants amphotériques ont donné de vastes interactions avec les acides et les bases à cause de leur compatibilité

Revision de la Teoria de Cromatografia de Gases Inversa Usada en el Analisis Termodinamico de las Superficies de Pigmentos y Polimeros—C.R. Hegedus and I.L. Kamel

JCT, 65, No. 820, 23 (May 1993)

La cromatografía de gases inversa ofrece la posibilidad de analizar la superficie de los polímeros y de una partícula de carga, así como las interacciones que se presentan entre estas especies en pinturas y otros materiales compuestos rellenos de partículas. Esto se lleva a cabo mediante la determinación de los tiempos de retención de las moléculas de solventes orgánicos, caracterizadas en columnas empacadas con partículas de polímeros y de carga. Se revisa la teoria de interacción molecular y la de cromatografia de gases, a la vez que se derivan las ecuaciones que permiten el cálculo de la energía de superficie del polímero y de la particula de carga, la energia libre, entalpia y entropia de adsorción del polímero sobre las partículas, y el trabajo de adhesión entre estos dos constituyentes.

Analisis Termodinamico de las Superficies del Polimero y el Pigmento, Usando Cromatografia de Gases Inversa—C.R. Hegedus and I.L. Kamel

JCT, 65, No. 820, 31 (May 1993)

Se caracterizaron las interacciones termodinámicas entre las superficies de pigmento y del polímero empleando cromatografia de gases inversa (IGC).

El uso de IGC en esta nueva aplicación extiende el análisis disponible al cálculo de las propiedades termodinámicas de las interacciones reversibles polímero-particula. Esta técnica IGC, se uso para determinar la energía de superficies sólidas de las particulas y los polímeros, la energia libre, entalpia y entropía de adsorcion, y el trabajo de adhesión.

Se revisaron las teorias disponibles para calcular las contribuciones polares y no polares (dispersivas) a estas propiedades de superficie y se encontró que fueron aplicables. Los valores obtenidos concuerdan favorablemente con los obtenidos con tecnicas alternas. Las fuerzas no polares proporcionaron una gran contribaución a la energia libre y a la entalpia de adsorción. Además, se encontró que los pares ácido-base experimentan interacciones favorables, montras que fué menos probable que los panes ácido-ácido y base-base experimentaran interacciones fuertes. Los adsorbentes anfotéricos experimentaron extensas interacciones tanto con los ácidos

ergy change were approximately 30% of the enthalpy changes.

Suitability of ASTM Test Method D 2369 for Determination of VOC in High Solids Coatings—Philadelphia Society for Coatings Technology Technical Committee

JCT, 65, No. 820, 45 (May 1993)

The Technical Committee of the Philadelphia Society for Coatings Technology conducted a "round-robin" study of ASTM D 2369-89, the method used to determine volatile organic content (VOC) in coatings. Initially seven laboratories undertook the testing of five coatings. Two of these coatings were single-component and the remaining three were two-component, high solids coatings.

An analysis of variance was performed on the data obtained. The results indicated that the procedure can produce reliable results, when single-component coatings are tested. However, data from two-component coatings yielded higher results than expected with large standard deviations.

New Oxazolidine-Based Moisture Scavenger for Polyurethane Coating Systems—G.N. Robinson, J.F. Alderman, and T.L. Johnson

JCT, 65, No. 820, 51 (May 1993)

A research program was initiated to determine whether an oxazolidine moisture scavenger could overcome the inherent disadvantages of physical and chemical moisture scavengers commonly employed in polyurethane coating systems. Oxazolidines were selected for this study because of their potential reactivity with water and generally good toxicity characteristics. Commercially available oxazolidines are unacceptable due to their high molecular weight and slow reactivity. A wide range of oxazolidine molecules were investigated. Oxazolidines from amino alcohol/ketone reactions demonstrated excellent hydrolysis characteristics. Ketone-based oxazolidines were then shown to effectively reduce moisture in single-package, moisture-cured, pigmented polyurethane coatings. Good accelerated viscosity stability was also observed. Polyurethane coating systems can be produced using a ketone-based oxazolidine as a moisture scavenger. Such a product might find utility as a drying agent for wet polyols, solvents, pigments, or fillers used in one- or two-component systems where moisture is undesirable.

Preparation and Properties of Blocked Dimethyl m-Isopropenyl Benzyl Isocyanate for One-Component Coatings—H.R. Lucas and K-J. Wu

JCT, 65, No. 820, 59 (May 1993)

A unique characteristic of dimethyl m-isopropenyl benzyl isocyanate is the flex-

La Convenance de la Méthode Analytique ASTM D 2369 pour la Détermination de COV dans les Revêtements à Haute Teneur en Matières Solides—Philadelphia Society for Coatings Technology

JCT, 65, No. 820, 45 (May 1993)

Le comité technique de la société de Philadelphie a mené une étude pour étudier la méthode analytique ASTM D 2369-89 utilisée pour la détermination de composés organiques volatiles dans les revêtements. Initialement, sept laboratoires ont testé cinq revêtements. Deux de ces revêtements étaient à un composé et le reste trois ou quatre composants.

Une analyse de la variance des données a été obtenue. Les résultats indiquent que la procédure peut produire des résultats fiables, quand les revêtements à un composé sont testés. Par contre, les données des revêtements à deux composants ont donné de meilleurs résultats avec une déviation standard plus grande.

Nouvel Extracteur d'Humidité à Base d'Oxazolidine pour les Revêtements de Polyuréthane—G.N. Robinson, J.F. Alderman, et T.L. Johnson

JCT, 65, No. 820, 51 (May 1993)

Un programme de recherche a été initié pour déterminer si un extracteur d'humidité à base d'oxazolidine pourrait résoudre les désavantages inhérents des extracteurs physiques et chimiques d'humidité communément employés dans des revêtements de polyuréthane. Les oxazolidines ont été sélectionné pour cette étude à cause de leur réactivité potentielle avec l'eau et leurs bonnes caractéristiques générales de toxicité. Les oxazolidines disponibles commercialement sont inacceptables à cause de leur haut poids moléculaire et leur lente réactivité. Une large gamme de molécules oxazolidine a été étudiée. Les oxazolidines de réactions amine-alcool/cétone ont démontré d'excellentes caractéristiques d'hydrolyse. Les oxazolidines à base de cétone ont réduit de façon efficace l'humidité de revêtements de polyuréthane à un composé. Une bonne stabilité de viscosité a également été observée. Les revêtements de polyuréthanes peuvent être fabriqués utilisant un oxazolidine à base de cétone comme extracteur d'humidité. Un tel produit pourrait être utile comme agent siccatif pour les polyols humides, solvants, ou matières de charges utilisées dans des systèmes à un

La Conveniencia del Metodo de Prueba ASTM D 2369 Para la Determinacion de VOC en Recubrimientos de Altos Solidos—Philadelphia Society for Coatings Technology Technical Committee

JCT, 65, No. 820, 45 (May 1993)

El comité técnico de la sociedad de Filadelfia condujo un estudio del método de prueba ASTM D 2369-89, que es el método para determinar el contenido de orgánicos volátiles en los recubrimientos. Inicialmente, fueron siete laboratorios los que tomaron la tarea de probar cinco recubrimientos de altos solidos. Dos de estos recubrimientos fueron de un solo componente y los tres restantes fueron de dos componentes.

Con los datos obtenidos se hizo un análisis de la desviación estándar. Los resultados indicaron que el procedimiento puede producir resultados confiables, cuando se evaluan recubrimientos de un solo componente. Sin embargo, los datos de recubrimientos de dos componentes, generaron resultados mas altos que los esperados con desviaciones estándares grandes.

Nuevo Removedor de Humedao Basado en Oxazolidina Para Sistemas de Recubrimientos De Poliuretano—G.N. Robinson, J.F. Aldeman, and T.L. Johnson

JCT, 65, No. 820, 51 (May 1993)

Se inició un programa para determinar si un removedor de humedad de oxazolidina podia superar las desventajas inherentes, físicas y químicas, de los removedores de humedad, empleados comúnmente en los sistemas de recubrimientos de poliuretano. Para este estudio de, selecoionaron las oxazolidinas debido a su reactividad potencial con el agua y sus buenas caracteristicas de toxicidad. Las oxazolidinas disponibles comercialmente sun inaceptables por su elevado peso molecular y su lenta reactividad. Se investigó un amplio rango de moléculas de oxazolidina. Las oxazolidinas generadas a partir de reacciones alcohol amino/cetonas demostraron excelentes caracteristicas de hidrólisis. Las oxazolidinas basadas en cetonas demostraron que reducen, de manera efectiva, la humedad en recubrimientos de poliuretano pigmentados de un componente, curados por humedad. También se observó buena aceleración en

como con las bases debido a su mutua compatibilidad y a su tendencia a la autoasociación. Se encontró que las entropias de adsorción para muchas interacciones fueron negativas, indicando cambios conformacionales a partir del estado gaseoso a una configuración absorbida mas resstringida, similar a aquellas encontradas en el proceso de condensación. Las contribuciones de entropia al cambio total de la energia libre fueron aproximadamente del 30% de los cambios de la entaipia.

mutuelle et leur tendance à s'associer à euxmêmes. Les entropies d'adsorption pour la plupart des interactions ont été négatives, indiquant des changements conformationels de l'état gazeux à la configuration plus restreinte d'adsorption, similaire à celle du procédé de condensation. Les contributions entropiques au changement de l'énergie libre totale étaient approximativement 30% des changements d'enthalpie.

ibility it offers to the coatings industry. Co-polymerization through its double bond with a variety of acrylic monomers offers the chemist broad possibilities in polymer design. By blocking the isocyanate moiety of the monomer, one can theoretically incorporate a hydroxyl functional acrylic monomer within the same polymer.

Aimed at the automotive's OEM, industrial, and coil coating applications, the coatings are true one-component systems. However, preparation of these polymers are not without difficulties. A methyl ethyl ketoximine blocked monomer is an ideal candidate in a one-component coating system, with onset deblocking at 50°C. However, the unsaturated isocyanate as well as its blocked monomer requires a minimum polymerization of 80°C for complete conversion of monomer to polymer. With onset deblocking of the isocyanate moiety at 50°C, premature crosslinking takes place with the acrylic hydroxyls located in the growing polymer chains before polymerization is complete. To prevent this, we have discovered that certain solvents used in the polymerization prevent this premature gelation without affecting the coatings cure response and properties.

ou deux composants, où l'humidité est indésirable.

Préparation et Propriétés d'Isocyanate de Diméthyl m-Isopropenyl Benzyl Encombré pour les Revêtements à un Composé—H.R. Lucas et K-J. Wu

JCT, 65, No. 820, 59 (May 1993)

Une caractéristique unique de l'isocyanate de diméthyl m-isopropenyl benzyl est la flexibilité qu'il offre à l'industrie des revêtements. La copolymérisation avec l'aide de sa double liaison avec une gamme de monomères acryliques, offre de vastes possibilités aux chimistes pour le design de polymère. En encombrant le monomère de l'isocyanate, on peut théoriquement incorporer un monomère de l'isocyanate, on peut théoriquement incorporer un monomère acrylique avec une fontionnalité hydroxy à l'intérieur du même polymère.

Développés pour les applications industrielles, automobiles et revêtement en continu, ces revêtements sont de vrais systèmes à un composé. Par contre, la préparation de ces polymères n'est pas sans difficultés. Un monomère encombré de méthyl éthyl kétoxime est un candidat idéal pour un système de revêtement à un composé, avec désencombrement à 50°C. Par contre, l'isocyanate non-saturé et son monomère encombré demande un minimum de polymérisation à 80°C pour une conversion complète du monomère en polymère. Avec le désencombrement de l'isocyanate à 50°C, une réticulation prématurée prend place avec l'hydrolyse de l'acrylique situé dans les chaînes de polymères grandissantes avant que la polymérisatioon ne soit complète. Afin de prévenir ceci, nous avons découvert que certains solvants utilisés dans la polymérisation empêchent la gélification prématurée sans affecter les propriétés des revêtements.

la estabilizacion de la visosidad. Los sistemas de recubrimientos de poliuretano se pueden producir usando una oxazolidina como removedor de humedad. Un producto como este puede encontrar utilidad como agente secante para polioles humedos, pigmentos o cargas usadas en sistemas de uno o dos componentes, en los cuales is humedad es indeseable.

Preparacion y Propiedades del Dimetil m-Isopropenil Bencil Isocianato Bloqueado Para Recubrimientos de un Solo Componente—H.R. Lucas and K-J. Wu

JCT, 65, NO. 820, 59 (May 1993)

La caracteristica principal del dimetil m-isopropenil bencil isocianato es la flexibilidad que ofrece a los recubrimientos. La polimización a través de su doble enlace con una variedad de monómeros acrílicos ofrece, a los químicos, amplias posibilidades en el diseño de polimeros. Mediante el bioqueo de la funcionalidad isocianato del monómero, se puede incorporar teóricamente un monómero acrilito hidroxy funcional dentro del mismo polímero.

Los recubrimientos dirigidos a aplicaciones de OEM automotrices, industriales y lámina en rollo (COI coating), son realmente sistemas de un solo componente. Sin embargo la preparación de estos polímeros presenta dificultades. Un monómero de metil etil cetoximina bioqueado es un candidato ideal en un sistema de recubrimiento de un solo componente, con la desincorporación del bloqueo a 50°C. Sin embargo, el isocianato insaturado, asi como su monómero bloqueado, requiere una temperatura de polimerización minima de 80°C para la conversión completa del monómero en polímero. Con la desincorporación del bolqueo de la moltad de isocianato a 50°C, tiene lugar una reticulación prematura con los bidroxilos acrílicos localizados en la cadena creciente del polímero, antos de quo la polimerización se complete. Para prevenir esto, se ha descubierto que ciertos solventes usados en la polimerización previenen esta gelación prematura sin afectar la respuesta, de estos recubrimientos, al curado ni a sus propiedades.

Federation of Societies for Coatings Technology

1993 Annual Meeting &
Paint Industries' Show

Hotel Information/Reservation Application
◆Advance Registration Form ◆
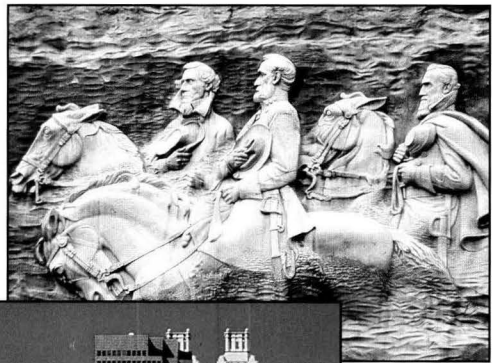
"Today's Competitive Coatings: Lean, Mean and Green"

Georgia World Congress Center
Wednesday, Thursday, Friday ◆ October 27-28-29, 1993
Atlanta, Georgia

From the President:

The whole world is coming to Atlanta! We expect that representatives from 60 countries will be in attendance at the Federation's 71st Annual Meeting & 58th Paint Industries' Show. It is recognized internationally as the coatings industry's premier showcase for both Raw Material and Equipment Suppliers and the end users. In addition, the technical sessions are unrivaled in quality and variety. And if that isn't enough, Atlanta's mild climate, ambiance and southern hospitality will delight you— not to mention its wonderful array of restaurants and stores.
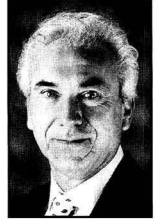
Together, the combination is unbeatable! Take a few minutes to review this outstanding program and make your reservations early.

Looking forward to seeing y'all in Atlanta!

Colin D. Penny
President, FSCT

"Today's Competitive Coatings: Lean, Mean and Green"

The basis for this year's Annual Meeting Technical Program theme is the understanding that success in the coatings industry both now and in the future belongs to those who take the lead in blending long-term profitability, coatings performance and environmental protection.

The theme recognizes these goals in the terms:
　　LEAN, as in cost effective; process efficient; and waste free
　　MEAN, by being consistent; tough; durable; and resistant; and
　　GREEN, through being environmentally friendly; renewable; recyclable; and safe.

The following program session themes are being developed:
- Advanced Topics in Coatings Research
- Environmental Affairs
- Latex Technology
- Lean, Mean, Green and CLEAN — Innovative Equipment Cleaning Techniques
- Environmentally Compatible Corrosion Inhibitive Coatings
- Radiation Curing

- Characterization of Physical Properties of Coatings
- Roon Award Competition Papers
- International Papers
- APJ/Voss Award (Society) Competition Papers

Papers presented at the event will cover the previously mentioned topics and also include the following:
- Powder Coatings
- Adhesion
- Organic Pigments
- UV-Curable Coatings
- Low VOC Coatings
- Formulating
- High-Solids Coatings
- Additives

The Keynote Address will be delivered on Wednesday morning to kick off the event. Highlighting the technical program will be the Mattiello Memorial Lecture, scheduled for Friday morning.

A Poster Session, featuring non-commercial work covering new ideas and techniques in coatings research, will be held all day Thursday.

World's Premier Paint Show to Feature Products of Over 267 Companies

The largest coatings-related exhibition in the world— the FSCT Paint Industries' Show—will be held in conjunction with the Annual Meeting at the Georgia World Congress Center. Over 267 supplier companies to the coatings manufacturing industry will be present to discuss their newest products and services. In over 90,000 sq. ft. of exhibits will be displayed a wide variety of raw materials, production equipment, containers and filling equipment, laboratory apparatus, and testing devices for the paint and coatings producer.

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

Exhibit hours will be:
Wednesday, October 27 .... 12:00 Noon-5:00 p.m.
Thursday, October 28 .... 9:00 a.m.-5:00 p.m.
Friday, October 29 .... 9:00 a.m.-12:00 Noon

Hotel Reservation Instructions

Whether you wish the value of a moderately priced hotel or the luxury of an upscale property, the choice is yours. The FSCT has arranged for convention rates at eight official Paint Show hotels. To obtain these rates, requests for accommodations must be placed through the FSCT Housing Bureau of the Atlanta Convention and Visitors Bureau, using the FSCT Housing Form. The reservation cut-off date is **September 24.**

Send your form to the FSCT Housing Bureau for processing. Be sure to include a phone number and keep a copy of the form for your records. Phone calls and faxes will not be accepted. The Housing Bureau will mail an acknowledgment of the reservation and the hotel assigned will then forward a confirmation. Allow two weeks for receipt of acknowledgment.

All hotels require a room deposit or credit card guarantee. If a credit card number has been given, a deposit is not required. Otherwise, please send the deposit amount indicated on the hotel confirmation **directly** to the hotel within two weeks of receipt of confirmation.

International attendees only may fax their request on the official housing form to (404) 521-6392.

All changes/cancellations must be placed in writing to the Housing Bureau. After September 24, changes and cancellations must be made directly with the hotel.

Participating Hotels

Atlanta Marriott Marquis
(FSCT Headquarters)

The Marriott Marquis, located in the city's Peachtree Center, features a 50-story interior atrium and glass-enclosed bubble elevators as well as an abundance of greenery and artwork. The guest rooms offer climate control, AM/FM radios, color TVs with in-room movies and sports, in-room video check-out, and a separate desk area. Concierge level guest rooms are also available. The hotel operates ten restaurants and lounges, an indoor/outdoor pool, a health club, a sauna and a shopping level.

(404) 521-0000
(Requests for rooms limited to 10 per company.)

Hyatt Regency Atlanta

This 23-story atrium hotel includes luxury guest rooms and suites, a landscaped outdoor pool, a whirlpool, and a fully equipped health and fitness center. The Hyatt is located in Peachtree Center, just across the street from the FSCT headquarters hotel. Available are three restaurants, including Polaris,

a 327-foot high revolving rooftop restaurant accessible by a glass elevator.

(404) 577-1234

Westin Peachtree Plaza

The Westin Peachtree Plaza is America's tallest hotel, boasting 73 stories, three restaurants, four lounges, an indoor/outdoor pool, 24-hour room service and a fully-equipped health club.

(404) 659-1400

Atlanta Hilton and Towers

Eight restaurants and lounges, including the four-star "Nikolai's Roof," are housed by this hotel, which also provides a fully equipped health club, tennis courts, and jogging trail. (NPCA headquarters)

(404) 659-2000

The Omni Hotel at CNN Center

The Omni is located in downtown Atlanta, adjacent to the Omni Coliseum, the Georgia World Congress Center and the Georgia Dome. The rooms and suites feature in-room movies, complimentary HBO, hair dryers, irons and ironing boards. Eighteen-hour room service is available, Bugatti for fine Northern Italian dining, and The Cafe for traditional American fare. The Take Two Lobby Lounge offers entertainment. Health Club facilities are also available.

(404) 659-0000
(Requests for rooms limited to 10 per company.)

The Radisson Hotel Atlanta

Guest services at this hotel include dry cleaning and laundry services, a hair salon, and gift shop. The garden setting pool, surrounded by trees, flowers, and a fountain, is covered by a sun roof. A restaurant, lounge, and fitness club are also available in the hotel.

(404) 659-6500

Ritz-Carlton Atlanta

The only four-star, four-diamond downtown property features a small, intimate club-like atmosphere. Award-winning cuisine is served in four restaurants and lounges. A health club facility is available.

(404) 659-0400

The Comfort Inn

The convenient location of the Comfort Inn places guests two blocks from the Georgia World Congress Center, Peachtree Center, the CNN Center, and the MARTA Rapid Transit System. The hotel offers spacious guest rooms and suites, a restaurant and lounge, and a courtyard/pool reception area.

(404) 524-5555
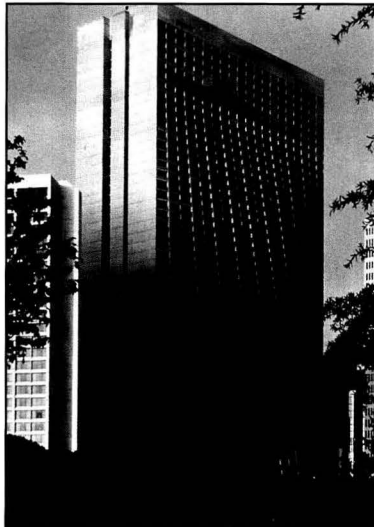
The Marriott Marquis—FSCT Headquarters Hotel

Hotel Room and Suite Rates

Property	Single	Double	1 BR Suite	2 BR Suite
Marriott Marquis*	$131	$146	$450-900	$550-1050
Concierge Level	$175	$175		
Hyatt Regency	$128	$143	$325-800	$400-875
Westin Peachtree	$120	$135	$350-1150	$500-1350
Atlanta Hilton & Towers	$122	$135		
Omni Hotel at CNN Center*	$139	$154	$650-800	$800-950
Radisson Hotel	$ 99	$115	$260-480	$365-585
Ritz-Carlton	$140	$155	$385-985	$580-1165
Comfort Inn	$ 89	$ 99		

*A limit of 10 rooms per company will be placed at the Marriott and also at the Omni Hotel. A parlor counts as one room.

Shuttle Service

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

Route 1

Marriott Marquis ....Courtland Street Entrance
Atlanta Hilton ....Courtland Street @ Marquis
Comfort Inn ....International Blvd.

Route 2

Westin Peachtree .... Spring Street - Motor Lobby Entrance
Hyatt Regency .... Harris Street - Clock of Five Entrance

Route 3

Ritz-Carlton ....Ellis Street
Radisson ....International Blvd. - Ballroom Entrance

Hours of Operation

Tuesday, October 26—
8:00 a.m. - 6:00 p.m.

Wednesday, October 27—
7:30 a.m. - 6:00 p.m.

Thursday, October 28—
7:30 a.m. - 6:00 p.m.

Friday, October 29—
7:30 a.m. - 3:00 p.m.

Legend

1—Georgia World Congress Center
2—Marriott Marquis
3—Atlanta Hilton & Towers
4—Atlanta Hyatt Regency
5—Omni at CNN Center
6—Radisson
7—Ritz-Carlton
8—Westin Peachtree
9—Comfort Inn

1993 FSCT Annual Meeting and Paint Industries' Show
Georgia World Congress Center, Atlanta, Georgia
Wednesday, Thursday, and Friday, October 27-29, 1993

HOTEL RESERVATION FORM

MAIL FORM & DEPOSIT TO:

FSCT Paint Show
Housing Bureau
233 Peachtree Street, NE
Suite 2000
Atlanta, GA 30303

Georgia World Congress Center
Atlanta, GA
October 27-28-29, 1993

HOTEL PREFERENCE

List the three hotels of your preference. First choice will be assigned if available. List other preferences to avoid delay. Requests for the Atlanta Marriott Marquis and the Omni Hotel will be limited to 10 rooms per company. **The deadline is September 24.**

(1) _____ (2) _____ (3) _____

RESERVATION REQUEST

Names of Occupants	Room Type	Rate	Arrival	Departure

For additional reservations, feel free to copy this form.

ROOM GUARANTEE

All hotels require a room deposit or credit card guarantee. List your credit card below or send a check directly to the hotel after you receive the hotel's confirmation.

CREDIT CARD (circle type): American Express, MasterCard, or VISA

☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐   ☐☐ ☐☐   _____
(Number)   (Expiration Date)   (Signature)

MAIL ALL CONFIRMATIONS TO:

Name: _____   Telephone: _____

Company: _____   FAX: _____

Address: _____

City/State (Province): _____   Zip Code (Mailing Code): _____

Country (if other than U.S.): _____

_____ has special needs: _____
(name of occupant)   (please specify)

Registration Information

The Advance Registration fees include three days of admission to the Paint Industries' Show and the Opening Session, and all concurrent program sessions of the Annual Meeting. To register, simply complete the Advance Registration Form and forward it with payment to FSCT. The Advance Registration prices will be available until **October 1**. The form with accompanying payment must be received at FSCT by October 1. Registration options are listed below:

Full Time	Advance	On-Site
Member	$ 75	$ 90
Non-member	$100	$125
Guest	$ 60	$ 70

Method of Payment

Acceptable types of payments include checks in U.S. funds made payable to FSCT, as well as credit cards: VISA, MasterCard, and American Express. Payment is due with registration form. Forms received without payment will not be processed. Please note: **if payment is made with a credit card, the form may be faxed to FSCT Headquarters Office. If faxing, please do not mail original form.**

The Registration Services Area will be located at the Georgia World Congress Center and will be open over the following days and times for you to pick up your badge and registration materials.

Day	Time
Tues., Oct. 26	8:00 a.m. - 5:00 p.m.
Wed.-Thurs., Oct. 27-28	7:30 a.m. - 5:00 p.m.
Fri., Oct. 29	7:30 a.m. - 12 Noon

On-site registration will also be available during the times noted above.

Cancellation and Refund Policy

All cancellations must be submitted in writing to the FSCT Headquarters Office. Cancellations received by October 15 will be subject to a $10 handling charge. A $25 charge will apply after that date. All cash refunds will be processed after November 1. Credit card cancellations received after October 1 will not be processed until after November 1.

Airport and City Transportation*

From Hartsfield Atlanta International Airport:

Airport Shuttle (service to downtown hotels, provided by Atlanta Airport Shuttle) is $8.00 one way, $14.00 round trip. The trip takes approximately 30 minutes.

Taxi Service from the airport to downtown hotels is $15.00 for one person, $8.00 per person with two passengers, and $6.00 per person with three passengers in the taxi.

Public Transportation from the airport to the downtown area nearby the official hotels is available via MARTA (rapid transit). The fee is $1.00 and the ride takes approximately 35 minutes.

* Prices are subject to change.

Airline Information

Convention discounts of up to 10% on domestic flights are available from Delta and Continental Air Lines.* Continental is also offering convention discounts on international flights. To make your transportation arrangements to the Paint Show, you may use the Federation's Official Travel Agency or contact the airlines directly.

FSCT Travel Desk 1-800-448-FSCT
Delta .... 1-800-241-6760 (ref. file # **U0235**)
Continental .... 1-800-468-7022 (ref. file # **ZJA35**)
*For each airline, certain restrictions apply.

Guest Program

Guest Activities begin on Wednesday, October 27, with a Welcome Social at the Marriott Marquis.

On Thursday, following a continental breakfast in the Marriott Marquis, registered guests will depart on deluxe motorcoaches for a visit to Stone Mountain Park which features the world's largest mass of exposed granite. There, guests will stop at Memorial Hall for a view of the Confederate Memorial—a carving in the granite of Robert E. Lee, Stonewall Jackson, and Jefferson Davis. The carving, even bigger than Mount Rushmore's, is the largest work of sculptured art in existence. Guests also will have the opportunity to tour the park's authentic antebellum plantation featuring 20 structures dating between 1780 and 1880.

From Stone Mountain, guests will board the motorcoaches for the Georgia Freight Depot, a historical building in Atlanta featured in scenes from the movie "Gone with the Wind." There, a delicious lunch will be served.

After lunch, guests will visit Underground Atlanta, a unique underground shopping district boasting retail shops and restaurants.

Making Your Arrangements

(1) To place airline reservations, call the FSCT Travel Desk at **1-800-448-FSCT** or (215) 628-2549 and mention **Paint Show '93**. To contact the carriers directly, call the following numbers and reference the file numbers provided:

Delta 1-800-241-6760 (ref. file # U0235)
Continental 1-800-468-7022 (ref. file # ZJA35)

(2) To make hotel reservations, mail your Hotel Reservations Form to the FSCT Housing Bureau. The cut-off date for reservations is **September 24**.

(3) Register in advance for the Annual Meeting and Paint Industries' Show by filling out the Registration Form and forwarding it to FSCT with payment. The deadline for Advance Registration is **October 1.**

(4) To register a spouse or guest, fill out the Guest portion of the Registration Form and provide the proper payment.

(5) Dates to keep in mind:

October 25-27—NPCA Annual Meeting, Hilton Hotel and Towers, Atlanta.

October 26—FSCT Board of Directors Meeting, 9:00 a.m., Marriott Marquis Hotel.

October 27-29—FSCT Annual Meeting & Paint Industries' Show, Georgia World Congress Center.

1993 Advance Registration

FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY
ANNUAL MEETING & PAINT INDUSTRIES' SHOW
ATLANTA, GA • OCTOBER 27-29, 1993

BADGE INFORMATION: This is how your badge will read. Please print clearly!

NICKNAME

FIRST NAME   LAST NAME

COMPANY

STREET (or P.O. Box)

CITY   STATE/PROV.

COUNTRY (OTHER THAN U.S.)   POSTAL CODE

TELEPHONE NO.   FAX NO.

GUEST BADGE INFORMATION:

NICKNAME

FIRST NAME   LAST NAME

CITY   STATE/PROV.

POSTAL CODE

BUSINESS CLASSIFICATION DATA FOR INDUSTRY REGISTRANT:

YOUR COMPANY (CHECK ONE ONLY)

AA ❏ Manufacturers of Paints, Varnishes, Lacquers, Printing Inks, Sealants, Adhesives
BB ❏ Manufacturers of Raw Materials
CC ❏ Manufacturers of Equipment and Containers
DD ❏ Sales Agents for Raw Materials and Equipment
EE ❏ Government Agency
FF ❏ Research/Testing/Consulting
GG ❏ Educational Institution/Library
HH ❏ Paint Consumer
YY ❏ Environmental Services
JJ ❏ Other

YOUR POSITION (CHECK ONE ONLY)

KK ❏ Management/Adm.
LL ❏ Mfg. & Engineering
MM ❏ Quality Control
NN ❏ Research & Development
PP ❏ Technical Sales Service
QQ ❏ Sales & Marketing
RR ❏ Consultant
SS ❏ Educator/Student/Librarian
TT ❏ Other

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

_____

Registration Fees:

A ❏	FSCT Member	$75.00
B ❏	Non-Member	$100.00
D ❏	Guest	$60.00
G ❏	Special Fee for Retired Members	$25.00
H ❏	Special Fee for Spouse of Retired Members	$25.00

Luncheon Tickets:

❏ NUMBER REQUIRED: _____
$25.00 EACH.

Method of Payment:

Payment is due with registration form.

Total Amount Due $ _____

Please Check One:

❏ Enclosed is Check # _____
payable in U.S. Funds to FSCT

❏ Charge to the following card:

___MC   ___VISA   ___AMEX

Card No.

_____

Expiration Date: _____ Mo. _____ Yr.

_____
Signature (Credit card registrations only)

_____
Please print cardholder's name

DEADLINE DATE FOR ADVANCE REGISTRATION IS **OCTOBER 1**. NONE WILL BE ACCEPTED AFTER THAT DATE. If paying by check, please fill out form and mail with a check in the correct amount (made payable to the FSCT) **to the Federation address shown below**. All checks must be payable in U.S. Funds. Any that are not will be returned. We are pleased to accept faxes on credit card charges only. *A $10.00 charge will be made for cancellations received prior to October 15. A $25.00 charge will be made for cancellations received after that date.*

492 Norristown Rd., Blue Bell, PA 19422-2350
Phone: 215-940-0777 • FAX: 215-940-0292

1993 PAINT INDUSTRIES' SHOW

Current List of Exhibitors

Aceto Corp.
Advanced Coating Technologies
Advanced Software Designs
Air Products & Chemicals, Inc.
Alcan-Toyo America, Inc.
Alcoa Industrial Chemicals
Allied Signal Corp.
Alt-Chem International
American Felt & Filter Co.
American Paint Journal Co.
Amoco Chemical Co.
ANGUS Chemical Co.
Anker Labelers USA, Inc.
Aqualon Co.
Arco Chemical Co.
A.R. Arena Products, Inc.
Atlas Electric Devices Co.
Ashland Chemical Co., IC&S Div.
Automated Filling Spec. Corp.
Aztec Catalyst Co.

B.A.G. Corp.
BASF Corp.
Blackmer Pump
Bohlin Instruments, Inc.
Brinkmann Instruments, Inc.
Brookfield Engineering Labs, Inc.
Brookhaven Instruments Corp.
Buckman Laboratories
Buhler Inc.
Bulk Lift International
Burgess Pigment Co.
BYK-Chemie USA
BYK-Gardner, Inc.

C&E News/ACS
Cabot Corp., Cab-O-Sil & Special
   Blacks Div.
Caframo Ltd.
Calgon Corp.
Cappelle Inc.
The Carborundum Co., Fibers Div.
Cardolite Corp.
Cargill, Inc.
Carri-Med Americas, Inc.
Carroll Scientific, Inc.
CB Mills, Div. Chicago Boiler Co.
CCP Polymers
Celite Corp.
Chemical Marketing Reporter
Chemical Week Associates
CIBA-GEIGY Corp.
The Coatings Laboratory Inc.
Coatings Magazine
Color Corp. of America
Colorgen, Inc.
Colortec Associates Inc.
Columbian Chemicals Co.
Compliance Services, Inc.
Consolidated Research, Inc.
Cookson Pigments Inc.
Coulter Corp.
CPI Purchasing Magazine
CPS Chemical Co., Inc.
CR Minerals Corp.
Cray Valley Products
Crosfield Co.
Cuno, Inc., Process Filtration Prods.
CYDEC Inc.
CYTEC Industries

D/L Laboratories
Daniel Products Co., Inc.
Datacolor International

Day-Glo Color Corp.
DeFelsko Corp.
Degussa Corp.
University of Detroit-Mercy
Distil-Kleen, Inc.
Dominion Colour Corp.
Dow Chemical
Dow Corning Corp.
Draiswerke, Inc.
Drew Industrial
Dry Branch Kaolin Co.
DSA Consulting, Inc.

Eagle Zinc Co.
Eastern Michigan University
Eastman Chemical Co.
Ebonex Corp.
ECC International
Eiger Machinery, Inc.
Elektro-Physik USA, Inc.
Elf Atochem
Elmar Worldwide
EM Industries, Inc.
Engelhard Corp.
Epworth Mfg. Co., Inc.
Etna Products, Inc., Specialty
   Chemical Div.
Exxon Chemical Co.

Fawcett Co., Inc.
Federation of Societies for Coatings
   Technology
Filter Specialists, Inc.
Fischer Technology Inc.
Fluid Management
FMC Div., Pharm. & Bio. Div.
FMJ International Publications Ltd.
H.B. Fuller Co.

Paul N. Gardner Co., Inc.
The BFGoodrich Co., Specialty
   Polymers & Chemicals Div.
The Goodyear Tire & Rubber Co.,
   Chemical Div.
Grace Davison (W.R. Grace & Co.)
Guertin Polymers

Haake Inc.
Halox Pigments
Harcros Pigments, Inc.
R.E. Hart Labs Inc.
Henkel Corp., Coatings & Inks Div.
Heraeus DSET Laboratories, Inc.
Heucotech Ltd.
Hilton Davis Co.
Hockmeyer Equipment Co.
Hoechst Celanese Corp., Pigments
Hoechst Celanese Corp., Waxes,
   Lubricants & Polymers
Horiba Instruments Inc.
J.M. Huber Corp.
Hüls America Inc.
Hunter Associates Laboratory, Inc.

Ideal Manufacturing & Sales Corp.
Industrial Finishing Magazine
International Compliance Center
International Resources, Inc.
International Specialty Products
ISP Filters, Inc.
ITT Marlow/ITT A-C Pump

SC Johnson Polymer
Journal of Coatings Technology

K-T Feldspar Corp.
Kenrich Petrochemicals, Inc.
Kinetic Dispersion Corp.
Kemira, Inc.
King Industries, Inc.
KRONOS, Inc.
KTA-Tator, Inc.

Labsphere, Inc.
Laidlaw Environ. Services, Inc.
LaQue Center for Corrosion Tech.
Lawter International
Leeds & Northrup/Microtrac Div.
Liquid Controls Corp.
The Lubrizol Corp.
Luzenac America

3M, Industrial Chemicals Prod. Div.
3M/Zeelan Industries, Inc.
Macbeth, Div. of Kollmorgen, Corp.
Magnesium Elektron Inc.
Malvern Instruments, Inc.
Malvern Minerals Co.
McWhorter, Inc.
The Mearl Corp.
Michelman, Inc.
Micro Powders, Inc.
Micromeritics Corp.
Micromet Instruments, Inc.
Mid-States Eng. & Mfg.
Millipore Corp.
Mineral Pigments
Ming-Zu Chemical Ind., Ltd.
MiniFIBERS, Inc.
Minolta Corp.
Mississippi Lime Co.
University of Missouri-Rolla
Modern Paint & Coatings
Morehouse-Cowles Inc.
Morton International, Inc.,
   Universal Color Dispersions
Mountain Minerals Co., Ltd.
Myers Engineering

Nacan Products Ltd.
National Chemical Co., Inc.
National Paint & Coatings Assn.
Netzsch Inc.
Neupak, Inc.
New Way Packaging Machinery Inc.
Norman International
North Dakota State University
NYCO Minerals, Inc.

Obron Atlantic Corp.
Ohio Polychemical Co.
Olin Chemicals
Opticolor, Inc.
Ortech International

Pacific Micro Software Engineering
Paint & Coatings Industry
   Magazine
Pen Kem Inc.
Peninsula Polymers
Perkin-Elmer
Physica USA
Pioneer Packaging Machinery, Inc.
Plastican, Inc.
Polar Minerals
Poly-Resyn, Inc.
PPG Industries, Inc., Silica Prods.
PPG Industries, Inc., Spec. Chems.
PQ Corp.
Praxair, Inc.
Premier Mill Corp.

Progressive Recovery, Inc.
Purity Zinc Metals Co., Ltd.
Pyosa, S.A de C.V.

Q-Panel Co.

Raabe Corp.
RadTech Intl. North America
Ranbar Technology, Inc.
Reichhold Chemicals Inc.
RHEOX, Inc.
Rhone-Poulenc Inc.
Rohm and Haas Co.
Rohm Tech Inc.
Ronningen-Petter
Rosedale Products, Inc.
Charles Ross & Son Co.
Russell Finex Inc.

Sandoz Chemicals Corp.
Sanncor Industries, Inc.
Sartomer Co.
Schenectady International
Schold Machine Corp.
Schuller Filtration
SCM Chemicals
Scott Bader, Inc.
Semi-Bulk Systems, Inc.
Serac, Inc.
Shamrock Technologies, Inc.
Sheen Instruments Ltd.
Shell Chemical Co.
Sherwin-Williams Co.
Shimadzu Scientific Inst., Inc.
Silberline Manufacturing Co., Inc.
Sino-American Pigment Systems
Sonoco Products Co.
South Florida Test Service
Southern Clay Products, Inc.
University of Southern Mississippi
Spartan Color Corp.
Specialty Minerals Inc.
Steel Structures Painting Council
Steeltin Can Corp.
Sub-Tropical Testing Service
Sullivan Chemical
Sun Chemical Corp.
Systech Environmental Corp.

Taber Industries
Tego Chemie Service USA
Texaco Chemical Co.
Thiele Engineering
Troy Corp.

U.S. Sack Corp.
U.S. Silica Co.
Union Carbide Corp.
Union Process Inc.
United Catalysts, Inc.
United Mineral & Chemical Corp.
United States Testing Co.

Van Waters & Rogers Inc.
R.T. Vanderbilt Co., Inc.
Velsicol Chemical Corp.
Versa-Matic Pump Co.
Vorti-Siv Div., of M&M Ind., Inc.

Wacker Silicones Corp.
Warren Rupp, Inc., a Unit of Idex
Wilden Pump & Engineering Co.
Witco Corp.

X-Rite, Inc.

Zeneca Biocides, Zeneca Inc.
Zeneca Resins

1992 Picture It Painted Professionally Winners Honored During PDCA Annual Convention, in Orlando, FL

The winners of the 1992 "Picture It Painted Professionally" (PIPP) contractor contest were honored during the Painting & Decorating Contractors of America's (PDCA) Annual Convention on February 17-19, in Orlando, FL. The contest is sponsored by the National Paint & Coatings Association in conjunction with PDCA.

The 11-year-old competition is designed to award and showcase the creative and skillful use of paint.

A total of 16 professional painting and decorating contractors were recognized for their exceptional painting abilities. Projects were rated on by a panel of industry representatives on the use of color and special painting techniques and treatments, and overall creativity and skill and execution.

The awards were presented in five categories: interior residential, exterior residential, interior commercial, exterior commercial, and industrial. The 1992 PIPP winners are:

INTERIOR RESIDENTIAL

First Place: The Strout Residence—Karl Heinz-Meschbach and Friends, Circle Pines, MN;

Second Place (tie): Wykeham Road Residence—D&L Painting, Torrington, CT; and

Second Place: Vaux Hill Estate—Theodore Hooven Sons, Inc., Edgemont, PA.

EXTERIOR RESIDENTIAL

First Place: Chateau Agape—Cal Crew Painting, Sausalito, CA; and

Second Place: Clayton Street Residence—Jack L. Phillips, General and Painting Contractor, Berkeley, CA.

INTERIOR COMMERCIAL

First Place: Holy Angels Korean National Roman Catholic Church—Theodore Hooven Sons, Inc.;

Second Place: Westminster Presbyterian Church—Ingersoll Painting & Construction, Alden, NY;

Honorable Mention (tie): St. John the Evangelist Church—MAPCO Enterprises Inc., Scotia, NY; and

Honorable Mention: Shrine of St. Joseph—Picco & Benson, Inc., St. Louis, MO.

EXTERIOR COMMERCIAL

First Place: Alice Arts Center—Rainbow Painting & Decorating Company, San Francisco, CA;

Second Place (tie): Thomas Fallon House—Joseph's Painting & Decorating, Inc., San Jose, CA;

Second Place: Fiesta Texas Theme Park—R.A. Travis Painting, Inc., San Antonio, TX;

Honorable Mention (tie): Empire of America Building—Ingersoll Painting and Construction, Inc.; and

Honorable Mention: 115th Street Rental Units—Cahill's Decorating, North Olmstead, OH.

INDUSTRIAL

First Place: Truck and Bus Flint Assembly Plant—Genesee Painting Company, Inc., Flint, MI;

Second Place (tie): Kaiser Engineering Blast Furnace Rehabilitation—Avalotis Painting Company, Inc., Pittsburgh, PA; and

Second Place: LTV Steel Pittsburgh Works Boiler House—Delbert L. Smith Company, Inc., McKees Rocks, PA.

Obron Atlantic Moves To New Corporate Headquarters In Painesville, OH

Obron Atlantic Corporation, Inc., Painesville, OH, a provider of metallic pigments and printing inks, has moved to a new and expanded 16,000-sq. ft. corporate headquarters in Painesville.

The new corporate facility houses various Obron Atlantic operations, including general administration, data management, sales, marketing, financial operations, and customer service.

In addition to the corporate offices, the new building also houses the manufacturing of metallic printing inks by Obron Atlantic's sister company, Eckart Ink Manufacturing Company (EMIC).

Obron Atlantic will continue to manufacture gold bronze pigment from its current Painesville production facilities. Technical service also will remain at the plant which is located near the new corporate headquarters facility.

Obron Atlantic provides metallic pigments for a range of industries, including: printing inks for offset, gravure, and flexography; plastics; paints and coatings; automotive; and other areas.

American Chemical Society's PMSE Division Seeks Nominations for Roy W. Tess Award in Coatings

Nominations are being sought for the Roy W. Tess Award in Coatings, bestowed annually by the American Chemical Society's (ACS) Division of Polymeric Materials: Science and Engineering (PMSE).

This major award is for $1,000 and will be presented during the 208th meeting of ACS, on August 21-26, 1994, in Washington, D.C.

The Tess Award recognizes outstanding individual achievements and noteworthy contributions to coatings science, technology, and engineering, confirming PMSE's long-standing and continuing support and dedication to excellence in the coatings field.

The Chairman of the Tess Award is George R. Pilcher, of Akzo Coatings Inc. Distinguished members of the Nominating Committee are: Professor Gordon P. Bierwagen, of North Dakota State University; Professor James O. Stoffer, of University of Missouri-Rolla; Dr. Donald W. Boyd, of PPG Industries, Inc.; and Dr. Robert F. Brady Jr., of Naval Research Laboratory.

Nominations are welcomed from all sections of industry, academia, and government, and should be forwarded to the Chairman at: Akzo Coatings Inc., P.O. Box 147, Columbus, OH 43216-0147. Upon receipt of names, the Chairman will provide a documentation form requesting information on the nominee relevant to patents, publications, overall qualifications, etc.

All finalized nominations for the 1994 Tess Award should be submitted prior to September 1, 1993. Nominations received after this date will be considered for the succeeding year's award.

New Construction Contracting Falls in February; Public Works and Utilities Projects Hit the Hardest

The F.W. Dodge Division, McGraw-Hill, New York, NY, has reported that contracting for new construction in the U.S. declined six percent in February, resulting from a steep downturn for the volatile nonbuilding construction sector (public works and utilities).

Housing realized a relatively modest slippage, while nonresidential building was able to show a slight gain for the month. The February nonbuilding sector volume was consistent with its performance during the second half of last year. Officials at the Dodge Division have indicated that "the construction industry is holding its own for the moment."

Nonbuilding construction dropped 17% in February, due mostly to the sharp reduction in utility projects. Excluding utilities, the dip registered by the public works segment is a more manageable five percent, which is within the normal variation shown by this group.

The value of residential contracts decreased five percent in February. Single family housing was responsible for most of this decline, as the multifamily side of the market held steady at a depressed pace. According to sources at Dodge, "single family housing remains a key element in determining how far the construction recovery goes in 1993."

Nonresidential building edged up two percent in February, aided by a particularly strong volume of school construction and the start of a convention center in Orlando, FL. After weakening towards the end of 1992, the institutional categories have shown modest improvement during 1993's initial months. An increase for store construction also has helped the nonresidential total, but office and manufacturing starts remained at "dismal" levels.

At the end of the first two months of this year, total construction on an unadjusted basis was down four percent from its performance at the same time in 1992. Two regions have reported substantially weaker showings this year as compared to 1992. The North Central and West have posted declines of 19 and 16%, respectively. The Northeast, up two percent, and the South Central, up four percent, have shown improvement. However, the South Atlantic realized a 15% gain during this period.

Mergers & Acquisitions...

Sumitomo Corporation and Summit Precision Polymers Open Polymer Plant in Mountaintop, PA

Sumitomo Corporation of America (SCOA), New York, NY, and its subsidiary, Summit Precision Polymers Corporation (SPPC), Fort Lee, NJ, have opened a new 42,000-sq. ft. manufacturing facility in Mountaintop, PA.

This new plant will produce high performance polymers used primarily as additives in coatings, lubricants, paints, adhesives, and plastics.

A wide variety of polymers including polytetrafluoroethylene, low and high density polyethylene, polypropylene, carnauba wax, resins, and paraffins, will be processed and distributed from the new facililty.

The Mountaintop, PA plant is SPPC's first manufacturing facility, and when in full operation may employ approximately 35-40 people.

All the equipment within the SPPC plant and lab is custom designed for the facility. The facility is outfitted with state-of-the-art environmental control equipment.

PPG Industries Acquires Catalyst Plant in The Netherlands; Operations Expected to Begin This Year

The Chemicals Group of PPG Industries, Inc., Pittsburgh, PA, has acquired an idle Netherlands catalyst plant and will convert it to production of precipitated amorphous silicas.

The facility was acquired from UOP B.V., a subsidiary of UOP, a Des Plaines, IL joint venture of Allied-Signal, Inc. and Union Carbide. Terms of the agreement were not disclosed.

The plant, at Delfzijl, near Groningen in the northern Netherlands, formerly produced fluid catalysts for UOP B.V.'s Katalistiks unit. It will have an annual silicas production capacity in excess of 10,000 metric tons when PPG begins operations later this year.

According to officials at PPG, the company has applied to Dutch authorities for permits to start operations.

PPG's initial work force will be 25-30 people, with additional employment as business expands.

Plans for initial production of several grades of Hi-Sil proprietary silicas, including those used to make microporous membranes for automotive and telephone batteries and as carriers for other chemicals, are in the works.

This silica plant is PPG's first in Europe.

ICI Paints and Lilly Industries Reach Agreement On Transfer of Businesses

ICI Paints, Cleveland, OH, a global business of Imperial Chemical Industries PLC, has reached an agreement with Lilly Industries Inc., Indianapolis, IN, for the transfer of ICI Paints U.S.-based liquid industrial coatings business in exchange for an undisclosed sum of cash and Lilly's packaging coatings business.

The agreement is subject to regulatory approval in the U.S. The transaction represents less than one percent of ICI group net assets.

Preservatives Production Plant Opened by Hüls in Toronto

Hüls America Inc., Piscataway, NJ, has opened a new manufacturing plant in Toronto, Ontario, Canada, for the production of Nuosept® preservatives and other biocide products.

The new production facility is adjacent to Hüls' existing driers and additives plant. This proximity allows for the single site consolidation of the company's manufacturing capacities for these related product lines.

Hüls' new plant is designed to meet safety and environmental standards. The three-story building houses a 6,000-gallon reactor system, water scrubber, treatment system, and miscellaneous equipment.

A Review of Inverse Gas Chromatography Theory Used in The Thermodynamic Analysis Of Pigment and Polymer Surfaces

Charles R. Hegedus
Naval Air Warfare Center*

Ihab L. Kamel
Drexel University†

Inverse gas chromatography offers the capability of analyzing filler particle and polymer surfaces, as well as the interactions which occur between these species in paints and other particulate-filled composites. This is accomplished by determining retention times of well characterized probe organic solvent molecules through columns packed with either filler or polymer particles. Intermolecular interaction and gas chromatography theory are reviewed and equations are derived which allow the calculation of filler particle and polymer surface energy; free energy, enthalpy and entropy of adsorption of polymers onto particles; and work of adhesion between these two constituents.

INTRODUCTION

Gas chromatography (GC) is one of the most widely used analytical techniques in the chemical research and industrial communities. In general terms, it is a separation process of a mobile vapor phase through a stationary, discontinuous solid phase. The vapor phase is injected into one end of a packed column containing the solid phase. An inert gas such as helium is also introduced at the column inlet in order to carry the vapor through the column bed. A detector at the end of the column analyzes the vapor as it exits. *Figure* 1 is a schematic of a typical gas chromatography apparatus.[1-4]

*Dr. Hegedus can be contacted at Air Products & Chemicals, 7201 Hamilton Blvd., Allentown, PA 18195.
†Dept. of Materials Engineering, 32nd & Chestnut Sts., Philadelphia, PA 19104.

While the vapor is flowing through the column, it is in intimate contact with the solid substrate, allowing chemical and physical interactions. Since unlike molecules will interact differently with the solid within the column, the "lag" time which it takes these molecules to travel through the column, referred to as retention time (t_r), will vary. Packing the column with a well-characterized solid can result in precise separation and identification of the injected species. Traditionally, gas chromatography has been used to identify and analyze gases and vapors, separate vapor mixtures, study chemical structure, determine mechanisms and kinetics of reactions, and study thermodynamics of mixtures and reactions.[4-7]

While gas chromatography was designed for analysis of the mobile vapor phase, inverse gas chromatography (IGC) was designed for analysis of the stationary solid phase.[5-7] In this case, specific reagents, called probes, which are well characterized, are introduced into a column to study their interaction with the solid surface of interest. The probe materials are typically low molecular weight organic solvents and the stationary phase may be any solid material which can be "packed" into a column (i.e., particles, whiskers, fibers, etc.). Except for the reversal of roles of the vapor and solid phases, the theoretical principles and instrumentation are basically the same for both GC and IGC.

Inverse gas chromatography is a relatively new technique for analysis of solid surfaces and their interactions with adsorbates. Reference (8) is a recent publication addressing the use of IGC, and *Table* 1 is a listing of polymer parameters which have been studied using this technique. Although it has been used to study polymers
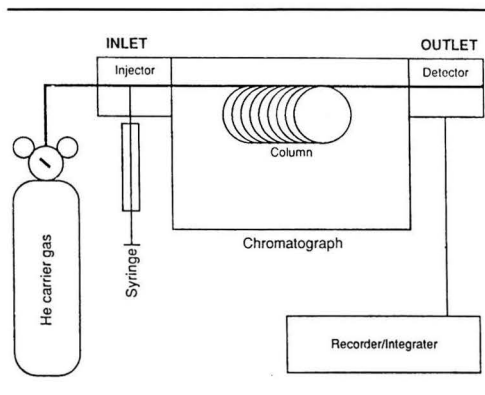
Figure 1—Schematic illustration of a typical gas chromatograph

and their interactions with solvents and fibers, its use as a tool for analysis of polymer-filler particle interactions has been limited.[23-27] As part of an overall effort to characterize and analyze pigment-polymer interactions and their effects on properties, an IGC study was conducted with the following objectives:

(1) Characterize the surface energetics of filler particles and polymers independently.

(2) Analyze the thermodynamics of polymer-pigment interactions.

(3) Advance the understanding and use of IGC as a technique to characterize filler materials and develop organic coatings, sealants, adhesives, and other polymeric composites.

The experimental details of this study will be presented in a subsequent paper. The following is a presentation of molecular interaction and IGC theory used to analyze the surface of solid materials, including polymers and particulate fillers.

INTERMOLECULAR INTERACTIONS

The interaction between a solid substrate and an adsorbing species is attributed to secondary bonding caused by both polar and nonpolar (dispersive) Van der Waals forces between adsorbent and adsorbate. Fowkes and coworkers have led the argument that these polar forces are actually Lewis acid-base interactions.[28-31] This paper further utilizes these concepts in describing adsorption phenomena, specifically in IGC. Before presenting this specific case of surface intermolecular interactions, consider the more generic case of any two dissimilar molecules which are in close enough proximity to interact with each other. This theory addresses why Van der Waals forces control intermolecular interactions. One model to describe the forces causing these interactions originates with the Van der Waals equation for real gases[32]:

$$P + \frac{a}{v^2} = \frac{RT}{v - b} \qquad (1)$$

where P is external pressure, v is molar volume, R is the gas constant, and T is absolute temperature. The a and b parameters provide for deviation from ideal gas behavior. The former accounts for intermolecular attraction-repulsion and the latter addresses molecular volume considerations. In other words, the parameter "a" provides a quantitative description for intermolecular forces. (Definitions for all notations used in this paper are provided in the *Appendix*.)

To define quantitatively the intermolecular interactions through the parameter "a," the various types of intermolecular forces should be identified and classified by their interaction mechanisms. The most common of these (and the most contributory) are London, Keesom, and Debye forces which affect potential energies as an inverse sixth power function of intermolecular distance. These forces are collectively referred to as the Van der Waals forces. The London forces are induced by momentary molecular dipole moments producing an electric field which interacts with other dipole moments in adjacent molecules. Although the time average of these induced dipoles indicates no overall directional polarity, instantaneous effects result in mutual attractions between molecules causing a driving force for dispersion, thus the common reference to London dispersion forces.

Keesom forces originate from the interactions between permanent dipoles present in adjacent molecules; Debye forces are caused by a molecule containing a permanent dipole and an adjacent molecule with an induced dipole. It is important to consider several points in comparing the characteristics of these intermolecular forces. First, London forces do not require the presence of a permanent dipole and, therefore, they are present in all materials, including those with nonpolar molecules. Second, the dispersion forces have relatively longer range than many other types of molecular interactions. Finally, Kaelble[32] has illustrated that even with the most polar molecules, Debye forces are relatively small compared to London and Keesom effects.

With these descriptions and considerations in mind, the molecular attraction constant, "a" in equation (1), can be mathematically expressed as:

$$a = A + B \qquad (2)$$

where A and B are the force contributions due to London and Keesom effects, respectively. These contributions can be represented by:

$$A = \frac{3 I_1 I_2}{2 (I_1 + I_2)} \alpha_1 \alpha_2 \qquad (3)$$

which can be mathematically approximated by:

$$A = 0.75 (I_1 I_2)^{0.5} \qquad (4)$$

where I_1 and I_2 are the ionization potentials, and α_1 and α_2 are the polarizabilities of isolated molecules 1 and 2, respectively.

$$B = \frac{2u_1^2 u_2^2}{3kT} \qquad (5)$$

where k is the Boltzmann constant, and u_1 and u_2 are the dipole moments of isolated molecules 1 and 2, respective-

Table 1—Polymer Properties Determined by IGC

Parameter	Reference
Percent crystallinity	(9-11)
Flory-Huggens interaction parameter	(12-14)
Solubility parameter	(15,16)
Enthalpy of solution	(17)
Diffusion coefficient	(18)
Activity coefficient	(19,20)
Free energy of mixing	(21)
Glass transition and melting temperatures	(22)

ly. Since the summation of London and Keesom forces account for the molecular attraction constant:

$$\frac{A}{a} + \frac{B}{a} = 1 \qquad (6)$$

Now, we can define the fractional energy contributions to intermolecular forces as:

$$d \equiv A/a \text{ for London dispersion forces}$$
and
$$p \equiv B/a \text{ for Keesom polar forces}$$

Thus:

$$p + d = 1 \qquad (7)$$

And, substituting equations (4) and (5) into (2) yields:

$$a_{12} = 0.75(I_1 I_2)^{0.5} \alpha_1 \alpha_2 + \frac{2u_1^2 u_2^2}{3kT} \qquad (8)$$

where a_{12} specifically addresses the interactions between molecules 1 and 2. Now, for the case of a homogeneous phase, $I_1 = I_2$, $\alpha_1 = \alpha_2$, $u_1 = u_2$, and equations (4) and (5) reduce to:

$$A = 0.75I \alpha^2 = a d \qquad (9)$$

and

$$B = \frac{2 u^4}{3kT} = a p \qquad (10)$$

Rearranging equations (9) and (10) for homogeneous materials 1 and 2 and substituting into equation (8) yields a relationship for intermolecular interactions:

$$a_{12} = (a_1 a_2)^{0.5}[(d_1 d_2)^{0.5} + (p_1 p_2)^{0.5}] \qquad (11)$$

SURFACE INTERACTIONS INVOLVING A CONDENSED PHASE

The concept and derivation presented has been for the interaction of any two molecules in close proximity. Now, consider the more specific case of surface interactions when at least one of the phases is a condensed phase. These interactions are also caused by Van der Waals forces. In this case, the interactions which occur originate from the surface energy (tension) of the individual materials involved. Good[33] has made a parallel correlation relating surface tension (γ), the contributory interaction energy fractions (d,p), and the work of adhesion (w_a) between two materials:

$$w_a = 2(\gamma_1 \gamma_2)^{0.5}[(d_1 d_2)^{0.5} + (p_1 p_2)^{0.5}] \qquad (12)$$

where γ_1 and γ_2 are introduced as the respective surface tensions of materials (1) and (2).

Fowkes[34] has indicated that surface tension and the work of adhesion, W_a, can be expressed as a sum of their contributory molecular interactions. In this case:

$$\gamma = \gamma^D + \gamma^P \qquad (13)$$

and

$$w_a = w_a^D + w_a^P \qquad (14)$$

where the superscripts D and P refer to the intermolecular dispersive and polar contributions, respectively. Relating equations (13) and (14) to (12) and the intermolecular concepts presented previously, it can be shown that:

$$\gamma^D = (\gamma)(d) \text{ and } \gamma^P = (\gamma)(p)$$

and thus:

$$w_a = 2[(\gamma_1^D \gamma_2^D)^{0.5} + (\gamma_1^P \gamma_2^P)^{0.5}] \qquad (15)$$

Fowkes[34,35] has independently derived a similar equation solely for the dispersive contributions of surface tension to the work of adhesion [first term on the right hand side of equation (15)]. *Figure* 2 compares the potential energy-distance relationship for primary bonds (covalent, ionic, metallic) versus that for dispersion forces. At relatively close distances (1-3 angstroms), primary bonds have a deep energy well, indicating strong but highly localized forces. In contrast, the dispersion forces have a shallow energy well but they are more significant at longer distances. Now, consider a molecule of condensed material (1) in two separate equilibrium conditions, within the bulk phase and at the surface of the phase. The atoms or molecular segments within the bulk phase are totally surrounded by similar species and they are fixed by the highly localized forces which dominate at short interatomic distances, thus resulting in a tightly bound, relatively symmetrical position. A similar species on the surface is in an asymmetric position where strong attraction by the bulk phase is only on one side, preventing it from escaping to the less condensed adjacent phase. It is
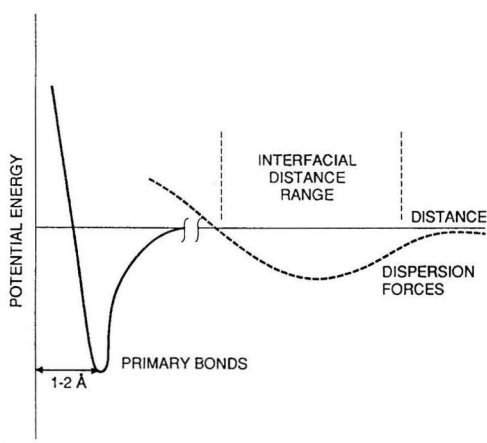
Figure 2—Potential energy versus distance relationship for atomic and molecular forces

Figure 3—Illustration of the de-adhesion process. ΔG equals the work of adhesion

$$\Delta G = W_{AB}$$

1 cm²
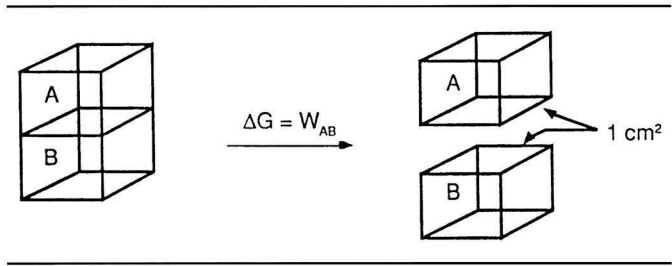
this condition at the surface which causes excess energy per molecule, usually referred to as surface tension. In addition, since the two phases are relatively far apart compared to the primary bonds in *Figure 2*, intermolecular forces dominate, causing secondary bonds between the phases.

If the phase adjacent to a condensed phase is gaseous, the intermolecular attractions between the surface molecules and the gaseous molecules are relatively few due to the relatively low density of gases. However, if the adjacent material (2) is in a condensed phase, the opportunity for intermolecular attractions between materials (1) and (2) are significantly greater. Therefore, there is an obvious difference in the energy state of a surface molecule residing next to a gaseous state versus that for a molecule next to a condensed state. Fowkes[34] has stated that, due to their exceptionally long range, only dispersion forces operate across phase boundaries. He assumed that the reduction in energy, ΔE^s, required to bring a molecule to the surface adjacent to a gaseous phase as opposed to a condensed phase was equal to the geometric mean of the dispersive component of the surface tensions of the two phases:

$$\Delta E^s = (\gamma_1{}^D\gamma_2{}^D)^{0.5} \tag{16}$$

Accordingly, the work required to bring a molecule of material (1) to the material (2) interface is:

$$(\text{Work})_1 = \gamma_1 - (\Delta E^s) = \gamma_1 - (\gamma_1{}^D\gamma_2{}^D)^{0.5} \tag{17}$$

A similar expression can be presented for bringing a molecule of material (2) to the surface, and the sum of these contributions is the resulting interfacial surface energy, γ_{12}:

$$\gamma_{12} = (\text{Work})_1 + (\text{Work})_2 = \gamma_1 + \gamma_2 - 2(\gamma_1{}^D\gamma_2{}^D)^{0.5} \tag{18}$$

The work of adhesion is the work required to separate two materials at their interface as illustrated in *Figure 3*.

Based on the difference in final and initial conditions of this system:

$$w_a = \gamma_1 + \gamma_2 - \gamma_{12} \tag{19}$$

Substituting equation (18) into equation (19) yields:

$$w_a = 2(\gamma_1{}^D\gamma_2{}^D)^{0.5} \tag{20}$$

Based on the initial assumption that dispersive forces are the only forces which transcend phase boundaries, Fowkes states equation (20) is true for all materials. Other researchers[36-39] consider this to be a specific form (addressing only dispersive forces) of the more general form presented in equation (15). Nonetheless, equation (20) is certainly valid if one of the two interacting materials is nonpolar as is the case with alkanes.

Equations (15) and (20) illustrate the importance of knowing surface characteristics, especially surface tension. Liquid surface tensions can be readily measured by several techniques. In contrast, the surface tension of solids is difficult to obtain. One method is contact angle analysis. When a liquid is placed onto a solid and is allowed to reach equilibrium, the liquid will form a contact angle, Θ, with the solid surface (*Figure 4*). The reaction of the liquid in forming this contact angle is caused by its interaction with both the solid phase and the surrounding vapor phase. This reaction is a direct result of the system's desire to minimize its free energy. In this regard, the contact angle is a thermodynamic characteristic of the interaction between the solid surface, the liquid drop, and the surrounding vapor phase. Young's equation for balancing the forces in this equilibrium condition is[40,41]:

$$\gamma_{sl} - \gamma_{sv} + \gamma_{lv}\cos\Theta = 0 \tag{21}$$

Since we are specifically addressing a liquid phase on a solid substrate, the more common subscripts sl, sv, and lv
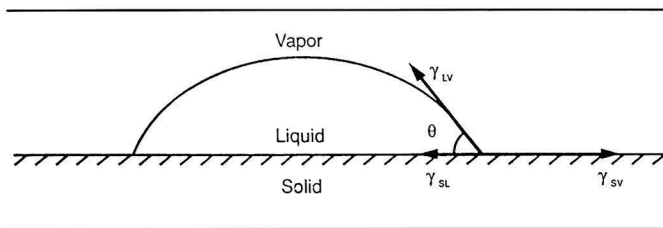
Figure 4—Relationship between contact angle and surface tension

will be used to designate solid-liquid, solid-vapor, and liquid-vapor interfaces, respectively. Rearranging equation (21) for γ_{sl} and substituting into equation (19) yields:

$$w_a = \gamma_{lv}(1 + \cos \Theta) \tag{22}$$

By equating equations (22) and (15) for the work of adhesion and rearranging, we can obtain:

$$\underbrace{\gamma_{lv}\left[\frac{1 + \cos\Theta}{2(\gamma_{lv}{}^D)^{0.5}}\right]}_{[Y]} = \underbrace{(\gamma_{sv}{}^D)}_{[INTERCEPT]} + \underbrace{\left[\frac{\gamma_{lv}{}^D}{\gamma_{lv}{}^D}\right]^{0.5}}_{[X]} \underbrace{(\gamma_{sv}{}^P)^{0.5}}_{[SLOPE]} \tag{23}$$

Using this relationship, the solid surface tension terms, which have been unobtainable by direct measurement, can be determined by measuring the contact angles of a series of polar and nonpolar liquids on the solid surface. Plotting the left side of equation (23) versus $(\gamma_{lv}{}^P/\gamma_{lv}{}^D)^{0.5}$ yields a slope equal to $(\gamma_{sv}{}^P)^{0.5}$ and an intercept equal to $(\gamma_{sv}{}^D)^{0.5}$. Therefore, applying this experimental and analytical approach can result in a comprehensive understanding of both the solid surface chemistry and the interactions between the solid and liquid materials.

IGC MEASUREMENTS AND THEORY

Unfortunately, measuring the contact angle of liquids on fine particulate solids is impossible due to their relatively small size, large surface area, and extreme surface curvature, thus nullifying the usefulness of this approach. However, polar and nonpolar interactions, as well as solid surface energetics, can be characterized using IGC data.

The recorded response from the IGC experiment is the retention time, t_r, of the probe through the column. In addition to the chemical nature of the probe and solid phases, other variables affecting this response are temperature, pressure drop across the column, and carrier gas flow rate. The retention time can be converted to the net retention volume, V_n, by[4,42]:

$$V_n = J F (t_r - t_o) = J F t_n \tag{24}$$

where F is the carrier gas flow rate, t_r is the time it takes the probe to travel through the column, t_o is the time it takes a non-interacting marker such as air to travel through the column, and t_n is the net retention time. J is the correction factor for pressure drop across the column causing gas compressibility[4]:

$$J = \frac{3 [(P_i/P_o)^2 - 1)]}{2 [(P_i/P_o)^3 - 1)]} \tag{25}$$

where P_i is the pressure at the inlet of the column and P_o is the atmospheric pressure. Figure 5 is a typical IGC response curve. It should be noted that a non-interacting species is necessary to determine the net retention time (t_n) or relative time lag between this species and the probe species traveling through the packed column.

The specific retention volume, $V_g{}^\circ$, is determined by[4,42]:

$$V_g{}^\circ = V_n \frac{273.16}{T w} \tag{26}$$
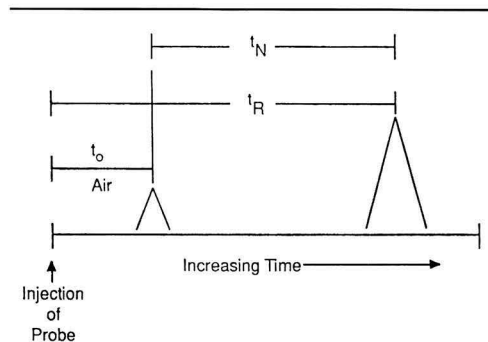
Figure 5—Representation of an idealized IGC response curve

where T is the temperature of the column in °K and w is the weight of the stationary phase in the column. $V_g{}^\circ$ in mL/g is a conversion of the net retention volume which is normalized to 0°C (273.16°K) per gram of stationary phase. Mathematical and graphical analysis of this data can result in determining the free energy, enthalpy, and entropy of adsorption of the probe molecules onto the stationary substrate. In addition, the surface energetics of the solid substrate and an understanding of its surface chemistry may be obtained, as will be shown in the following.

Martin[43] has proposed that the net retention volume is caused by both bulk and surface sorption processes which occur independently according to:

$$V_n = K_s A_i + K_1 V_1 \tag{27}$$

where A_i is the total surface area of the stationary phase, V_1 is the total volume of stationary phase, and K_s and K_1 are the surface and bulk partition coefficients, respectively. Now with sorption of organic molecules onto a relatively nonporous substrate, bulk absorption is negligible and surface adsorption is the dominant retention mechanism, thus reducing equation 27 to[32,44]:

$$V_n = K_s A_i = K_s w S_a \tag{28}$$

where w is the weight of adsorbent in the column and S_a is the specific surface area of the adsorbent.

For isothermal adsorption of a molecule from the standard gaseous state to a standard adsorption state, the free energy change of adsorption per mole of adsorbate, $\Delta G_a{}^\circ$ is given by[45]:

$$\Delta G_a{}^\circ = - \Delta G_d{}^\circ = - RT \, Ln \, (P_{s,s}/P_{s,g}) \tag{29}$$

where $\Delta G_d{}^\circ$ is the standard free energy change of desorption, $P_{s,g}$ is the adsorbate vapor pressure in the gaseous standard state, $P_{s,s}$ is the vapor pressure in equilibrium with the standard adsorption state, R is the gas constant, and T is the column temperature in °K.

Now, equations (28) and (29) can be related by first defining K_s:

$$K_s = \Gamma/C \tag{30}$$
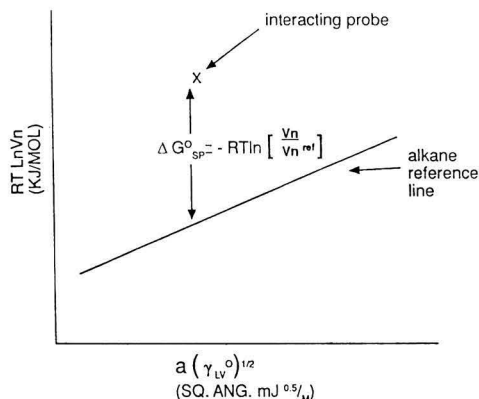
Figure 6—Graphical representation of the (RT Ln V_n) versus $(a(\gamma_{lv}{}^D)^{0.5}$ graph and the calculation of $\Delta G_{a\text{-}sp}$

where Γ is the surface concentration of adsorbate and C is the adsorbate concentration in the gas phase. However, assuming ideal conditions:

$$C = P/RT \qquad (31)$$

where P is the partial pressure of the adsorbate. Therefore, substituting equation (31) into equation (30) yields:

$$K_s = \Gamma RT/P \qquad (32)$$

The surface concentration, Γ, is related to the surface pressure, π, by the Gibb's equation[46]:

$$\Gamma = (1/RT)\,(d\pi/dLnP) = (P/RT)\,(d\pi/dP) \qquad (33)$$

In the Henry's Law region of infinite dilution of adsorbate:

$$d\,\pi/dP \longrightarrow \pi/P \qquad (34)$$

Substituting into equation (33) and subsequently into equation (32) yields:

$$\Gamma RT/P = \pi/P = K_s \qquad (35)$$

In the standard adsorption state:

$$P_{s,s} = \pi/K_s \qquad (36)$$

Substitution into equation (29) yields:

$$\Delta G_a{}^\circ = -RT\,Ln(K_s\,P_{s,g}/\pi) \qquad (37)$$

Rearranging equation (28) to $K_s = V_n/wS_a$ and substituting into equation (37) yields:

$$\Delta G_a{}^\circ = -RT\,Ln(V_nP_{s,g}/\pi wS_a) \qquad (38)$$

DeBoer[45] proposed the standard surface pressure as the pressure where the distance of separation between molecules in the adsorbed state equals that in the standard gas state. With this postulate:

$$P_{s,g} = 1.013 \times 10^5 \text{ Pa and } \pi = 3.38 \times 10^{-4} \text{ N/m}$$

Now, equation (38) can be used directly to calculate the standard free energy of adsorption and, since w, S_a, $P_{s,g}$, and π are constants, equation (38) can be reduced to:

$$\Delta G_a{}^\circ = -RT\,LnV_n + K \qquad (39)$$

where K is a constant. In addition, obtaining retention volumes at various temperatures and subsequently plotting the change in free energy versus temperature results in a linear relationship according to:

$$\Delta G_a{}^\circ = \Delta H_a{}^\circ - T\Delta S_a{}^\circ \qquad (40)$$

where, $\Delta H_a{}^\circ$ and $\Delta S_a{}^\circ$ are the enthalpy and entropy of adsorption. These quantities are determined as the intercept and slope of the $\Delta G_a{}^\circ$ versus T graph, respectively.

The work of adhesion (w_a, per surface area) between an adsorbate and an adsorbent is related to the free energy of adsorption by[47]:

$$\Delta G_a{}^\circ = -N\,a_i\,w_a \qquad (41)$$

where N is Avagodro's number and a_i is the area which an adsorbed molecule covers on the surface of an adsorbent. For purely dispersive interactions (i.e., no polar interactions) which would occur in the case of dispersion of particles in alkanes, w_a can be determined by equation (20).

By substituting equations (41) and (20) into equation (39):

$$\frac{RT\,LnV_n}{[Y]} = \frac{2N(\gamma_{sv}{}^D)^{0.5}}{[SLOPE]}\,\frac{a_i\,(\gamma_{lv}{}^D)^{0.5}}{[X]} + \frac{K}{[INTERCEPT]} \qquad (42)$$

Plotting [RT LnV_n] as a function of $[a(\gamma_{lv}{}^D)^{0.5}]$ results in a linear function with a slope equal to $[2N(\gamma_{sv}{}^D)^{0.5}]$, the nonpolar component of the solid surface energy. Schultz[48] determined the $\gamma_{sv}{}^D$ of untreated carbon fibers to be 51 ± 3 mJ m^{-2} using this method compared to 50 ± 8 mJ m^{-2} from the contact angle/wetting method previously described, thus illustrating the validity of the IGC approach.

Dorris and Gray[49] have presented an empirical approach to determine the nonpolar component of the solid surface tension, $\gamma_{sv}{}^D$. This method is based on the contribution of a CH_2 group in the alkane series to free energy of adsorption (or desorption), $\Delta G^\circ{}_{CH2}$. The resulting relationship is:

$$\gamma_{sv}{}^D = \frac{[RT\,Ln\,(V_{n2}/V_{n1})]^2}{4\,N^2\,a_{CH2}{}^2\,\gamma_{CH2}} \qquad (43)$$

where V_{n1} is the net retention volume of an alkane, V_{n2} is the retention volume of the succeeding alkane in the homologous series, a_{CH2} is the surface area of a CH_2 group (6 Å^2), and γ_{CH2} is the surface energy of a CH_2 group as constituted by close packed groups within polyethylene ($\gamma_{CH2} = 35.6$ mJ m^{-2}). Nardin[50] found that the surface energies of carbon fibers determined by this method and that from plotting equation (42) were typically within four percent.

This derivation specifically addresses nonpolar interactions which are solely present in the case of nonpolar probes. However, polar probes will undergo both polar and nonpolar interactions which are typical when filler particles are dispersed in polymer systems. In these cases, retention times will naturally be longer than with nonpolar probes. This is reflected in the IGC data analysis by points on the [RT LnV_n] versus $[a(\gamma_{lv}{}^D)^{0.5}]$ graph which are above the straight line corresponding to the

alkane probe response. Thus, the difference between the value of $[RT \, LnV_n]$ for a polar probe and that for a reference alkane probe with an equivalent $[a(\gamma_{lv}^D)^{0.5})]$ results in $\Delta G_{a\text{-sp}}°$, the polar contribution to the standard free energy of adsorption. This is graphically illustrated in *Figure 6* and mathematically determined by[48,51]:

$$\Delta G_{a\text{-sp}}° = -RT \, Ln(V_n/V_n^{ref}) \qquad (44)$$

Furthermore, it should be noted that the total free energy of adsorption is composed of both nonpolar and polar contributions. Therefore, it is possible to write:

$$\Delta G_{a\text{-np}}° = \Delta G_a° - \Delta G_{a\text{-sp}}° \qquad (45)$$

where $\Delta G_{a\text{-np}}°$ is the nonpolar contribution to the free energy of adsorption.

The implications of the relationships presented are that the surface energetics of particulate solids which are unobtainable by conventional contact angle measurements are obtainable from IGC experiments. In fact, several researchers have used this technique to analyze interactions of polymers with fibers[40,43,46,47] and fillers.[24,26] Since our immediate objectives are to analyze the surface energies of particulate fillers and to determine thermodynamic parameters of adsorption of molecules onto these fillers, IGC offers an approach. In experimental terms, we can fill an IGC column with pigment particles and independently inject a quantity of various probes with different chemical characteristics. The interactions which occur give rise to changes in the free energy, enthalpy, and entropy of the system. These changes in thermodynamic properties are reflected in the retention time and the desired thermodynamic parameters are determined and analyzed with the previously presented equations.

It must be noted that experimental complications and difficulties exist with the IGC technique. IGC demands extremely accurate monitoring of the gas flow rate, pressure drop across the column, and weight determination of the stationary phase. One critical step is packing of the column. Filling a column solely with pigment will provide excessive surface area and a huge pressure drop across the column, causing it to be unusable. In addition, inconsistencies within the column such as large voids will cause misleading results. References (24) and (27) describe experimental studies which address these issues in detail.

SUMMARY

The filler surface-polymer interactions which occur in paints and other particulate-filled composites are caused by secondary bonding, that is, nonpolar dispersion forces and acid-base interactions. The thermodynamics of these interactions have been difficult to experimentally characterize and analyze. One traditional method of studying surface energies of solid surfaces and their interactions with other materials is contact angle measurements. These measurements cannot be performed on filler particles due to their size and geometry. One technique which does allow analysis of filler-polymer surface thermodynamics is inverse gas chromatography. Retention times of different molecules through IGC columns packed with filler particles will vary due to the different interactions

which occur. Surface energies of polymers and particles; free energy, enthalpy, and entropy of adsorption; and work of adhesion between polymer and pigment can be obtained by measuring the retention times of well-characterized "probes" through IGC columns packed with filler and polymer particles and applying this data to theoretically derived equations. This approach also permits analysis of both nonpolar and polar contributions to interaction events.

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APPENDIX
Notation for IGC

a .... Molecular attraction correction constant in the Van der Waal equation

a_i ...... Surface area which an adsorbed molecule covers on an adsorbate

b ...... Molecular volume correction constant in the Van der Waal equation

d ...... Fractional contribution of Keesom forces to the molecular attraction constant

k ...... Boltmann constant

p ...... Fractional contribution of London forces to the molecular attraction constant

p_i ...... Vapor pressure of the adsorbate

t_n ...... Net retention time

t_o ...... Retention time of an inert marker

t_r ...... Retention time of probe

u ...... Dipole moment

v ...... Molar volume

w ...... Weight of stationary phase in GC column

w_a ..... Work of adhesion

A ...... London dispersion force contribution to the molecular attraction constant

A_i ..... Total surface area of stationary phase in GC column

B ...... Keesom force contribution to the molecular attraction constant

C ...... Adsorbate concentration in the vapor phase

E^s ..... Energy required to bring a molecule in the bulk of a condensed phase to the surface

F ...... Flow rate of carrier gas through GC column

G^o_a ..... Standard free energy of adsorption

G^o_d ..... Standard free energy of desorption

G^o_{a-np} ... Nonpolar component of the standard free energy of adsorption

G^o_{a-sp} ... Polar component of the standard free energy of adsorption

H^o_a ..... Standard enthalpy of adsorption

I .... Ionization potential

J ...... Correction factor for pressure drop across the GC column

K ...... Free energy of adsorption-Ln V_n constant

K_s ..... Surface adsorption partition function

K_1 ..... Bulk adsorption partition function

N ...... Avagodro's number

P ...... External pressure on a gas

P_i ...... Pressure at the inlet of the GC column

P_o ..... Pressure at the outlet of the GC column

$P_{s.s.}$ .... Equilibrium vapor pressure of a probe in the standard adsorption state

$P_{s.g.}$ .... Adsorbate vapor pressure in the gaseous standard state

R ...... Universal gas constant

S^o_a ..... Standard free entropy of adsorption

S_a ..... Specific surface area

T ...... Column temperature

V_n ..... Net retention volume

V_g^o .... Specific retention volume

AN ..... Guttman acceptor number

AN* .... Fowkes acceptor number

DN ..... Guttman donor number

α ...... Polarizability

γ_{lv} ..... Liquid-vapor surface tension

γ_{sl} ..... Solid-liquid surface tension

γ_{sv} ..... Solid-vapor surface tension

π ...... Adsorbate surface pressure

Γ ...... Adsorbate surface concentration

Θ ...... Contact angle

Thermodynamic Analysis of Pigment and Polymer Surfaces Using Inverse Gas Chromatography

Charles R. Hegedus*
Naval Air Warfare Center

Ihab L. Kamel
Drexel University†

The thermodynamics of pigment particle surface-polymer interactions have been characterized using inverse gas chromatography (IGC). The use of IGC in this new application extends available analysis to the calculation of the thermodynamic properties of reversible particle-polymer interactions. This IGC technique was used to determine solid surface energies of particles and polymers; free energy, enthalpy, and entropy of adsorption; and work of adhesion. Available theories to calculate the nonpolar (dispersive) and polar contributions to these surface properties were reviewed and found applicable. The values obtained agree favorably with those obtained from alternate techniques. Nonpolar forces provided a large contribution to the free energy and enthalpy of adsorption. In addition, acid-base pairs were found to undergo favorable interactions while acid-acid and base-base pairs were less likely to undergo strong interactions. Amphoteric adsorbates underwent extensive interactions with both acids and bases due to their mutual compatibility and their tendency to self-associate. Entropies of adsorption for most interactions were found to be negative, indicating conformational changes from the gaseous state to a more restricted adsorbed configuration, similar to those in the condensation process. Entropy contributions to the total free energy change were approximately 30% of the enthalpy changes.

INTRODUCTION

The interactions which occur between pigment particles and polymer binder in paints have considerable effects on the properties of these coatings both prior to and after

application. These interactions are typically a form of secondary intermolecular bonding which occurs between the polymer chains and surface groups on the pigment. A number of studies have illustrated the magnitude of these effects on coating properties,[1-6] and reference (7) provides a recent review of this subject. Nonetheless, the causes and effects of pigment-polymer interactions are among the least understood and under utilized phenomena in coatings technology. One reason for this may be the difficulty in studying pigment particle surfaces due to their size and geometry.

Reference (8) is a review of inverse gas chromatography (IGC) and it illustrates that this technique can be used to analyze thermodynamic properties of particulate solid surfaces and their interactions with other materials. This theory and the IGC technique were used in the current study to pursue the following objectives:

(1) Characterize the surface energetics of pigment particles and polymers independently.

(2) Analyze the thermodynamics of pigment-polymer interactions.

(3) Advance the understanding and use of IGC as a technique to characterize filler materials and develop organic coatings, sealants, adhesives, and other polymer composites.

EXPERIMENTAL

Equipment

A Perkin Elmer Sigma 2000 gas chromatograph equipped with a high sensitivity flame ionization detector (FID) was used for the IGC experiments. The injector and detector temperatures were 200°C and 250°C, respectively. For filler particle analysis, the column experimental temperatures ranged from 70° to 110°C. For polymer samples, a temperature range of 37.5° to 50°C was uti-

*Dr. Hegedus can be contacted at Air Products & Chemicals, 7201 Hamilton Blvd., Allentown, PA 18195.
†Dept. of Materials Engineering, 32nd and Chestnut Sts., Philadelphia, PA 19104.

Table 1—Composition of TiO₂ Pigments

Pigment	TiO₂	Al₂O₃	SiO	P₂O₅	Sb₂O₃	K₂O	ZnO	C
TIO-1 ...	91.10	2.20	6.67	—	—	—	—	—
TIO-2 ...	85.80	3.59	10.42	0.14	0.03	0.02	—	—
TIO-3 ...	99.17	0.02	—	0.59	—	0.03	—	0.19

lized. It was essential to test polymer samples below their glass transition temperature (T_g) in order to maintain consistent sorption characteristics. These temperature ranges were also selected because they were the lowest temperatures resulting in retention times of less than two hours for most probes. Lower temperatures resulted in extremely long retention times ($\gg$ two hours), while higher column temperatures resulted in retention times which were too short to distinguish statistically the differences between probes. Helium was used as the inert carrier gas at a flow rate of 30 mL/min. The gas chromatography (GC) instrument was equipped with a carrier gas flow rate regulator which was confirmed with a soap bubble flow meter. (The carrier gas flow rate is critical for calculation of retention volumes.) The column outlet was at atmospheric pressure which was obtained daily from a barometer. The column inlet pressure (gage pressure) was determined daily for each column at temperature by inserting a syringe attached to a mercury monometer into the inlet port. The total pressure at the inlet was determined by adding the atmospheric pressure to the gage pressure.

The gas chromatograph was attached to a Perkin Elmer LCI-100 computing integrator/recorder. The recorder had a variable span selection up to 1024 V. A span of 4 V was used to obtain high sensitivity and to ensure that extreme dilution of the probes (Henry's Law region) was obtained. The recorder speed was set at 5 mm/min and a positive offset of five percent was used to detect negative deviations and baseline shifts. Retention times (t_r) of the inert marker (air) and interactive probes were determined from their peak maxima as indicated by the recorder. Net retention times, t_n, were determined as the lag time (difference) between exiting of the probe and air.

Materials

Both filler particles (titanium dioxide and silica) and polymers (poly(methyl methacrylate) and poly(acrylic acid)) were analyzed using the IGC technique. *Table 1* lists the composition of the titanium dioxide samples as determined from atomic absorption spectroscopy as performed according to ASTM D 1394 and D 4563. The

silica was nearly 100% SiO_2. *Table 2* lists the physical properties of all of the filler samples studied. *Table 3* lists the PMMA and PAA average molecular weight obtained from gel permeation chromatography and T_g from differential scanning calorimetry (DSC).

In an initial attempt to analyze the filler materials, a sample of TIO-1 was packed into a stainless steel tube with a length of 0.5 m and an internal diameter of 5.3 mm. This column produced probe retention times in excess of two hours due to the large amount of titanium dioxide surface area and the huge pressure drop across the column ($\gg$ 600 mm Hg gage). In order to reduce this retention time, filler samples were mixed with 250 micron diameter glass beads (Catalogue No. 5-9203, Supelco Inc., Bellefonte, PA). Prior to mixing with the pigment samples, the beads were acid washed and coated with dimethyldicholosilane (DMCS), which is a typical treatment for IGC support materials to render them inert. In order to prove that the glass would have no significant effect on probe interactions when mixed with the filler particles of interest, a column consisting solely of treated glass beads was prepared and tested. The net retention time of all the probes through this column at 70°C was zero seconds. Therefore, all probe effects in the pigment-glass columns are attributable to pigment effects.

From this point, columns for pigment analysis were prepared by mixing the glass beads and titanium dioxide samples at a 5 (glass) to 1 (TiO₂) ratio (by weight) in a DMCS treated beaker. Silica samples were mixed in a 25 to 1 ratio due to their high surface area (130 m²/g). Treating the beaker was necessary to eliminate attraction of the pigment to the beaker walls. This mixture was then stirred with a glass rod until a homogeneous mixture with no clumps was obtained. The mixture was then introduced into a stainless steel tube. Titanium dioxide columns produced in this manner had inlet pressures ranging from 100 to 350 mm Hg gage which is comparable to those stated in previous studies. Silica columns had inlet pressures up to 800 mm Hg gage. *Table 4* provides a description of the columns analyzed. Prior to testing, all pigment columns were conditioned in the GC at 120°C for a minimum of 15 hr with a free flow of helium through the column.

The polymer samples had to be analyzed by first depositing them onto a support phase. A technique designed by Al-Saigh and Munk[9] was utilized. Nine percent solutions of PMMA in methyl ethyl ketone and PAA in methanol were slowly titrated onto separate samples of acid washed, DMCS treated Chromosorb W powder (Supelco Inc.), a common GC support material. Care was taken to allow the solution to contact the particles but not their

Table 2—Pigment Characteristics

Pigment Designation	Density (gr/mL)	Surface Area (sq m/gr)	Average Diameter (nm)	Packing Factor φ	Oil Absorption (g oil/100 g pig)	Theoretical CPVC (%)
TIO-1	4.0	14.3	300	0.640	29.3	47.6
TIO-2	3.8	35.8	192	0.634	44.5	35.2
TIO-3	3.8	7.6	147	0.636	31.6	42.6
SIO-1	2.2	130.5	16	0.640	175.7	19.2

container. Enough solution was applied to the support phase to obtain a minimum of eight percent polymer by weight. Following this process, the powder was dried for 20 hr at 60°C followed by 24 hr in a vacuum oven at 60°C. Throughout the latter stages of this procedure, a constant sample weight was obtained, indicating evaporation of all solvent.

To confirm the removal of all solvent and the concentration of polymer on the resulting particulates, thermal gravimetric analysis (TGA) of treated and untreated Chromosorb W samples was performed. The samples were heated in air to 500°C at a rate of 5°C per minute. The untreated sample remained at a constant weight. The PMMA and PAA treated samples lost 14 and 9 w/o, respectively, due to extensive oxidation of the polymers. The TGA data indicated no presence of solvent in the polymer-glass samples. After confirming the removal of solvent and the polymer concentration on the PMMA and PAA treated Chromosorb W, columns were prepared in stainless steel tubes (*Table* 4).

In order to analyze thoroughly the surface characteristics of the fillers and polymers, a range of Lewis acid and base probes was required. Gutmann[10] and Fowkes[11] have characterized materials according to electron donor and acceptor numbers, DN and AN, respectively. The donor number reflects the ability of the material to share its electrons based on the molar enthalpy of the reaction with a reference acceptor $SbCl_5$. The Gutmann acceptor number characterizes the ability to accept the shared electrons as determined from the NMR shift of ^{31}p contained in $(C_2H_5)_3PO$ when reacted with the acceptor. However, Fowkes[11] has reported that acceptor numbers determined by NMR are misleadingly high because the probe Van der Waals contributions are significant but not accounted for. He corrected acceptor numbers by subtracting out the probe Van der Waals contributions. These conventions provide convenient means of characterizing and selecting candidate probe materials. In addition, Schultz et al.[12] have determined the surface area per molecule adsorbed, a_i, and dispersive surface energies, γ_{lv}^D, of a number of probes. These parameters are required for use in equations to determine filler surface tensions.[8] *Table* 5 is a list of probes (reagent grade or better) used in this study and their pertinent parameters.

Procedures

Initially, three separate replicates were performed for each condition. Using this procedure, a standard deviation of less than one percent typically was obtained for Ln V_n (net retention volume) values. However, while performing experiments with the first column tested (Column H-1, TIO-1), it was determined that there was a risk of changing the column characteristics over several weeks of testing. This was reflected in a change in probe retention times. Therefore, since reproducibility between replicates was easily obtained, each probe was performed in duplicate at each column temperature in order to speed analysis of the columns. Using this procedure, column conditions remained relatively constant during the one to two week period which it took to complete the column testing. A detailed discussion of the changes in column

Table 3—Polymer Sample Designations, Suppliers, Molecular Weight, and Glass Transition Temperature

Polymer	Supplier	M_w	M_n	M_z	M_w/M_z	T_g(°C)
PMMA	Rohm & Haas	41,900	19,000	19,000	2.21	70
PAA	Polysciences	250,000[a]	—	—	—	—

(a) Data provided by supplier.

Table 4—IGC Columns Analyzed

Designation	Support Material	Support Weight (grams)	Filler Material	Filler Weight (grams)	Total Weight (grams)
H-2	Glass	13.26	TIO-1	2.65	15.91
H-3	Glass	13.88	TIO-1	2.78	16.66
H-4	Glass	12.79	TIO-1	2.56	15.35
I	Glass	12.17	TIO-3	2.44	14.61
J	Glass	11.77	TIO-2	2.36	14.13
K	Glass	10.48	SIO-1	0.44	10.92
KK	Glass	10.44	SIO-1	0.44	10.88
M-3	Chromosorb W	9.09	PMMA	0.91	10.00
O	Chromosorb W	4.72	PAA	0.27	4.99
N	Chromosorb W	8.91	—	—	8.91
G	Glass	13.77	—	—	13.77

All columns were constructed of stainless steel tubing 6.4 mm (0.25 in.) in diameter and approximately 50 cm long.
Glass beads and Chromosorb W were acid washed and DMCS treated.

Table 5—IGC Probe Characteristics

Probe	a_i[a] (sq. ang.)	γ_{lv}^D[a] (mJ/m²)	DN[b]	AN[b]	AN[c]
Pentane	46.1	16.0	—	—	—
Hexane	51.5	18.4	—	—	—
Heptane	57.0	20.3	—	—	—
Octane	62.8	21.3	—	—	—
Benzene	46.0	26.7	0.1	8.2	7.6
$CHCl_3$	44.0	25.0	0.0	23.1	6.4
CCl_4	46.0	26.7	0.0	8.6	6.3
Ether	47.0	15.0	19.2	3.9	1.0
THF	45.0	22.5	20.0	8.0	6.1
Ethyl acetate (E.A.)	48.0	19.6	17.1	9.3	4.0
Acetone	42.5	16.5	17.0	12.5	3.8

(a) a_i and γ_{lv}^D data obtained from reference (12).
(b) AN and DN numbers obtained from reference (10).
(c) AN obtained from reference (11).

Table 6—The Nonpolar (Dispersive) Component of Solid Surface Tension in (mJ/sq m)

Values Obtained from the Current IGC Analysis

Material	Equation (1)	Equation (2)
TIO-1	124.8	122.0
TIO-2	104.3	94.8
TIO-3	50.3	47.7
SIO-1	49.8	49.2
PMMA	40.9	—
PAA................	42.0	—

Values Reported in the Literature

TiO₂................	92, 89, 141[16]	
SiO₂................	100,[17] 75[18]	
PMMA	43[19]	

characteristics is provided in the Results and Discussion Section.

Probes were injected using a 10 µL Hamilton (air tight) syringe. The procedure for this injection consisted of inserting the syringe needle into the head space of a vial containing the desired probe at 20 ± 3°C and drawing 5 µL of vapor into the syringe. At this time, the syringe contained probe vapors and air which were subsequently injected into the column. Following the FID (detector) response on the recorder which indicated the exiting of the probe from the column, traces of the probe remaining in the syringe from the previous injection were introduced into the inlet port. In many cases this procedure still introduced an excess of probe into the column as evidenced by: an overload reading on the recorder, asymmetric peaks, and/or a variation in retention time with probe concentration.

In order to eliminate these responses which are indications of excessive probe concentration and to ensure equilibrium and Henry's law conditions within the column,[13] the syringe was purged with air and re-injected into the column. This procedure was repeated until two reproducible, symmetrical (Gaussian) peaks were obtained. Other researchers have reported that the amount of probe necessary to obtain these conditions is 10^{-4} to 10^{-3} ppm.[14] A least-squares linear regression analysis procedure was followed to fit data relationships to linear equations.

RESULTS AND DISCUSSION

General

The average net retention time and volume of the chemical probes through each of the pigment and polymer-packed columns were determined and applied to the appropriate equations presented in reference (8) in order to determine the various thermodynamic properties of interest. The equations are summarized, along with a presentation and discussion of results. Prior to presenting specific results, a general discussion on probe retention times and column performance is warranted.

Based on previous literature and preliminary in-house experimental efforts, the IGC column characteristics (length, diameter, packing, temperature, and flow rate) were selected and designed to obtain discernible vari-

ations in retention times between the probes. It was desirable to have retention times ranging from 0.2 min up to 2 hr. Responses within this time frame clearly illustrated differences between probes without requiring excessive time for completion of the experiment and, in general, the probe responses were within this time frame. However, several of the probes introduced into the columns had extremely short (<0.2 min) or extremely long (>2 hr) retention times. Those probes with short t_rs were virtually unreactive with the packing material and therefore exited the column quickly. One example is THF through both the TIO-1 and TIO-2 columns. As will be illustrated, these pigments exhibit Lewis base characteristics and will not undergo acid-base interactions with another base such as THF. Therefore, this probe traveled through these columns relatively unaffected.

Several probes had extremely long t_rs through columns. In fact, there is some evidence that they may not have exited the column at all. For example, ethyl acetate and acetone injected into the first TIO-1 column tested (H-1) had surprisingly small peaks at approximately 2 hr. Both of these probes are amphoteric, exhibiting both Lewis acid and base characteristics and therefore able to undergo strong interactions with either acids or bases. It is suspected that these probes irreversibly reacted with TIO-1, causing a major fraction of the injected probe to remain in the column, associated with the pigment surface.

When previously injected probes were re-injected into TIO-1 column H-1 (after ethyl acetate and acetone), their retention times were 10 to 30% shorter than those previously observed (prior to ethyl acetate and acetone). It is suspected that the ethyl acetate and acetone remained associated with TIO-1 and lessened the number of reactive sites on the pigment surface. When two additional TIO-1 columns were constructed and tested, t_n and V_n values of probes other than ethyl acetate and acetone were equivalent to those initially obtained with H-1, thus confirming reproducibility. From this time on, if a polar probe remained in a column for more than 2 hr, a new column was constructed and analyzed, and the use of that probe in the column was discontinued. This permitted reproducible testing of columns in less than two weeks. The inability to use probes which adsorb irreversibly is an obvious limitation of the IGC procedure at this time. This is the reason that larger and more polar probes could not be analyzed in the columns. Further research may uncover methods to overcome this limitation.

$\gamma_{sv}{}^D$, THE NONPOLAR (DISPERSIVE) COMPONENT OF THE SOLID SURFACE TENSION: The nonpolar component of the

Table 7—The Dispersive Work of Adhesion (in mJ/m²) for PMMA and PAA Adsorption onto Titanium Dioxide and Silica Pigments

Filler	PMMA	PAA
TIO-1	143	145
TIO-2	131	132
TIO-3	91	92
SIO-1	90	91

Table 8—The Free Energy of Adsorption of Probes on Pigment Surfaces (in kJ/mol)

Pigment	Probe	70°C (343°K)	80°C (353°K)	90°C (363°K)	100°C (373°K)	110°C (383°K)
TIO-1	Pentane	—	− 14.52	− 13.55	− 12.47	− 11.44
TIO-1	Hexane	—	− 19.44	− 18.21	− 17.12	− 15.95
TIO-1	Heptane	—	− 24.29	− 22.98	− 21.89	− 20.87
TIO-1	Octane	—	—	− 27.76	− 26.59	− 25.73
TIO-1	Benzene	—	− 26.98	− 26	− 25.42	− 24.6
TIO-1	CHC13	—	− 22.09	− 21.02	− 20.25	− 19.48
TIO-1	CC14	—	− 18.8	− 17.83	− 16.86	− 16.17
TIO-1	Ether	—	− 13.67	− 12.95	− 11.81	− 11.25
TIO-3	Hexane	—	− 14.52	− 12.53	− 12.83	− 11.43
TIO-3	Heptane	—	− 17.66	− 16.77	− 15.87	− 14.79
TIO-3	Octane	—	− 20.68	− 19.69	− 18.73	− 17.69
TIO-3	Decane	—	− 26.83	− 25.65	− 24.51	− 23.4
TIO-3	Benzene	—	− 20.77	− 19.76	− 18.78	− 17.8
TIO-3	CHC13	—	− 20.86	− 20.01	− 19.19	− 18.05
TIO-3	CC14	—	− 16.38	− 15.5	− 14.71	− 13.83
TIO-3	Ether	—	− 23.54	− 22.67	− 21.4	− 20.16
TIO-3	THF	—	− 28.88	− 28.06	− 27.03	− 25.69
TIO-3	Eth. Ac.	—	—	− 31.07	− 30.72	− 29.33
TIO-3	Acetone	—	− 29.85	− 28.81	− 27.58	− 26.33
TIO-2	Pentane	− 15.88	—	− 14.1	− 12.87	− 12.82
TIO-2	Hexane	− 21.33	—	− 18.3	− 16.86	− 16.91
TIO-2	Heptane	− 25.79	—	− 22.53	− 21.07	− 21.09
TIO-2	Benzene	—	—	− 27.27	− 26.58	− 26.06
TIO-2	CHC13	− 23.93	—	− 20.74	− 19.27	− 19.01
TIO-2	CC14	− 22.22	—	− 18.76	− 17.47	− 17.09
TIO-2	Ether	− 15.85	—	− 13.55	− 12.32	− 11.9
SIO-1	Pentane	− 9.88	—	—	—	—
SIO-1	Hexane	− 12.7	—	− 10.72	—	—
SIO-1	Heptane	− 15.85	—	− 13.61	− 12.73	− 11.47
SIO-1	Octane	− 19.07	—	− 16.62	− 15.63	− 14.43
SIO-1	Decane	—	—	− 22.64	− 21.25	− 19.87
SIO-1	Benzene	− 17.7	—	− 15.42	− 14.35	− 13.28
SIO-1	CHC13	− 13.47	—	− 11.75	− 10.84	− 9.79
SIO-1	CC14	− 12.98	—	− 11.16	− 10.46	− 7.24
SIO-1	Ether	− 28.2	—	− 25.22	− 23.67	− 22.21
SIO-1	THF	—	—	− 28.6	− 27.09	− 25.72
SIO-1	Eth. Ac.	—	—	—	—	− 26.55
SIO-1	Acetone	—	—	− 26.75	− 25.49	− 23.98

solid surface tension was determined by two methods. The first method is a graphical approach using:

$$RT \, LnV_n = 2N(\gamma_{sv}^D)^{0.5} \, a_i \, (\gamma_{lv}^D)^{0.5} + K \qquad (1)$$

where:
R = gas constant
T = column temperature (°K)
N = Avagodo's number
γ_{sv}^D = dispersive component of the solid-vapor surface tension
γ_{lv}^D = dispersive component of the liquid probe-vapor surface tension
a_i = area which an adsorbed probe molecule covers on the surface of an adsorbent
K = constant

Plotting the left side of equation (1) versus $[a_i(\gamma_{lv}^D)^{0.5}]$ for a series of alkanes yields a linear plot with a slope of $[2N(\gamma_{sv}^D)^{0.5}]$ from which γ_{sv}^D can be determined. The second method is from the empirical equation (2):

$$\gamma_{sv}^D = \frac{[RT \, Ln \, (V_{n2}/V_{n1})]^2}{4 \, N^2 \, a_{CH2}^2 \gamma_{CH2}} \qquad (2)$$

where: V_{n1} = net retention volumne of an alkane

V_{n2} = net retention volume of the succeeding alkane in the homologous series
a_{CH2} = surface area of a CH_2 group (6 angstroms)
γ_{CH2} = surface energy of a CH_2 group as constituted by close packed groups within polyethylene (35.6 mJ m^{-2})

Figure 1 is a plot [RT LnV$_n$] versus [$a_i(\gamma_{lv}^D)^{0.5}$] for TIO-3 at 100°C and it is representative of all of the pigment plots at each of the experimental temperatures. The first observation from this and graphs of the other data was the perfectly linear relationship exhibited by the alkane probes. Correlation coefficients for these functions were typically greater than 99.9, providing a good indication that the chromatography method was being performed properly.

The alkane response lines for each solid over the temperature range tested were relatively parallel, indicating a negligible change in surface tension. This is reasonable since physical and chemical properties of titanium dioxide and silica do not change significantly between 70 and 110°C,[15] and PMMA and PAA surface characteristics
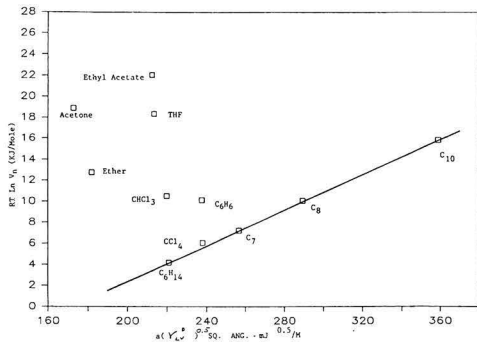
Figure 1—IGC retention volume response for probes on TIO-3
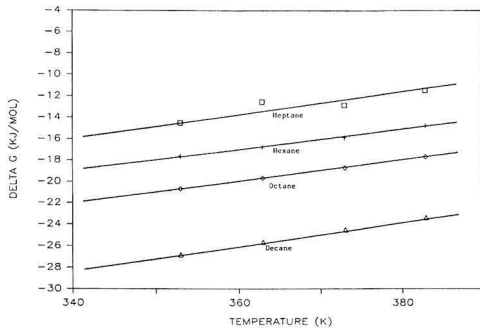
Figure 2—Change in free energy of adsorption for alkane probes on TIO-3
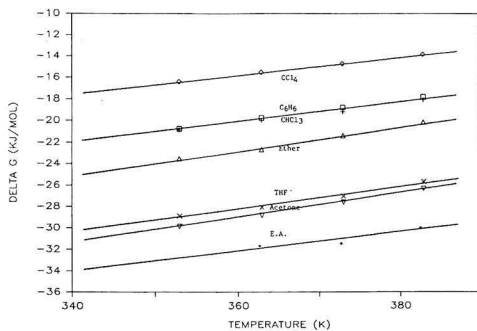
Figure 3—Change in free energy of adsorption for polar probes on TIO-3

should also be stable in the 37.5 to 50°C temperature regime. Therefore, in order to determine γ_{sv}^{D} for each pigment and polymer of interest, an average slope was calculated from the four graphs generated for each (i.e., one graph for each temperature). In addition, V_n for the alkanes through pigment columns were utilized in equation (2) as an alternate approach to determine γ_{sv}^{D}. The resulting values are presented in *Table* 6 along with γ_{sv}^{D} values reported in the literature for TiO_2, SiO_2, and PMMA.

Both approaches taken in this study to determine γ_{sv}^{D} [equations (1) and (2)] yield comparable values. However, the values reported in the literature for TiO_2 and SiO_2 are quite different from those obtained in this study. It should be noted that the TIO-1 and TIO-2 samples were surface treated and that TIO-3 has the only true TiO_2 surface (*Table* 1). Nonetheless, pigment processing parameters and procedures play a critical role in determining surface properties. Small concentrations of foreign species, such as water or other chemicals, can significantly alter surface characteristics. Furthermore, the concentration of surface groups can vary. Therefore, it is not surprising that reported surface energy values for various pigment samples differ. In contrast, polymer surfaces are typically formed from solutions or melts in controlled laboratory conditions prior to surface analysis. This allows the formation of a more consistent surface which is reflected in the agreement of γ_{sv}^{D} values for PMMA, 40.9 and 43 mJ/m², respectively.

From the results in *Table* 6, it is obvious that the TIO-1 and TIO-2 pigments have much higher dispersive surface tensions than TIO-3 and SIO-1. The general relationships can be summarized by:

$$\gamma_{TIO-1}^{D} > \gamma_{TIO-2}^{D} >> \gamma_{TIO-3}^{D} \approx \gamma_{SIO-1}^{D}$$

Since all of the pigment surface tensions are greater than those for the polymers, if it is assumed that the polar surface tension contributions of the pigments are relatively similar, both PMMA and PAA will wet all of the pigments analyzed. However, the difference between polymer and pigment surface tension is significantly greater with TIO-1 and TIO-2, indicating that they have a greater potential for wetting these polymers than TIO-3

Table 9—The Free Energy of Adsorption of Probes on PMMA and PAA (in kJ/mol)				
	37.5°C (310.5°K)	40°C (313°K)	45°C (318°K)	50°C (323°K)
PMMA . . . Octane	− 22.7	− 22.31	− 21.92	− 22.07
PMMA . . . Decane	− 28.2	− 27.55	− 27.04	− 27.32
PMMA . . . CHC13	− 20.83	− 20.56	—	—
PMMA . . . Ether	− 20.7	− 20.18	—	—
PMMA . . . THF	− 25.77	− 25.35	− 24.28	− 24.96
PMMA . . . Eth. Ac.	− 25.34	− 24.97	− 24.45	− 25.05
PMMA . . . Acetone	− 26.41	− 25.7	− 25.84	− 26.62
PAA . . . . . . Octane	− 23.82	− 23.49	− 23.14	− 23.59
PAA . . . . . . Decane	− 29.13	− 28.97	− 28.47	− 28.96
PAA . . . . . . Ether	− 23.44	− 23.27	− 22.47	− 22.41
PAA . . . . . . THF	− 27.85	− 27.52	− 27.18	− 27.48
PAA . . . . . . Eth. Ac.	− 24.97	− 26.25	− 25.62	− 26.42
PAA . . . . . . Acetone	− 25.16	− 26.81	− 26.83	− 27.37

Table 10—Free Energy of Adsorption (kJ/mol) and Work of Adhesion (mJ/m²) for Hexane and Heptane Interactions

Pigment	Free Energy of Adsorption		Work of Adhesion	
	Hexane	Heptane	Hexane	Heptane
TIO-1	− 18.21	− 22.98	96	101
TIO-2	− 18.30	− 22.53	87	92
TIO-3	− 12.53	− 16.77	61	64
SIO-1	− 10.72	− 13.61	61	64

and SIO-1. An increase in wetting usually leads to increased adhesion and yields more consistent dispersion of pigment throughout the polymer phase. In fact, $\gamma_{sv}{}^D$ properties listed in *Table 6* can be used to determine the dispersive work of adhesion, $w_a{}^D$, for PMMA and PAA on the pigments studied (*Table 7*). No conclusions on polar interactions can be drawn from this information, however it is obvious that dispersive attraction forces are much higher with the TIO-1 and TIO-2 pigments and it is suspected that they will have better dispersion and other physical properties when incorporated in a PMMA or PAA matrix. (As will be reported in a subsequent paper,[20] this is the case.) Although there is no apparent correlation between these results and pigment composition as listed in *Table 1*, it is interesting to note that TIO-1 and TIO-2 are marketed by their manufacturers as "high quality" pigments compared to TIO-3. Polar contributions to the adsorption process will be discussed.

It must also be noted that the thermodynamic properties presented herein are normalized per mole of adsorbate and surface area of adsorbent. These properties are real indications of the likelihood, strength, and stability of filler-polymer interactions. However, they do not account for differences in particulate specific surface area (surface area per weight) which can vary dramatically from pigment to pigment. For example, the titanium dioxide specimens have specific surface areas in the 10 to 30 m²/g range while that of SIO-1 is approximately 130 m²/g. Consequently, SIO-1 has up to 13 times more surface area and more surface sites available for adsorption (on a per weight basis) than titanium dioxide.

$\Delta G_a{}^o$, $\Delta H_a{}^o$, $\Delta S_a{}^o$, THE STANDARD FREE ENERGY, ENTHALPY, AND ENTROPY OF ADSORPTION: $\Delta G_a{}^o$ values for adsorption of the chemical probes on the solid surfaces were determined from equation (3):

$$\Delta G_a{}^o = - \ RT \ Ln \ \frac{V_n P_{s,g}}{w \ S_a} \qquad (3)$$

where: $P_{s,g}$ = adsorbate vapor pressure in the gaseous standard state $(1.013 \times 10^5 \ Pa)^{21}$

w = weight of adsorbent in the column

S_a = specific surface area of the adsorbent

The resulting values for fillers and polymers are presented in *Tables 8* and *9*, respectively. A representation of this data for TIO-3 is illustrated in *Figures 2* and *3*. It should be noted that $\Delta G_a{}^o$ for alkanes is solely caused by dispersion forces. $\Delta G_a{}^o$ is negative for every probe-particle/polymer interaction investigated, thereby fulfilling the thermodynamic requirement for spontaneous interac-

tion and indicating a stable association. For alkane probes through pigment columns, the change in free energy of adsorption increases linearly with the length of the molecular chain. This result is in agreement with previous reports[22–24] and indicates good performance of the columns.

By comparing the electron donor-acceptor numbers presented in *Table 5* with the $\Delta G_a{}^o$ results, a general trend of acid-base interactions becomes evident. For the pigments investigated, TIO-1 and TIO-2 have lower (more negative) free energies of adsorption with acidic probes (benzene, methyl chloroform, and carbon tetrachloride), indicating a more favorable acid-base interaction. This result provides evidence that the TIO-1 and TIO-2 surfaces are Lewis bases (electron donors). *Table 1* illustrates that these pigments have an alumina surface treatment which has been found to be primarily basic.[25] In contrast, TIO-3 and SIO-1 have lower free energies with basic probes indicating that they have acidic sur-

Table 11—The Enthalpy and Entropy of Adsorption of Probes on Pigment Surfaces. Enthalpy Results Are in mJ/mol and Entropy Results Are in mJ/mol K

Pigment	Probe	ΔH (kJ/mol)	ΔS (kJ/mol K)	Correlation Coefficient (%)
TIO-1	Pentane	− 50.97	− 0.1032	99.98
TIO-1	Hexane	− 60.22	− 0.1156	99.98
TIO-1	Heptane	− 64.28	− 0.1135	99.83
TIO-1	Octane	− 65.55	− 0.1015	99.61
TIO-1	Benzene	− 51.45	− 0.07	99.51
TIO-1	CHC13	− 52.36	− 0.086	99.64
TIO-1	CC14	− 50.02	− 0.089	99.7
TIO-1	Ether	− 43.33	− 0.084	99.21
TIO-3	Hexane	− 45.84	− 0.09	90.55
TIO-3	Heptane	− 51.27	− 0.095	99.88
TIO-3	Octane	− 55.74	− 0.099	99.99
TIO-3	Decane	− 67.16	− 0.114	99.99
TIO-3	Benzene	− 55.67	− 0.099	100
TIO-3	CHC13	− 53.57	− 0.093	99.68
TIO-3	CC14	− 46.16	− 0.084	99.98
TIO-3	Ether	− 63.93	− 0.114	99.67
TIO-3	THF	− 66.42	− 0.106	99.4
TIO-3	Eth. Ac.	− 62.82	− 0.087	94.53
TIO-3	Acetone	− 71.53	− 0.118	99.91
TIO-2	Pentane	− 43.86	− 0.082	97.42
TIO-2	Hexane	− 61.5	− 0.118	96.2
TIO-2	Heptane	− 68.36	− 0.125	96.22
TIO-2	Benzene	− 49.2	− 0.061	99.67
TIO-2	CHC13	− 67.97	− 0.129	97.63
TIO-2	CC14	− 67.65	− 0.133	97.57
TIO-2	Ether	− 50.93	− 0.103	98.85
SIO-1	Hexane	− 46.66	− 0.099	—ᵃ
SIO-1	Heptane	− 52.87	− 0.108	99.88
SIO-1	Octane	− 58.57	− 0.115	99.93
SIO-1	Decane	− 72.91	− 0.138	100
SIO-1	Benzene	− 55.61	− 0.111	99.98
SIO-1	CHC13	− 44.81	− 0.091	99.9
SIO-1	CC14	− 58.41	− 0.131	93.55
SIO-1	Ether	− 79.69	− 0.15	100
SIO-1	Eth. Ac.	− 80.85	− 0.144	99.96
SIO-1	Acetone	− 77.07	− 0.139	99.86

(a) Linear regression based on two data points.
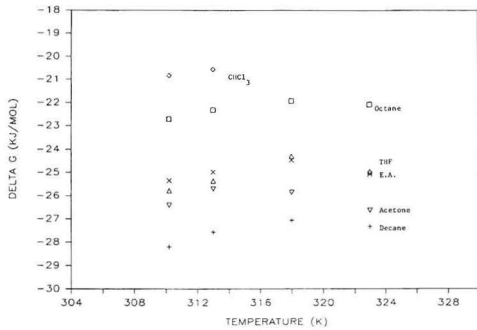
Figure 4—Change in free energy of adsorption for probes on PMMA
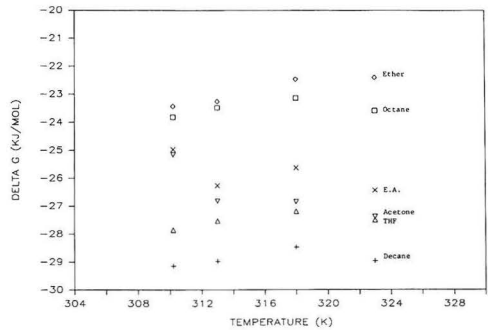
Figure 5—Change in free energy of adsorption for probes on PAA

Table 12—The Polar Component of the Free Energy of Adsorption of Probes on Pigment Surfaces (in kJ/mol)

Pigment	Probe	70°C (343°K)	80°C (353°K)	90°C (363°K)	100°C (373°K)	110°C (383°K)
TIO-1	Pentane	0	0	0	0	0
TIO-1	Hexane	0	0	0	0	0
TIO-1	Heptane	0	0	0	0	0
TIO-1	Octane	0	0	0	0	0
TIO-1	Benzene	—	− 5.24	− 5.46	− 6.03	− 6.21
TIO-1	CHC13	—	− 2.77	− 2.87	− 3.25	− 3.5
TIO-1	CC14	—	2.98	2.77	2.57	2.28
TIO-1	Ether	—	0.48	0.09	0.102	− 0.043
TIO-3	Hexane	0	0	0	0	0
TIO-3	Heptane	0	0	0	0	0
TIO-3	Octane	0	0	0	0	0
TIO-3	Decane	0	0	0	0	0
TIO-3	Benzene	—	− 4.83	− 4.73	− 4.41	− 4.53
TIO-3	CHC13	—	− 6.49	− 6.5	− 6.31	− 6.28
TIO-3	CC14	—	− 0.4	− 0.45	− 0.31	− 0.54
TIO-3	Ether	—	− 12.55	− 12.42	− 11.76	− 11.58
TIO-3	THF	—	− 15.09	− 15.08	− 14.72	− 14.47
TIO-3	Eth. Ac.	—	—	− 18.16	− 18.49	− 18.18
TIO-3	Acetone	—	− 19.7	− 19.34	− 18.73	− 18.54
TIO-2	Pentane	0	0	0	0	0
TIO-2	Hexane	0	0	0	0	0
TIO-2	Heptane	0	0	0	0	0
TIO-2	Benzene	—	—	− 7.03	− 7.23	− 7.08
TIO-2	CHC13	− 3.11	—	− 2.62	− 1.95	− 2.15
TIO-2	CC14	1.14	—	1.53	1.94	1.93
TIO-2	Ether	− 0.34	—	0.05	0.63	0.43
SIO-1	Pentane	0	0	0	0	0
SIO-1	Hexane	0	0	0	0	0
SIO-1	Heptane	0	0	0	0	0
SIO-1	Octane	0	0	0	0	0
SIO-1	Decane	0	0	0	0	0
SIO-1	Benzene	− 3.75	—	− 3.34	− 3.13	− 3.16
SIO-1	CHC13	− 1.18	—	− 1.2	− 1.09	− 1.12
SIO-1	CC14	1.06	—	0.97	0.79	2.91
SIO-1	Ether	− 18.8	—	− 17.97	− 17.06	− 16.65
SIO-1	THF	—	—	− 18.62	− 17.88	− 17.58
SIO-1	Acetone	—	—	− 20.3	− 19.66	− 19.2

faces. TIO-3 and SIO-1 are almost completely TiO_2 and SiO_2, respectively. Both of these compounds have been found previously to be primarily acidic.[25,26]

It should be noted that $\Delta G_a°$ and w_a results (*Table* 10) illustrate that TIO-1 and TIO-2 wet and adhere to alkanes significantly better than TIO-3 and SIO-1. This confirms conclusions from γ_{sv}^D data which were presented and discussed in the previous section.

Responses through the polymer columns were not as predictable and were partially surprising. The first observation from *Figures* 4 and 5 is that the relationship between change in free energy of adsorption and temperature does not exhibit the linearity observed with the pigment results. Although DSC and TGA data indicate no changes in these materials over this temperature range, including glass transition, it is suspected that they are far more sensitive to thermal environment than the TiO_2 and SiO_2 materials. This would explain the more linear relationship observed with these latter materials.

A second observation is that basic probes had longer retention times and lower free energy changes than acidic probes with PAA. This is expected since PAA is an acidic material. However, the same trend is true for PMMA which has been reported by a number of researchers as being primarily basic. One explanation for this may be the following. IGC analysis of simply Chromosorb W (Column N) indicated that it is acidic. This is not surprising since it was acid washed prior to analysis. Although it was initially believed that the PMMA titrated onto the Chromosorb W covered the surface, the Chromosorb W has a rough surface and may be somewhat porous. Therefore, it is possible that the PMMA did not saturate and cover the surface and that the acidic nature of the Chromosorb W is controlling the IGC retention time through the PMMA columns.

In all cases the amphoteric probes had long retention times and lower (more negative) changes in free energy. This is expected for two reasons which are interrelated. First, these probes inherently exhibit both acidic and basic tendencies and, second, due to their amphoteric nature, they can easily self-associate. Therefore, they should undergo spontaneous and relatively strong interactions with both acidic and basic surfaces.

The $\Delta G_a°$ versus T graphs are linear for all of the pigment-probe systems analyzed. Correlation coefficients were typically greater than 99%. This provided a convenient means for determining the respective standard enthalpies and entropies of adsorption using the classic Gibbs free energy equation:

$$\Delta G = \Delta H - T\Delta S \qquad (4)$$

$\Delta H_a°$ and $\Delta S_a°$ were determined as the intercepts and slopes in the ΔG versus T graphs. These values are presented in *Table* 11. The enthalpies of adsorption are all negative and ranged from -80 to -43 kJ/mol, thus indicating exothermic interactions. This is a significant heat of interaction considering the strength of a hydrogen bond can be as high as 30 kJ/mol. The enthalpy data also agrees with the acid-base interaction trends observed with the free energy, namely TIO-1 and TIO-2 are basic while TIO-03 and SIO-1 are acidic. Since enthalpy of interac-

Table 13—The Polar Component of the Free Energy of Adsorption of Probes on PMMA and PAA (in mJ/mol)

Polymer	Probe	37.5°C (310.5°K)	40°C (313°K)	45°C (318°K)	50°C (323°K)
PMMA	CHCL3	-3.87	-3.6	—	—
PMMA	Ether	-6.78	-6.14	—	—
PMMA	THF	-9.32	-8.89	-8.16	-8.43
PMMA	Eth. Ac.	-8.98	-8.59	-8.4	-8.59
PMMA	Acetone	-13.22	-12.37	-12.79	-13.11
PAA	Ether	-9.56	-8.86	-7.46	-5.56
PAA	THF	-11.57	-10.66	-9.65	-8.03
PAA	Eth. Ac.	-10.29	-9.46	-8.17	-7.07
PAA	Acetone	-13.92	-13.14	-12.57	-11.1

tion is directly related to the strength of the resulting bond, and a decrease in free energy will yield a spontaneous process, there is ample evidence that the acid-base interactions occurring in these studies are forming strong and stable associations between pigment and adsorbate.

Values of $\Delta S_a°$ for pigment-probe interactions range from -0.144 to -0.061 kJ/mol-K but, in general, entropy values are relatively consistent between systems with an average value of -0.106 ± 0.021 kJ/mol-K. The fact that the magnitude of entropy change is similar for the

Table 14—The Polar Component of the Enthalpy and Entropy of Adsorption of Probes on Pigment Surfaces. Enthalpy Results Are in kJ/mol and Entropy Results Are in kJ/mol K

Pigment	Probe	ΔH(a-sp) (kJ/mol)	ΔS(a-sp) (kJ/mol K)	Correlation Coefficient
TIO-1	Benzene	7.07	0.035	97.78
TIO-1	CHC13	6.36	0.0257	97.94
TIO-1	CC14	11.11	0.023	99.61
TIO-1	Ether	5.88	0.0156	89.34
TIO-3	Benzene	-9.11	-0.012	82.9
TIO-3	CHC13	-9.41	-0.008	91.11
TIO-3	CC14	0.61	0.003	37.62
TIO-3	Ether	-25.22	-0.036	96.18
TIO-3	THF	-23.01	-0.0222	95.29
TIO-3	Eth. Ac.	-17.9	-0.001	5.4
TIO-3	Acetone	-34.13	-0.0412	98.27
TIO-2	Benzene	-6.18	0.002	24.02
TIO-2	CHC13	-12.6	-0.028	91.51
TIO-2	CC14	-6.24	-0.022	96.5
TIO-2	Ether	-8.07	-0.023	90.01
SIO-1	Benzene	-9.17	-0.016	95.37
SIO-1	CHC13	-1.89	-0.002	67.62
SIO-1	CC14	-11.34	-0.034	60.2
SIO-1	Ether	-37.92	-0.056	98.73
SIO-1	THF	-37.42	-0.052	97.14
SIO-1	Acetone	-40.23	-0.054	99.56

Enthalpies of Adsorption Determined from the Drago Equation.[8,28] E and C parameters obtained from the literature[29]

TiO2	Benzene	-16.1		
TiO2	Ether	-37.1		
TiO2	THF	-41.5		
TiO2	Eth. Ac.	-30.6		
TiO2	Acetone	-33.6		
SiO2	Benzene	-15.5		
SiO2	Ether	-33.1		
SiO2	THF	-38.1		
SiO2	Acetone	-29.1		

Figure 6—Change in free energy of adsorption due to polar interactions between probes and TIO-3

Figure 9—Change in free energy of adsorption due to dispersive interactions between probes and TIO-3

Figure 7—Change in free energy of adsorption due to polar interactions between probes and PMMA

Figure 10—Change in free energy of adsorption due to dispersive interactions between probes and PMMA
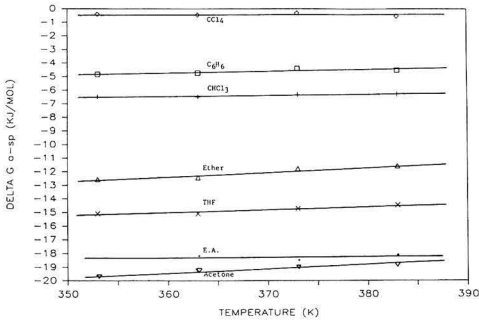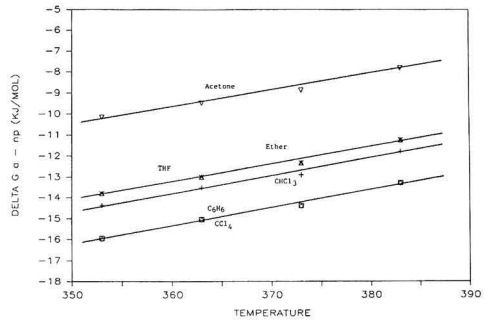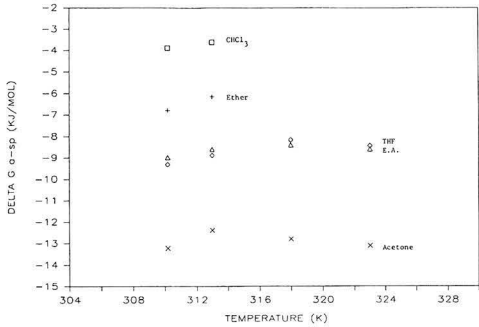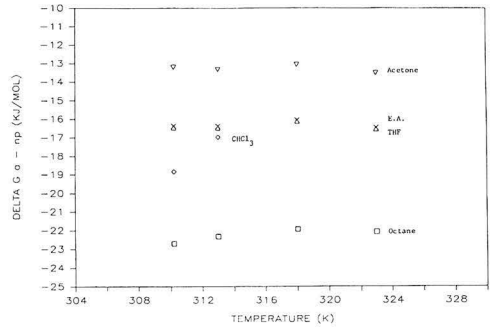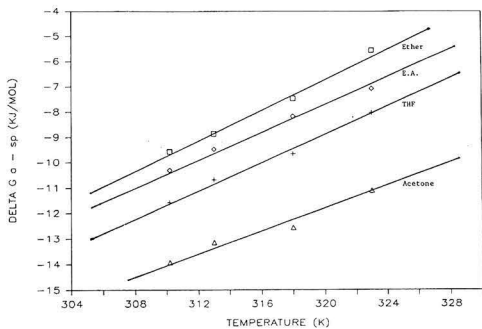

Figure 8—Change in free energy of adsorption due to polar interactions between probes and PAA
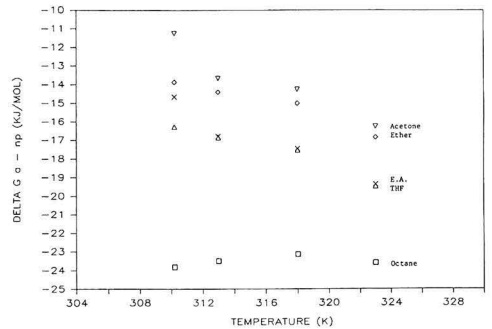
Figure 11—Change in free energy of adsorption due to dispersive interactions between probes and PAA

Table 15—The Nonpolar (Dispersive) Component of the Free Energy of Adsorption of Probes on Pigment Surfaces (in kJ/mol)

Pigment	Probe	70°C (343°K)	80°C (353°K)	90°C (363°K)	100°C (373°K)	110°C (383°K)
TIO-1............	Pentane	—	−14.52	−13.55	−12.47	−11.44
TIO-1............	Hexane	—	−19.44	−18.21	−17.12	−15.95
TIO-1............	Heptane	—	−24.29	−22.98	−21.89	−20.87
TIO-1............	Octane	—	—	−27.76	−26.59	−25.73
TIO-1............	Benzene	—	−21.74	−20.54	−19.39	−18.39
TIO-1............	CHCl3	—	−19.32	−18.15	−17	−15.98
TIO-1............	CCl4	—	−21.78	−20.6	−19.43	−18.45
TIO-1............	Ether	—	−14.15	−13.04	−11.912	−11.21
TIO-3............	Hexane	—	−14.52	−12.53	−12.83	−11.43
TIO-3............	Heptane	—	−17.66	−16.77	−15.87	−14.79
TIO-3............	Octane	—	−20.68	−19.69	−18.73	−17.69
TIO-3............	Decane	—	−26.83	−25.65	−24.51	−23.4
TIO-3............	Benzene	—	−15.94	−15.03	−14.37	−13.27
TIO-3............	CHCl3	—	−14.37	−13.51	−12.88	−11.77
TIO-3............	CCl4	—	−15.98	−15.05	−14.4	−13.29
TIO-3............	Ether	—	−10.99	−10.25	−9.64	−8.58
TIO-3............	THF	—	−13.79	−12.98	−12.31	−11.22
TIO-3............	Eth. Ac.	—	—	−12.91	−12.23	−11.15
TIO-3............	Acetone	—	−10.15	−9.47	−8.85	−7.79
TIO-2............	Pentane	−15.88	—	−14.1	−12.87	−12.82
TIO-2............	Hexane	−21.33	—	−18.3	−16.86	−16.91
TIO-2............	Heptane	−25.79	—	−22.53	−21.07	−21.09
TIO-2............	Benzene	—	—	−20.24	−19.35	−18.98
TIO-2............	CHCl3	−20.82	—	−18.12	−17.32	−16.86
TIO-2............	CCl4	−23.36	—	−20.29	−19.41	−19.02
TIO-2............	Ether	−15.51	—	−13.6	−12.95	−12.33
SIO-1............	Pentane	−9.88	—	—	—	—
SIO-1............	Hexane	−12.7	—	−10.72	—	—
SIO-1............	Heptane	−15.85	—	−13.61	−12.73	−11.47
SIO-1............	Octane	−19.07	—	−16.62	−15.63	−14.43
SIO-1............	Decane	—	—	−22.64	−21.25	−19.87
SIO-1............	Benzene	−13.95	—	−12.08	−11.22	−12.36
SIO-1............	CHCl3	−12.29	—	−10.55	−9.75	−6.206
SIO-1............	CCl4	−14.04	—	−12.13	−11.25	—
SIO-1............	Ether	−9.4	—	−7.25	−8.99	−6.76
SIO-1............	THF	—	—	−9.98	−9.21	−10.27
SIO-1............	Eth. Ac.	—	—	—	—	−10.18
SIO-1............	Acetone	—	—	−6.45	−5.83	−6.71

different systems is a reasonable result considering that this is a configurational entropy effect and adsorbates should undergo similar configurational changes going from the vapor or liquid phases to an adsorbed state, regardless of the adsorbent. Although these entropy values appear small, if we quantify the [TΔS] function of equation (4) at a temperature of 363°K, this term has an average value of −38.5 kJ/mol which is a significant contribution to the free energy of adsorption (*Table* 8). In fact, the entropy change has a detrimental effect on the change in free energy in that it is negative, indicating a decrease in entropy. Considering the phenomenon of adsorption, this is to be expected. The system is going from a less ordered phase (gaseous) to a more ordered adsorbed phase. An analogy can be drawn with condensation of a vapor to a liquid or solid in which entropy is significantly decreased. For example, Trouton's rule empirically states that the entropy of condensation equals −0.088 kJ/mol − K.[27]

$\Delta G_{a\text{-}sp}°$, $\Delta H_{a\text{-}sp}°$, $\Delta S_{a\text{-}sp}°$, THE POLAR CONTRIBUTIONS TO THE STANDARD FREE ENERGY, ENTHALPY, AND ENTROPY OF ADSORPTION: *Figure* 1 illustrates that in most cases polar probes have responses significantly above the reference alkane response line due to longer retention times and higher retention volumes. These longer t_n and V_n values are a direct result of polar intermolecular interactions. The polar contribution to the standard free energy of adsorption can be determined from:

$$\Delta G_{a\text{-}sp}° = - RT \, Ln(V_n/V_n^{ref}) \qquad (5)$$

where: V_n = net retention volume of the adsorbing probe
V_n^{ref} = net retention volume on the reference alkane line

Values for $\Delta G_{a\text{-}sp}°$ are presented in *Tables* 12 and 13 for the pigment and polymer samples, respectively. This data is illustrated for TIO-3, PMMA, and PAA in *Figures* 6-8, respectively. It should be noted that $\Delta G_{a\text{-}sp}°$ for all of the alkane probes is inherently zero due to their lack of polarity. In addition, the polar contributions to the free energy change for adsorption of the polar probes are significantly higher (less negative) than the total change in free energy. This indicates that the dispersive contributions are greater than the polar contributions as Fowkes has suggested.[19] In fact, several $\Delta G_{a\text{-}sp}°$ values were greater than 0 kJ/mole, indicating nonspontaneous (repulsive) interactions. This mainly occurred with base-base

Table 16—The Nonpolar (Dispersive) Component of the Free Energy of Adsorption of Probes on PMMA and PAA (in kJ/mol)

Polymer	Probe	37.5°C (310.2°K)	40°C (313°K)	45°C (318°K)	50°C (323°K)
PMMA	. . . Octane	− 22.7	− 22.31	− 21.92	− 22.07
PMMA	. . . Decane	− 28.2	− 27.55	− 27.04	− 27.32
PMMA	. . . CHCl3	− 18.83	− 16.96	—	—
PMMA	. . . Ether	− 13.92	− 14.04	—	—
PMMA	. . . THF	− 16.45	− 16.46	− 16.12	− 16.53
PMMA	. . . Eth. Ac.	− 16.36	− 16.38	− 16.05	− 16.46
PMMA	. . . Acetone	− 13.19	− 13.33	− 13.05	− 13.51
PAA	. . . . . . Octane	− 23.82	− 23.49	− 23.14	− 23.59
PAA	. . . . . . Decane	− 29.14	− 28.97	− 28.47	− 28.96
PAA	. . . . . . Ether	− 13.88	− 14.41	− 15.01	− 16.85
PAA	. . . . . . THF	− 16.28	− 16.86	− 17.53	− 19.46
PAA	. . . . . Eth. Ac.	− 14.67	− 16.79	− 17.45	− 19.35
PAA	. . . . . . Acetone	− 11.24	− 13.67	− 14.27	− 16.27

Table 17—The Nonpolar (Dispersive) Component of the Enthalpy and Entropy of Adsorption of Probes on Pigment Surfaces

Pigment	Probe	ΔH(a-np) (kJ/mol)	ΔS(a-np) (kJ/mol K)	Correlation Coefficient
TIO-1	. . . . . . Pentane	− 50.97	− 0.1032	99.98
TIO-1	. . . . . . Hexane	− 60.22	− 0.1156	99.98
TIO-1	. . . . . . Heptane	− 64.28	− 0.1135	99.83
TIO-1	. . . . . . Octane	− 65.55	− 0.1015	99.61
TIO-1	. . . . . . Benzene	− 61.23	− 0.112	99.92
TIO-1	. . . . . . CHCl3	− 58.72	− 0.112	99.95
TIO-1	. . . . . . CCl4	− 61.13	− 0.112	99.91
TIO-1	. . . . . . Ether	− 49.19	− 0.099	99.48
TIO-3	. . . . . . Hexane	− 45.84	− 0.09	90.55
TIO-3	. . . . . . Heptane	− 51.27	− 0.095	99.88
TIO-3	. . . . . . Octane	− 55.74	− 0.099	99.99
TIO-3	. . . . . . Decane	− 67.16	− 0.114	99.99
TIO-3	. . . . . . Benzene	− 46.56	− 0.087	99.57
TIO-3	. . . . . . CHCl3	− 44.15	− 0.084	99.43
TIO-3	. . . . . . CHl4	− 46.77	− 0.087	99.54
TIO-3	. . . . . . Ether	− 38.72	− 0.078	99.32
TIO-3	. . . . . . THF	− 43.41	− 0.084	99.5
TIO-3	. . . . . . Eth. Ac.	− 44.92	− 0.088	99.15
TIO-3	. . . . . . Acetone	− 37.4	− 0.077	99.19
TIO-2	. . . . . . Pentane	− 43.86	− 0.082	97.42
TIO-2	. . . . . . Hexane	− 61.5	− 0.118	96.2
TIO-2	. . . . . . Heptane	− 68.36	− 0.125	96.22
TIO-2	. . . . . . Heptane	− 43.02	− 0.063	97.27
TIO-2	. . . . . . CHCl3	− 55.37	− 0.101	97.83
TIO-2	. . . . . . CCl4	− 61.41	− 0.112	97.17
TIO-2	. . . . . . Ether	− 42.87	− 0.08	99.37
SIO-1	. . . . . . Hexane	− 46.66	− 0.099	—[a]
SIO-1	. . . . . . Heptane	− 52.87	− 0.108	99.88
SIO-1	. . . . . . Octane	− 58.57	− 0.115	99.93
SIO-1	. . . . . . Decane	− 72.91	− 0.138	100
SIO-1	. . . . . . Benzene	− 30.63	− 0.05	72.69
SIO-1	. . . . . . CHCl3	− 60.31	− 0.138	92.43
SIO-1	. . . . . . CCl4	− 46.05	− 0.093	99.98
SIO-1	. . . . . . Ether	− 26.44	− 0.05	66.37
SIO-1	. . . . . . THF	− 4.41	− 0.145	26.46
SIO-1	. . . . . . Acetone	− 1.48	− 0.012	28.75

(a) Linear regression based on two data points.

and acid-acid pairs. However, as observed with previous results, acid-base interactions were generally favorable. One notable exception is that the acidic CCl_4 does not undergo much interaction with any of the pigments or polymers, including the basic TIO-1 and TIO-2. This lack of polar interaction is probably due to the geometric and electronic symmetry of the CCl_4 molecule which would prefer not to donate or accept electrons in the scenarios presented.

All of the $\Delta G_{a\text{-}sp}°$ versus T plots for pigment-probe interactions are linear, once again permitting calculation of ΔH and ΔS values from equation (4). These results are presented in *Table 14* along with $\Delta H_{a\text{-}sp}°$ for TiO_2 and SiO_2 determined from Drago's equation.[28] Although the values obtained from the two studies are not in total agreement, there is a general trend in the strength of adsorption and the respective acid-base interactions between fillers and probes.

There is a slightly different trend in the entropy effects for polar interactions compared to the total change in entropy of adsorption. Values of $\Delta S_{a\text{-}sp}°$ are still low and these changes are negative for TIO-2, TIO-3, and SIO-1 with coinciding exothermic enthalpy values. However, entropy changes are positive for TIO-1 and the enthalpy values are endothermic. This TIO-1 response is contrary to other results and was unexpected. These results indicate that polar associations with TIO-1 are not favorable. As expected, the polar enthalpy contributions are only a fraction of those for the total enthalpy of adsorption. The acid-base interaction trends are still evident and enthalpy values for these interactions tend to be significantly higher than acid-acid and base-base pairs. In addition, amphoteric probes are observed to interact with both the basic and acidic pigments.

$\Delta G_{a\text{-}np}°$, $\Delta H_{a\text{-}np}°$, $\Delta S_{a\text{-}np}°$, THE NONPOLAR (DISPERSIVE) CONTRIBUTIONS TO THE STANDARD FREE ENERGY, ENTHALPY, AND ENTROPY OF ADSORPTION: The total free energy of adsorption is the sum of both nonpolar and polar contributions. Therefore, the nonpolar contributions can be determined from:

$$\Delta G_{a\text{-}np}° = \Delta G_a° - \Delta G_{a\text{-}sp}° \qquad (6)$$

The values calculated for $\Delta G_{a\text{-}np}°$ are presented in *Tables* 15 and 16. *Figures* 9-11 illustrate the $\Delta G_{a\text{-}np}°$ versus temperature graphs for TIO-3, PMMA, and PAA, respectively. Since alkane probes cannot undergo polar interactions $\Delta G_{a\text{-}np}° = \Delta G_a°$, $\Delta H_{a\text{-}np}° = \Delta H_a°$, and $\Delta S_{a\text{-}np}° = \Delta S_a°$. As previously mentioned, the change in free energy becomes more negative with increasing molecular weight for the alkanes. The $\Delta G_{a\text{-}np}°$ values for the polar probes were comparable to those for the alkanes. As might be expected, more symmetrical molecules such as benzene, carbon tetrachloride, and chloroform had lower $\Delta G_{a\text{-}np}°$. This is due to the greater effect London dispersion (nonpolar) forces have on these molecules. It should be noted that although this trend is observed with all of the pigments, it does not overshadow acid-base interactions. That is, the total change in free energy and enthalpy of adsorption results still display acid-base interaction trends.

The $\Delta G_{a-np}°$ versus temperature plots were linear for all of the pigment-probe graphs except the 383°K data points on the SIO-1 graph. Since these points were confirmed several times, it is believed that they are valid. One explanation for this trend may be a removal of loosely bound water on the silica surface at temperatures above 100°C, altering the surface characteristic to some extent. It is not clear why this would decrease the nonpolar component of free energy. These data points made analysis of the SIO-1 data ($\Delta H_{a-np}°$ and $\Delta S_{a-np}°$) difficult. Nonetheless, $\Delta H_{a-np}°$ and $\Delta S_{a-np}°$ are provided in *Table 17*. Once again, entropy changes are small and negative. The average entropy change for the probes on TIO-1, TIO-2, and TIO-3, (SIO-1 excluded) was -0.100 ± 0.017 kJ/mol-K.

CONCLUSIONS

Inverse gas chromatography was applied to and found useful in the characterization of the dispersive component of the surface energy of both polymers and solid particles. This new application of the technique to a study involving filler particles extends available analysis to the calculation of the thermodynamic properties of reversible particle-polymer interactions. Specifically, IGC and thermodynamic theories indicated that retention times of nonpolar and polar reagents, called probes, through a chromatography column packed with a solid material of interest can be used to determine: solid surface energies; free energy, enthalpy, and entropy of adsorption; and work of adhesion. Available theories to calculate the nonpolar (dispersive) and polar contributions to these surface interactions were applied. The values obtained from the IGC technique and the associated theory were comparable with those obtained from other techniques (infrared spectroscopy and calorimetry) and Drago's empirical approach. The nonpolar (dispersive) forces were found to provide a large contribution to the total free energy and enthalpy of adsorption, even with highly polar species. Typically, acid-base pairs had favorable interaction while acid-acid and base-base pairs were less likely to have strong interaction. Alumina was found to be basic; silica, titania, and poly(acrylic acid) were found to be acidic. In contrast to previous reports, poly(methyl methacrylate) was found to be acidic. Amphoteric adsorbates underwent extensive interactions with both acids and bases due to their mutual compatibility and their tendency to self-associate. Although acid-base trends were discernible with the polymer materials (PAA and PMMA), they did not display a linear relationship between free energy of adsorption and temperature as did silica and titanium dioxide. Irreversible interactions could not be analyzed with the existing technique because these probes have an infinite retention time within the column.

Entropies of adsorption were negative. This reduction in entropy indicates a conformational change from the gaseous state to a more restricted adsorbed configuration (i.e., firmly attached to a solid surface), similar to that for condensation. This contribution to the total free energy change was approximately 30% of the enthalpy changes.

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10/92

Suitability of ASTM Test Method D 2369 for Determination of VOC in High Solids Coatings

B. Ancona, R. Alexander, J. Aviles, J. Bove, R. Granata, P. Kuzma, P. Peterson,
C. Pohan, D. Schober, and N. Shearer

Philadelphia Society for Coatings Technology
Technical Committee

The Technical Committee of the Philadelphia Society for Coatings Technology conducted a "round-robin" study of ASTM D 2369-89, the method used to determine volatile organic content (VOC) in coatings. Initially seven laboratories undertook the testing of five coatings. Two of these coatings were single component and the remaining three were two-component, high solids coatings.

An analysis of variance was performed on the data obtained. The results indicated that the procedure can produce reliable results, when single-component coatings are tested. However, data from two-component coatings yielded higher results than expected with large standard deviations.

INTRODUCTION

Among the many government regulations with which the formulators and manufacturers of coatings must comply is the Maximum Allowable Volatile Organic Content of coatings or "VOC." This simple calculation on the formulator's speadsheet has become the cause of extensive changes in the coatings industry. Increased awareness of environmental issues has led to aggressive enforcement of regulations. Large civil penalties for violations are not uncommon.[1] No longer can a product be formulated to meet certain application properties alone. Often the main consideration becomes the product's compliance with VOC regulations. Presently, meetings are taking place between government regulators and the manufacturers of coatings and raw materials to establish a national ruling on VOC in architectural coatings.[2]

The members of the Technical Committee of the Philadelphia Society for Coatings Technology have become concerned with the suitability of ASTM D 2369 for all coatings now produced. The membership set out to test the applicability of this method on several different coating technologies, including one- and two-component systems.

Satisfying the VOC regulations may appear straightforward, particularly if the theoretical calculation of VOC is assumed to be valid. This assumption is made by many coatings manufacturers. Can the manufacturer rely on this computation to produce compliant coatings? One must assume that the ASTM procedure or EPA Method 24, which is similar, yields results that will be equivalent to the calculated values. The validity of the theoretical VOC calculations will not be dealt with, as it is beyond the scope of the project undertaken by the Technical Committee.

Most manufacturers believe ASTM D 2369 to be the only method to determine VOC that will satisfy government regulators. The EPA states that: "VOC may be measured by a reference method, an equivalent method, or an alternative method or by procedures specified under any subpart."[3] If data can be offered that proves another procedure more appropriate for a particular material, the manufacturer may present this evidence and obtain approval for the procedure.[4] This option should be considered, when another procedure can be shown to produce data that is more accurate or reproducible.

Those applying the test procedure to their coatings must be alert to the difficulties they may encounter. Because of the variability of the test method, results obtained are often misleading. A coating may appear to pass, when it is noncompliant. Or conversely, coatings that fail the test may satisfy the regulations. A manufacturer cannot depend on one set of duplicate determinations to insure compliance. The VOC of a product should be monitored in the same way that other physical tests are, in the course of a product's production. Tracking this data can be a very valuable tool in

Table 1—Raw Data Obtained in Study of ASTM D 2369-89

		Coating 1			Coating 2			Coating 3			Coating 4			Coating 5		
		% Nonvolatile D 2369-89 Method														
		1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
A	1	36.43	36.59	36.03	26.87	29.65	26.99	25.96	25.80	25.13	14.20	12.51	13.35	29.61	29.68	29.49
	2	35.99	35.18	36.02	26.88	27.35	26.84	25.43	25.67	25.55	13.71	14.36	13.61	29.88	30.16	28.70
B	1	33.94	33.90	33.67	25.92	27.73	28.09	25.47	25.30	24.88	11.04	11.20	11.56	28.85	28.81	28.10
	2	34.46	34.55	34.20	26.10	26.55	26.19	24.70	25.12	24.47	9.61	9.84	9.63	29.14	29.06	29.13
C	1	38.02	38.06	40.46	26.84	27.84	27.36	26.13	26.02	26.16	13.34	13.35	13.16	30.05	29.85	30.11
	2	36.60	35.73	36.32	26.54	26.60	26.94	24.94	25.13	24.94	12.05	12.03	12.20	29.03	28.99	29.05
D	1	36.63	36.46	36.81	28.75	27.61	29.47	25.45	25.77	25.23	14.19	14.32	13.16	29.61	29.61	29.64
	2	36.85	37.21	36.51	28.63	28.46	28.94	25.36	24.77	25.42	13.28	13.46	13.08	29.39	28.52	28.57
		% Nonvolatile Air Dry Method														
A	1	35.21	34.84	33.44	23.03	22.97	22.65	24.76	24.36	24.78	7.88	8.43	7.49	28.28	27.86	28.09
	2	34.67	34.55	33.86	22.88	23.08	23.38				8.84	8.62	7.47	27.4	27.75	27.59

controlling process variables, as well as raw material consistency. The coatings manufacturer that does not perform this test can be misled in areas of compliance, as well as product quality and consistency.

Much has changed in coatings technology since concern with VOC first became an issue. Although work has been done to establish a valid test method, the advent of high solids, multiple component, and cure by heat and radiation, have posed many questions concerning the universal applicability of the method.

Members of the Society's Technical Committee felt that there could be several coating technologies, whose calculated VOC would not be confirmed by D 2369. Five coatings were chosen representing different coating chemistries. The coatings studied were a very small sampling of the systems that might prove challenging to the analyst measuring VOC.

The coatings selected for the study were all solvent systems and were samples supplied by manufacturers of formulations, either in production or considered for production. Because of proprietary considerations, the formulations of these coatings are not available for publication. However, these formulations are in the possession of the Technical Committee, along with the manufacturers' data on theoretical or calculated VOC.

EXPERIMENTAL PLAN

Representatives of seven laboratories, having previous experience with the test method, conducted a preliminary trial of the procedure. The protocol for the test was then reviewed. Five coatings, along with necessary equipment, were distributed to these laboratories with an outline of the test procedure. At the time of the study, D 2369-89 was the most current form of the method. For multi-component coatings, the procedure allowed a one-hour induction time before sample insertion in the oven.

One operator in each of the seven labs was to run triplicate determinations on each coating system on separate days, using ASTM D 2369-89.

In addition to the usual determinations, one of the operators also ran triplicate determinations on each coating sys-
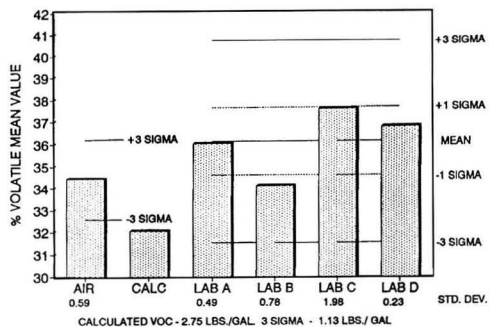
Table 2—Analysis of Variance of ASTM D 2369-89 Data

Sums of Squares

Source of Variation	Sum of Squares	D.O.F.	Mean Sq.	F-Ratio	Sig. Level
Main Effects					
A:Lab	52.3231	3	17.441	66.6824	0.0000
B:Day	8.4217	1	8.4217	32.1988	0.0000
C:Coating	7090.2286	4	1772.5572	6777.0397	0.0000
Interactions					
AB	6.3094	3	2.1031	8.0408	0.0001
AC	41.6924	12	3.4743	13.2833	0.0000
BC	0.8187	4	0.2047	0.7926	0.5399
ABC	11.2309	12	0.9359	3.5782	0.0003
Residual	20.9243	80	0.2616		
Total (Corrected)	7231.9491	119			

No missing values have been excluded.
All F-ratios are based on the residual mean square error.
Residual is a measure of the replicate variance.

Figure 1—Percent volatile. 1-Polyester urethane (two components)

CALCULATED VOC - 2.75 LBS./GAL. 3 SIGMA - 1.13 LBS./GAL

AIR 0.59 CALC LAB A 0.49 LAB B 0.78 LAB C 1.98 LAB D 0.23 STD. DEV.

Figure 4—Percent volatile. 4-Epoxy amidoamine (two components)

CALCULATED VOC - 1.32 LBS./GAL. 3 SIGMA - 0.06 LBS./GAL

AIR 0.54 CALC LAB A 0.66 LAB B 1.31 LAB C 1.30 LAB D 0.70 STD. DEV.

Figure 2—Percent volatile. 2-Polyester (two components)

CALCULATED VOC - 2.84 LBS./GAL. 3 SIGMA - 0.09 LBS./GAL.

AIR 0.22 CALC LAB A 1.07 LAB B 0.80 LAB C 0.55 LAB D 0.82

Figure 5—Percent volatile. 5-Alkyd enamel (one component)

CALCULATED VOC - 2.92 LBS./GAL. 3 SIGMA - 0.05 LBS./GAL.

AIR 0.30 CALC LAB A 0.23 LAB B 0.48 LAB C 0.48 LAB D 0.41

Figure 3—Percent volatile. 3-Baking enamel (one component)

CALCULATED VOC - 2.71 LBS./GAL 3 SIGMA - 0.04 LGS./GAL

AIR 0.19 CALC LAB A 0.29 LAB B 0.31 LAB C 0.66 LAB D 0.33 STD. DEV.

Figure 6—Standard deviation, coating

Figure 7—Standard deviation, lab

Coating Systems

(1) POLYESTER/ALIPHATIC URETHANE: An isocyanurate of isophorone diisocyanate (IPDI) crosslinked with a high solids polyester.

(2) UNSATURATED POLYESTER: Peroxide curable unsaturated polyester oligomer crosslinked with a polyunsaturated aliphatic oligomer.

(3) BAKING ENAMEL: Hexamethoxymelamine curing at 325°F.

(4) EPOXY/AMIDOAMINE: Epoxy crosslinked with amidoamine including benzyl alcohol as reactive diluent.

(5) VOC COMPLIANT ALKYD ENAMEL: Common medium oil soya alkyd.

RESULTS AND ANALYSIS

A summary of the test results that were considered for the analysis of variance is shown in *Table* 1. The analysis of variance was then performed on the ASTM D 2369-89 data. As noted in *Table* 2, the main effects and interaction terms, except the Day x Coating interaction, are significant at the greater than 99.9% confidence level.[6] As expected, the coating term is by far the strongest effector variable, followed by Lab, Day, and Lab x Day interaction. The experimental error term as expressed by the Residual, showed a relatively low variance of 0.26.

Figures 1-5 show the mean value obtained by the ASTM test method for each coating system, and the individual mean values for each laboratory. Along with these are plotted the values obtained for the air dried samples and the calculated values for each coating. Also, 1 and 3 sigma limits are indicated on these graphs.

Comparison of the one- and two-component coatings yields an obvious difference. The data for the one-component coatings (No. 3 and No. 5) produce a close range of results falling within the 1 sigma limit (68.3% confidence). The two-component coatings show a marked increase in the 1 sigma value. This range is from 2.5-3% volatile. In the epoxy coating, the 1 sigma range is much larger, representing about 25% of the mean value.

Table 3 shows a comparison of the volatile content determined by ASTM D 2369-89, calculated as lb/gal VOC, using the theoretical weight per gallon in the calculation from percent volatile. The 3 sigma (99.7% confidence) limit has been calculated as a percent of the mean for each coating system. Examination of these values shows clearly that individual results for two-component systems could vary over a wide range of values.

For the two-component coatings: Nos. 1, 2, and 4, the test results for VOC and percent volatile are 12.8%, 12.6%, and 5.0%, respectively, higher than the theoretical values. These results suggest several possibilities: (1) chemical reactions at 110°C producing volatile by-products, (2) volatile impurities in the coatings' raw materials, or (3) a tendency for raw material suppliers to make products on the low end of the reported solids content range. If the test yields higher VOC results because of the elevated temperature, the discrepancy between the measured and theoretical values could be reflected in a coating's cost, when test results are used to

tem, drying these at ambient temperature. These samples were prepared as in ASTM D 2369-89, to the oven insertion step. At this point the samples were placed in a protected area and dried to a constant weight at room temperature, 77°F.

Results of all tests were reported on data sheets supplied. All weights taken on samples were reported. Results were computed and the percent volatile by weight tabulated for the five coatings, consisting of two triplicate determinations. The experimenters did not include the weight per gallon determination, as this would introduce one more source of error, while yielding no additional information on the suitability of the oven loss procedure for the coatings tested.[5]

Initial data analysis indicated that one of the laboratories exhibited a much larger error than the others. The oven used by this tester was not a forced draft oven, as prescribed in the test method. For this reason, the data from this lab was removed from the set. Another lab reported 22 of the required 30 analyses. The results from this incomplete data set were deleted from the study. A third laboratory failed to complete the tests and presented no data. From the initial group of seven laboratories, only four supplied data that could be used in the study.

Table 3—Pounds/Gallon VOC

ASTM D 2369-89

	Coating	Theory	Mean	3 Sigma	As % of Mean
2 Component	1	2.75	3.10	0.39	12.58
	2	2.84	3.15	0.30	9.52
	4	1.32	1.39	0.48	34.53
1 Component	3	2.78	2.78	0.15	5.40
	5	2.86	2.87	0.16	5.57

Air Dried

	Coating	Theory	Mean	3 Sigma	As % of Mean
2 Component	1	2.75	2.96	0.15	5.07
	2	2.84	2.41	0.15	6.22
	4	1.32	0.89	0.18	20.22
1 Component	3	2.78	2.74	0.06	2.19
	5	2.86	2.73	0.09	3.30

formulate solvent content to achieve regulation compliance.

In two-component systems, the air drying yields significantly lower values for percent volatile and VOC than the ASTM D 2369-89 test procedure, which indicates that slow curing proceeds by a different mechanism, yielding few, if any, volatile by-products.

DISCUSSION

Analysis of the data shows a large variability. The variability is evenly distributed among the variables: coating, lab, and day. The major affecter variable is coating type. *Figure* 6 clearly shows that the variability for one-component systems (Nos. 3 and 5) is factor of 3 to 4 lower than that of two-component systems (Nos. 1, 2, and 4).

Each of the variables for the two-component systems: coating, lab, day, and replicate, exhibit a large standard deviation when compared to that of the one-component coatings (*Figures* 6-9). It is clear that some factor, not present in the one-component systems, is contributing to the variance of the two-component systems.

Some of the variability in the two-component systems may be caused by the separate weighing and mixing of the two components prior to sampling. The testers were instructed to weigh and mix the two components of each coating once each test day. The three test samples were taken from the same mixed sample. The large replicate variability for the two-component systems, as compared with the single-component systems, indicates that some additional factor has caused the overall large variability in the two-component systems (*Figure* 9).

Coating No. 4, the epoxy-amidoamine, shows a much lower percent volatile when air dried. This formulation contains the reactive diluent, benzyl alcohol. Some manufacturers of this product report benzyl alcohol to be 100% nonvolatile. In calculating the theoretical, the benzyl alcohol was considered volatile, since it will evaporate at the test method temperature. The disparity in the air and oven dried values could be caused by this product's limited volatility and entrapment within the matrix, when the film is dried slowly.

When data is converted to lb/gal VOC, and the three sigma value computed, a comparison of one- and two-component coatings indicates the large error resulting from use
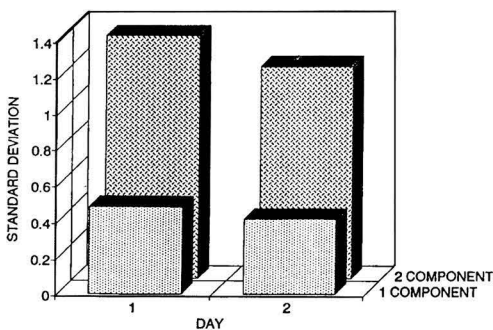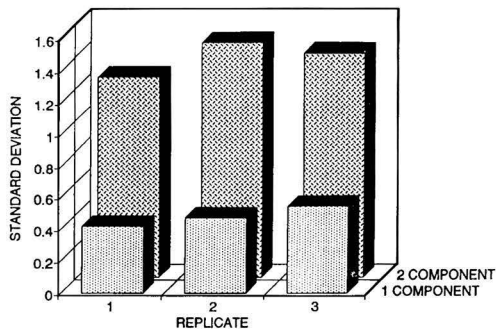

Figure 9—Standard deviation, replicate

of the ASTM procedure on two-component systems (*Table* 3). The two-component polyester and polyester urethane have a 3 sigma value, more than twice that of the one-component baking enamel and alkyd. The error is unacceptable when + and - 3 sigma is 0.6 and 0.8 lb/gal VOC. The two-component epoxy, when tested by ASTM D 2369, produces a + and - 3 sigma of 1.0 lb/gal VOC.

Evidently, this variability is a factor of the chemistries involved in the two-component systems. These coatings most likely contain or produce low boiling residues that are volatilized during the test. While in the oven at 220°F, these residues are volatilized—a process that may be dependent on amount of draft at different locations within the oven, degree of reaction of the components, or other unknown factors.

The participants in this study made every effort to minimize error. Laboratories that use this test for their products must be aware of the difficulties that may befall the tester. A single test result is insufficient. Data must be gathered in replicates over a period of time. Samples of multiple component systems will exhibit large error and must be carefully evaluated.

This study did not, by any means, approach every situation that will be faced by the analyst.

The systems tested represented a very small sample of the possibilities. Water based coatings, for example, introduce the problem of water content determination, as well as volatile determination. The analyst must be aware of the discrepancies between theoretical calculations and results obtained by ASTM D 2369-89. Also, the test may yield results with large variability when applied to two-component systems.

CONCLUSIONS

(1) Multiple component coatings tested with ASTM D 2369-89 have large variations in their test results.

(2) Laboratory and procedural variations play a significant part in the variability.

(3) ASTM D 2369-89 yields results higher than either the theoretical or air dried determination.

The multiple-component, high solids coatings produced the largest variance when ASTM D 2369-89 is used to

Figure 8—Standard deviation, day

determine VOC. This indicates that some unknown factors are important affecters of the test method. In order to make D 2369 work with nonstandard coatings, the individual laboratories and the coatings industry, as a whole, must improve their testing capabilities. However, this study, as a preliminary investigation, does validate using this method.

Reliance on calculated VOC values alone, or ignorance of the deficiencies in ASTM D 2369 when applied to multi-component coatings, can cause the manufacturer to report erroneous values for VOC. The coatings industry must be able to rely on results obtained by a VOC determination. Considerable work needs to be done to establish a procedure that, when applied to unusual coating systems, yields consistent and accurate results. This procedure must prove reproducible in the average laboratory.

Presently, Subcommittee D01.21 is considering changes in ASTM D 2369 to "broaden the scope of the method to include multicomponent paint systems." This aim is quite ambitious because this study clearly demonstrated that the VOC of two-component systems cannot be reproducibly determined by ASTM D 2369. This study does validate the method for conventional coatings.

An alternative procedure, in which volatiles are collected on tubes filled with activated charcoal, is also being evaluated.[5] We hope that as a result of these investigations, changes will be made in D 2369 that will make the method more reliable. Until then, all of us must be very cautious when making absolute conclusions concerning a product's VOC.

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New Oxazolidine-Based Moisture Scavenger for Polyurethane Coating Systems

Glenn N. Robinson, John F. Alderman, and Thomas L. Johnson
ANGUS Chemical Company*

A research program was initiated to determine whether an oxazolidine moisture scavenger could overcome the inherent disadvantages of physical and chemical moisture scavengers commonly employed in polyurethane coating systems. Oxazolidines were selected for this study because of their potential reactivity with water and generally good toxicity characteristics. Commercially available oxazolidines are unacceptable due to their high molecular weight and slow reactivity. A wide range of oxazolidine molecules were investigated. Oxazolidines from amino alcohol/ketone reactions demonstrated excellent hydrolysis characteristics. Ketone-based oxazolidines were then shown to effectively reduce moisture in single-package, moisture cured, pigmented polyurethane coatings. Good accelerated viscosity stability was also observed. Polyurethane coating systems can be produced using a ketone-based oxazolidine as a moisture scavenger. Such a product might find utility as a drying agent for wet polyols, solvents, pigments, or fillers used in one- or two-component systems where moisture is undesirable.

INTRODUCTION

Reduction of moisture is an essential step in producing polyurethane coating systems. Due to the reactivity of isocyanates with water in urethane systems, the presence of moisture leads to poor shelf life in single-component coatings and reduced pot life in two-part systems.[1,2]

Moisture is typically introduced into such systems in the form of dissolved water in solvent, adsorbed water in fillers, pigments, and polyol compounds, and water from the atmo-

sphere. If the moisture is not removed, it reacts with isocyanates to form carbon dioxide and amines [equation (1)]. The amines can react further with isocyanate to form ureas [equation (2)]. These by-products can adversely affect the entire system.

$$R - N = C = O + H_2O \rightarrow RNH_2 + CO_2 \tag{1}$$

$$R' - N = C = O + RNH_2 \rightarrow R' NHCO - NHR \tag{2}$$

Generation of CO_2 results in gassing in the can, and bubbles in cured films. Amines formed in the reaction will react further with isocyanates in the system, rapidly increasing polymer molecular weight as well as product viscosity. To prevent these reactions, it is essential that moisture scavengers react faster with moisture than does an isocyanate.

Current Moisture Scavenger Technology

A number of physical and chemical treatments are currently applied to urethane raw materials and formulated systems in order to reduce moisture. Vacuum extraction or high temperature drying are expensive, short-term solutions. For this reason, manufacturers frequently employ moisture scavengers capable of efficiently removing water from a system. Such treatments include monomeric isocyanates (e.g., paratoluenesulfonyl isocyanate)[3] and molecular sieves[4]—probably the most common method.

Reactive monomeric isocyanates are generally regarded as efficient alternatives to physical dehydration, chemically eliminating most of the moisture present. However, the potentially hazardous nature of these materials,[5,6] safe handling requirements, and cost limit their use as moisture scavengers in urethane coatings.

Molecular sieves are easily the largest group of moisture scavengers used in polyurethane systems. While molecular sieves, such as the zeolites, are widely accepted, the technology is not totally satisfactory. Since they do not react with moisture but rather adsorb it, the water remains in the system
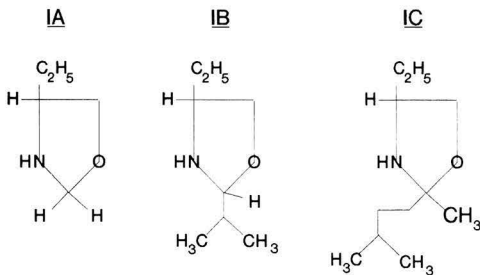
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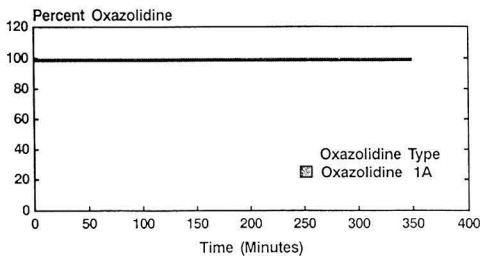
Figure 1—Oxazolidine structures

Figure 2—Oxazolidine reactivity (1:5 M:M oxazolidine:water). 0.2 M oxazolidine in acetonitrile
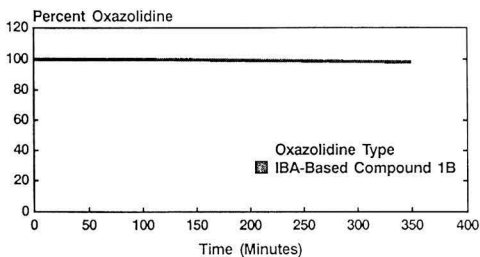

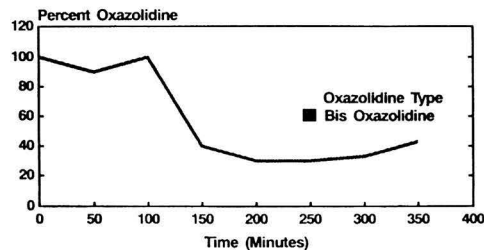Figure 3—Oxazolidine reactivity (1:5 M:M oxazolidine:water). 0.2 M oxazolidine in acetonitrile

Figure 4—Oxazolidine reactivity (1:5 M:M oxazolidine:water). 0.2 M oxazolidine in acetonitrile

and may later be released. It has been reported[7] that molecular sieves tend to lessen gloss and can contribute to foaming problems. Since molecular sieves are inert to the NCO/OH reaction, and remain in the finished product, they can also embrittle cured polyurethane finishes.

General Characteristics of Oxazolidines

The various drawbacks associated with current moisture scavengers for polyurethane systems stimulated the search for a possible replacement. It is known the oxazolidines are a class of compounds that can react with water. Oxazolidines are saturated heterocyclic compounds which can be prepared by reacting primary amino alcohols with aldehydes or ketones.[8-11]

In general, oxazolidine compounds are relatively easy to use, especially in comparison with monomeric isocyanates. This property was an added inducement to investigate the suitability of oxazolidines as moisture scavengers.

Certain bis alkyl oxazolidines are used in urethane systems as reactive diluents or latent hardeners. Their high molecular weight makes them unacceptable as moisture scavengers.[9,12,13]

EXPERIMENTAL

The 5-methyl-2-hexanone (MIAK) and isobutyraldehyde were obtained from Aldrich Chemical Company. Paraformaldehyde was obtained from Hoechst Celanese. The 2-amino-1-butanol was obtained from ANGUS Chemical Company.

The 4-ethyl oxazolidine was prepared by stoichiometrically mixing paraformaldehyde into 2-amino-1-butanol at ambient temperature under nitrogen atmosphere for 10 hr. The reaction mixture was then vacuum distilled at a pressure of 15 mm Hg and 40°C temperature to remove water. The material was stored under nitrogen atmosphere. The liquid product 4-ethyl oxazolidine was identified by gas chromatography-mass spectrometry.

The preparation of 4-ethyl-2-isopropyl oxazolidine was accomplished by stoichiometrically mixing isobutyraldehyde into a flask containing 2-amino-1-butanol under agitation and nitrogen atmosphere. The reaction mixture was then heated to reflux continuously for 10 hr. After the initial water was removed, the reaction mixture was distilled at a pressure of 40 mm Hg and a temperature of 70°C to remove any remaining water and co-reactants. The material was stored
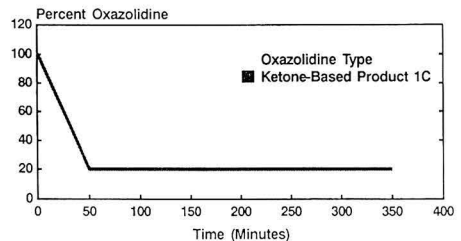

Figure 5—Oxazolidine reactivity (1:5 M:M oxazolidine:water). 0.2 M oxazolidine in acetonitrile

under nitrogen atmosphere. The yellow liquid was deter-
mined to be 4-ethyl-2-isopropyl oxazolidine by gas chroma-
tography-mass spectrometry.

The 4-ethyl-2-methyl-2-(3-methylbutyl)-1,3 oxazolidine
was prepared by stoichiometrically mixing 5-methyl-2-
hexanone (MIAK) into 2-amino-1-butanol at ambient tem-
perature. The reaction mixture was heated to reflux and
water was removed. The crude product was purified by
vacuum distillation at a pressure of 0.5 mm Hg and a tem-
perature of 45°C. The material was stored under a nitrogen
atmosphere. Gas chromatographic-mass spectrometric
analysis demonstrated the liquid was the product 4-ethyl-2-
methyl-2-(3-methylbutyl)-1,3 oxazolidine.

GC Moisture and Hydrolysis Method

Test samples were prepared at a concentration of 0.2M
oxazolidine in acetonitrile. Aliquots were injected at time
zero as well as the subsequent times in *Figures* 2 through 5.

The GC analyses were performed on an Hewlett-Packard
5890 thermal conductivity instrument using a 30 m by 0.25
mm (i.d.) by 1 μm (film thickness) DB-5 capillary column
with temperature programming. The detailed GC conditions
were:

Column head pressure .... 14 psi
Column flow .... helium at 1 mL/min
Auxiliary flow .... helium at 5 mL/min
Reference flow .... helium at 20 mL/min
Split ratio 35:1
Detector temperature .... 250°C
Injector temperature .... 220°C
Initial temperature .... 60°C with 2 min hold
Ramp 1 .... 10 to 150°C with 5 min hold
Ramp 2 .... 10 to 250°C with 7 min hold
TCD sensitivity .... S_2-helium
TCD sensitivity .... S_1-high

RESULTS AND DISCUSSION

Oxazolidine Structure/Activity

It is known that most commercial oxazolidine products,
produced by reacting an amino alcohol with formaldehyde,
could not serve as moisture scavengers due to their
unreactivity with moisture. In fact, they are so nonreactive
with moisture that they can be used in aqueous composi-
tions.[11]

Certain oxazolidines, however, are very reactive with wa-
ter. Obviously, changes to the molecule affect moisture reac-
tivity. In fact, the compound based on formaldehyde, identi-
fied as 1A in *Figure* 1, is extremely stable. The oxazolidine
identified as 1B is isobutyraldehyde-based with very slow
reactivity while the oxazolidine 1C is ketone-based and highly
reactive and shows promise as a moisture scavenger. After
investigating a wide range of molecules, it was noted that
ketone-based oxazolidines exhibit the desired combination
of characteristics—high reactivity with water and low mo-
lecular weight.

Hydrolysis Data

Selected developmental and commercial oxazolidines were
screened in hydrolysis studies through GC analysis (see

Table 1—Performance as a Moisture Scavenger

# Experiment	Initial $H_2O\%$	1 Hr $H_2O\%$	2 Hr $H_2O\%$
1	0.27	0.01	0.03
2	0.24	0.01	0.01

Experimental Section). This technique was employed as a
practical measurement for oxazolidine decomposition in the
presence of water.

This testing resulted in the hydrolysis data represented in
Figures 2-5, showing percent oxazolidine reactivity plotted
against time in minutes. The oxazolidines in *Figures* 2 and 3
demonstrate their stability in the presence of water, while the
urethane-bis-oxazolidine in *Figure* 4 (Hardener OZ, Miles
Inc.) shows reactivity with water after an extended period of
time. On the other hand, the ketone-based oxazolidine in
Figure 5 shows rapid reactivity in the presence of water.

The ketone-based oxazolidine with a molecular weight of
185 and a functionality of 1, is a low viscosity material that
reacts quickly with water, generating a ketone plus a primary
amino alcohol with an equivalent weight of 45 and a func-
tionality of 2. It has been used successfully to dry a variety of
urethane system components at ambient temperatures.

Application of the Concept

The demonstrated ability to react readily with water sug-
gests a number of possible uses for a ketone-based
oxazolidine. It could be used to dry basic urethane system
raw materials such as wet polyols, solvents, pigments, or any
other moisture-containing components, including isocyanates.
It could also be used as a package stabilizer in one-compo-
nent systems.

While ketone-based oxazolidines are theoretically suitable
for use as moisture scavengers in urethane systems, their
true efficacy had to be proved in a viable formulation. For this
purpose, a single-component, highly water sensitive, pig-
mented aliphatic urethane coating was formulated with the
ketone-based oxazolidine utilized as a moisture scavenger
(*Figure 6*).

After grinding the pigment portion of the formulation
from *Figure* 6 for 15 min, the moisture was determined by
the method described in the Experimental Section. The
moisture content of the grind paste is shown in *Table* 1.

Oxazolidine moisture scavenger was added, based on 18-
22 parts per part of water. Dibutyltin dilaurate catalyst was
utilized in the grind at the typical level of 0.1-0.2% on the
weight of the resin solids to promote cure. Agitation contin-

Table 2—Viscosity Stability

# Experiment	Initial Viscosity[a]	1 Week at 60°C Viscosity[a]	2 Weeks at 60°C Viscosity[a]
1	65KU	85KU NG	89KU NG
2	72KU	83KU NG	85KU UN

(a) Viscosities were done on a Stormer Viscometer (Krebs Units).
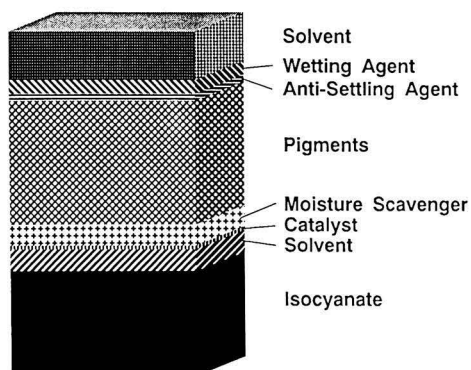NG = No gassing occurred.

Figure 6—One-package moisture cure polyurethane coating

Table 3—Performance as a Moisture Scavenger

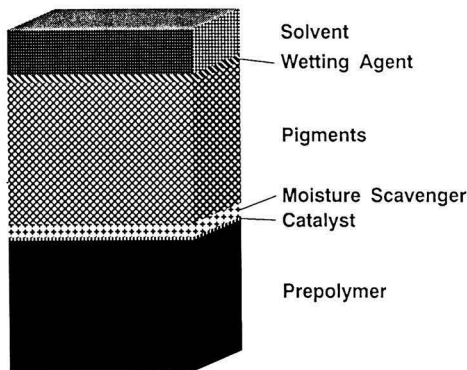|	Oxazolidine[a]	Monomeric Isocyanate[a]	Molecular Sieve[a]
Initial	0.32	0.20	0.23
Final[b]	0.05	0.03	0.05 GEL

(a) Percent moisture.
(b) Two hours.

Figure 7—One-package moisture cure pigmented polyurethane coating

Table 4—Moisture Scavenger Film Performance Rating[a]

	Oxazolidine	Monomeric Isocyanate
Pencil hardness	4	5
Adhesion	5	4
Impact resistance	5	3

(a) Rated: 1 = poor; 5 = best.

Table 5—Moisture Scavenger Film Performance Rating[a]

	Oxazolidine	Monomeric Isocyanate
Sheen	5	5
Sward hardness	4	5
IPA double rubs	5	5

(a) Rated: 1 = poor; 5 = best.

ued throughout a treatment period of 1 to 2 hr. Since duration of treatment depends on the efficiency of the moisture scavenger in the urethane system, periodic moisture analysis was performed by the GC method previously described (Experimental Section) to determine water content. Immediately after adding the final two ingredients, argon or nitrogen was used continuously to purge the system until the experiment was finished.

As soon as moisture reduction was less than 0.05%, the grind paste was allowed to cool to below 38°C, and the letdown portion was added to complete the formulation. Containers were purged with argon or nitrogen before the batch was sealed.

Viscosity Stability

The one-component moisture-cure, pigmented polyurethane coating for which moisture reduction results were reported in *Table* 1 also demonstrated good accelerated viscosity stability (*Table* 2). No gassing occurred in any of the samples treated with the ketone-based oxazolidine moisture scavenger.

Film Performance

In order to develop information on the effects of the ketone-based oxazolidine moisture scavenger technology versus monomeric isocyanate and molecular sieve technologies, a conventional one-package moisture cure urethane coating was formulated for film testing (*Figure* 7). The coating was prepared utilizing standard production procedures described in the previous performance study. Since the performance of the moisture scavenger depends on its efficiency, it was necessary to record moisture levels before and after treatment. As indicated in *Table* 3, the ketone-based oxazolidine and monomeric isocyanate reduced moisture of less than 0.05%. In the case of the molecular sieve, moisture levels were reduced, but since the moisture was not eliminated, the system gelled shortly after letdown with isocyanate. Due to this instability, the molecular sieve system was discontinued from the film studies.

The coating films prepared with the oxazolidine and monomeric isocyanate were evaluated for the mechanical properties of pencil hardness (ASTM D 3363), impact resistance (ASTM D 2794), and adhesion (ASTM D 3359). The film characteristics were evaluated for sheen and Sward hardness (ASTM D 2134). Chemical resistance performance was tested utilizing solvent rubs (ASTM D 4752). The performance properties (*Tables* 4 and 5) for the cured films were rated utilizing the ASTM Standardized Scoring System approach. The results indicate that the moisture scavengers have little

to no effect on film properties. The data also confirm the fact that in the case of the oxazolidine-based moisture scavenger, it is fully reacted and will not be available to affect film performance.

CONCLUSION

Ketone-based oxazolidines have been shown to perform as effective moisture scavengers. Modification of the oxazolidine structure produced a low molecular weight ketone-based oxazolidine with the rapid water reactivity necessary to serve as a moisture scavenger in urethane systems.

Experiments involving the use of a ketone-based oxazolidine in single-component polyurethane coatings proved the ability of this material to function as an effective moisture scavenger while not adversely affecting the stability or film performance of a coating.

ACKNOWLEDGMENTS

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Regulatory

UPDATE

This digest of current regulatory activity pertinent to the coatings industry is published to inform readers of actions which could affect them and their firms, and is designed to provide sufficient data to enable those interested to seek additional information. Material is supplied by National Paint and Coatings Association, Washington, D.C. and edited by members of the FSCT Environmental Affairs Committee.

Lead—Legislation amending the Toxic Substances Control Act (TSCA) that would ban certain uses of lead was introduced on April 1 by Sen. Harry Reid (D-NV).

The bill, S. 729, would also require the Environmental Protection Agency to create an inventory of all lead-containing products, mandate additional TSCA rule makings to control lead exposure, and require federally funded, state-run inspections of day care centers and elementary schools for lead contamination.

Sen. Reid maintains that EPA has not been responsible enough in its efforts to regulate lead under TSCA, and the bill will eliminate the need to rely on EPA. This is the third time Sen. Reid has introduced the legislation over the last three years. Parts of his 1992 bill were incorporated into last year's housing bill but most of the legislation has been stymied by unexpected lobbies. One of those lobbies is a group representing collectors of toys that contain lead.

S. 792 would ban or restrict the lead content of several products including industrial paint and coatings, pesticides, glass coatings, and inks. It also bans the intentional use of lead in packaging and reduces the incidental presence of lead in packaging. All products containing lead would have to be labelled as specified.

This bill differs from the 1992 proposal in that it has a built-in appropriation of $54 million to pay for the state inspection and reporting of lead hazards in day care centers and elementary schools. Sen. Reid has indicated he will use his position on the appropriations committee to obtain the necessary funding.

In a related matter, EPA's Office of Pollution Prevention and Toxics (OPPT) has completed a management review of non-residential lead-based paints prompting the agency to request a meeting with representatives of industry, unions, and government agencies regarding the use of lead paint on steel structures.

Reportedly, the analysis indicates that when bridges containing lead-based paint undergo renovation or repair, workers and the general public are exposed to high levels of air and soil lead contamination. EPA has called the meeting to discuss ways to reduce those exposures; the discussions will focus on performance, cost, and availability of lead-based paint substitutes on steel structures as well as paint removal technologies.

It is likely that this issue will receive a good deal of attention because of the provision in the Clinton Administration's economic stimulus package designed to increase investments in bridge renovation.

Environmental Statutes—Reauthorization—Sen. Max Baucus (D-MT) and Rep. Al Swift (D-WA) have indicated they will continue to push for recycling legislation, as part of a "RCRA Lite" bill, despite a full plate of other environmental issues facing this congress.

Sen. Baucus, Chairman of the Senate Environment and Public Works Committee, recently announced that he plans to introduce legislation later this year. Rep. Swift, who is Chairman of the House Energy and Commerce Subcommittee on Transportation and Hazardous Materials, is also drafting a bill.

Meanwhile, both legislators must begin the reauthorization process for the Clean Water Act, the Federal Superfund Law and the Endangered Species Act.

Responding to demands by environmental groups that Congress include stringent recycling requirements in an RCRA reauthorization package, Rep. Swift expressed concern that by trying to attempt action on Superfund and RCRA at the same time would render no return on either program. He also said he would prefer to begin the Superfund reauthorization as soon as possible as it is likely to take two years to complete.

EPA Cabinet Status—On April 2, the Senate Environment and Public Works Committee unanimously approved a provision to eliminate the White House Council on Environmental Quality (CEQ) as part of a bill to elevate the EPA to Cabinet level. Under the bill, S. 171, the EPA will acquire the CEQ's previous authority.

The approved provision would amend both the National Environmental Policy Act and the Clean Air Act. Oversight of environmental impact statements prepared by federal agencies and departments will be shipped from CEQ to EPA, and CEQ's report writing responsibilities will also go to EPA.

The full Senate will likely debate the bill after the spring recess.

Toxic Substances—Budget—Included in President Clinton's fiscal 1994 budget, released April 8, is $23.6 mil-

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lion that will be used for, among other programs, the Toxics Release Inventory (TRI) and the EPA's Office of Pollution Prevention and Toxics (OPPT) lead program.

According to budget documents, the plan provides an extra $15 million to fund OPPT's residential lead-based paint abatement program and an extra $5 million to expand the TRI program. Of the $15 million requested for lead paint programs, $10 million is targeted for state grant funding. According to EPA, the money would be used to support the development of programs to train lead paint abatement workers, accredit worker training programs, and certify abatement contractors. EPA said the remaining $5 million would be used to support the agency's lead strategy.

Of the $5 million budgeted to expand TRI activities, $4 million will be used to refund a fiscal 1993 appropriations cut and $1 million will be used to enhance the program. The enhancements include the addition of new reporting facilities and some new standard industrial codes. TRI is mandated in Section 313 of the Emergency Planning and Community Right-to-Know Act (EPCRA) which requires manufacturers file yearly reports on certain chemicals released into the environment.

Comprehensive Occupational Safety and Health Act

—Rep. William Ford (D-MI) recently re-introduced the Comprehensive Occupational Safety and Health Act. This year's bill (H.R. 1280) is multifarious and extends beyond the scope of the Occupational Safety and Health Act (OSH Act) itself. The following provisions are included in the legislation:

- Mandated safety and health committees for employers with 11 or more employees;
- Detailed written safety and health programs, including a plan for providing employee training and education;
- Expansion of National Institute for Occupational Safety and Health authority in OSHA enforcement; increased involvement in the settling of standards, priorities, and permissible exposure limits;
- Expanded employee participation rights from inspection to settlement in OSHA enforcement actions; employees would be provided with new whistle blower protections and refusal-to-work rights;
- Expanded existing OSHA criminal penalties;
- Provisions from earlier high-risk notification proposals;
- Increased OSHA enforcement authority and changes in OSHA's reporting/recordkeeping requirements; and
- Mandated that OSHA issue generic standards for exposure monitoring, medical surveillance, and ergonomics hazards.

Hearings in the House Education and Labor Committee, of which Rep. Ford is the Chairman, are expected after the spring recess.

Sen. Edward M. Kennedy (D-MA) has introduced similar legislation in the Senate (S. 575), but no hearings have been scheduled.

How to Obtain Congressional Documents

Bills, Reports, and Public Laws—House and Senate bills, reports, and public laws are free of charge, but there is a limit to the number that may be obtained at one time. Policies differ in the House and Senate.

Senate—Senate bills, committee reports, public laws, and conference reports are available in the Senate Document Room, B-04 Hart Building. The public may obtain as many as six different items per request, either in person or by mail. Only one copy of each item is permitted. One request per day will be filled. Mail orders may be sent to: Senate Document Room, B-04 Hart Building, Washington, D.C. 20510.

Information on status and availability of legislative documents is available from the Senate Document room at (202) 224-7860. This is an information number only. Phone orders are not accepted.

House—House bills, committee reports, public laws, and conference reports are available in the House Document Room, B-18, House Annex No. 2. The public may obtain as many as six different items per request in person. Up to 12 items may be requested by mail and six items by telephone. Only one copy of each item is permitted. One request per day will be filled. Mail orders may be sent to: House Document Room, B-18, House Annex No. 2, Washington, D.C. 20515. For information on status and availability of legislative documents or to order documents by phone, call the House Document Room at (202) 225-3456.

Hearings and Prints

In Person: Generally, all House and Senate hearings and prints can be purchased at the Government Printing Office, Congressional Sales Office, 710 North Capitol Street, N.W., Washington, D.C.

By Mail: Mail requests for hearings and prints should be sent to the Superintendent of Documents, Government Printing Office, Congressional Sales Office, Washington, D.C. 20402.

Payment may be made by cash, check, money order, credit card (VISA, MasterCard, Choice) and through GPO deposit accounts. Prices given for each document include postage and handling. No tax is charged. For questions and information, call (202) 512-2470 or 2471.

Lead Subcommittee Convenes—The Lead Subcommittee of the Policy Integration Project of the National Advisory Council for Environmental Policy and Technology has scheduled two meetings in May.

The subcommittee, which held its first meeting on March 31, will develop recommendations for improving and coordinating government efforts to reduce public exposure to lead by examining federal government policies for reducing lead exposure in the workplace, soils, paint, drinking water, and other means of contamination.

The meetings will be held May 5 and 19, 1993. For information on the meeting location, contact Donna Fletcher: EPA, Office of Cooperative Environmental Management, (202) 260-6883.

The following regulations are due to go into effect in May:

May 8, 1993

RCRA: Lead-bearing hazardous wastes (D008) placed in a staging area immediately prior to being introduced into a secondary smelter are prohibited from land disposal. (57 FR 28632; June 26, 1992.)

OSHA: Employers of 19 or fewer employees that are subject to the standards for occupational exposure to cadmium under 29 CFR 1910.1027 or 1926.63 must

establish properly signed, regulated areas where cadmium concentrations exceed or possibly can exceed the permissible exposure limit. (57 FR 42339; September 14, 1992.)

OSHA: Employers of 19 or fewer employees that are subject to the standards for occupational exposure to cadmium under 29 CFR 1910.1027 or 1926.63 must implement a respiratory protection program for employees exposed to prescribed levels of cadmium that conforms with the respirator selection, use and fit testing procedures. (57 FR 4234; September 14, 1993.)

OSHA: Employers of 19 or less employees that are subject to the standards for occupational exposure to cadmium under 29 CFR 1926.63 must comply with a permissible exposure limit of five micrograms per cubic meter of air, calculated as an eight-hour time-weighted average. (57 FR 42453; September 14, 1993.)

May 15, 1993

CAA*: Any product containing a Class I substance and any container holding a Class I or II substance introduced into interstate commerce must bear the label described in CAA Section 611(b).

CAA: Any product containing a Class II substance and introduced into interstate commerce must bear a label described under CAA Section 611(b) if the EPA administrator determines that safe substitute products or alternative manufacturing processes are available.

CAA: Any product manufactured with a Class II substance and introduced into interstate commerce must bear a label described under CAA Section 611(d) if the EPA administrator determines that safe substitute products or alternative manufacturing processes are available.

CAA: Any product manufactured with a Class I substance must bear a label described under CAA Section 611(d) unless the EPA administrator determines that safe substitute products or alternative manufacturing processes are not available.

*Clean Air Act

May 17, 1993

CWA*: Each group of dischargers of storm water associated with industrial activity that is required to obtain a permit and that is owned or operated by a municipality with a population of less than 250,000 must submit Part 2 of the group application of the EPA Office of Wastewater Enforcement and Compliance. (56 FR 12100; March 21, 1991, as amended by 57 FR 11412; April 2, 1992.)

CWA: Each operator of a medium municipal separate storm sewer system that is required to obtain a storm water discharge permit must file Part 2 of the application with the applicable permitting authority. (56 FR 12100; March 21, 1991, as amended by 57 FR 11412; April 2, 1992.)

*Clean Water Act

States Proposed Legislation and Regulations

California

Lead (Regulatory Activity)—The California Paint Council (CPC) and the National Paint and Coatings Association (NPCA), along with a small group of paint manufacturers, may join the petroleum industry in a legal action to challenge the Childhood Lead Poisoning Prevention Act of 1991. Alleging that the Act, which imposes an annual per-gallon "fee" on all "architectural coatings" manufactured in or imported into the state of California, was passed by the legislature in violation of state law, the paint industry is hoping to get the statute invalidated and the first year "fees," which were due on April 1, 1993, refunded to paint and petroleum industry "feepayers."

The suit, which may be filed by late April, will likely focus on the applicability of Proposition 13 to the statute. Proposition 13 requires that legislation imposing a tax be passed by a two-thirds vote of both houses of the legislature. Paint industry representatives contend that the "fees" imposed by the Childhood Lead Poisoning Prevention Act, which was passed by a simple majority, are actually taxes.

Earlier efforts by the California Paint Council to obtain an extension of time for paying the "fees" failed, although several paint companies took advantage of an extension that waives the penalty but not the interest for 30 days. Information on this extension, which will remain available until May 1, can be obtained by contacting CPC.

The paint industry plaintiff's are hopeful of getting an injunction either to prevent further collection of the "fees" or

to prevent the expenditure of "fees" already collected. Unfortunately, another state statute, which prohibits the issuance of an injunction to halt the collection of taxes, will likely be applicable in this case. Since the plaintiffs will argue that the "fees" are actually taxes for purposes of invalidating the statute, they may have to concede that this "anti-injunction statute" will prevent the issuance of an injunction in this action. An injunction against the expenditure of the "fees" should not be affected by the statute.

Meanwhile, a separate action challenging the regulations implementing the Act is also being discussed. The California Department of Health Services is holding a public hearing on April 14, 1993, to receive public input on its controversial regulations. The paint industry contends the regulations improperly targeted some paint companies that have never used lead in any of their products while ignoring several significant sources of environmental lead contamination. The CPC and the Western States Petroleum Association (WSPA), which represented the petroleum industry in earlier public workshops, are expected to oppose the regulations at the hearings.

Lead—CA S. 1063 (Calderon) authorizes a credit against the Personal Income Tax Law for each taxable year beginning on or after January 1, 1993, and before January 1, 1998, in an amount equal to 25% of the cost paid or incurred by the taxpayer during the taxable year in connection with the abatement of lead-based paint in any building containing one to four dwelling units, not to exceed $5,000 per building. The bill was referred to the Senate Committee on Revenue and Taxation on March 18.

Colorado

Recycled Paper—CO H. 1146 (Gordon) requires all documents submitted to State agencies to be on recycled paper, beginning January 1, 1994. The bill passed the House on February 24. It was amended and passed by the Senate on March 29 and referred back to the House for concurrence.

Connecticut

Air Quality—CT H. 6843 (Committee on Environment) concerns fees to implement the Clean Air Act Amendments of 1990. The bill was reported from the Joint Committee on Environment on March 16 and referred to the Joint Committee on Transportation.

Inhalant Abuse—ID S. 1012 (Committee on Judiciary and Rules) relates to possession of inhalants by minors; prohibits the possession of inhalants or aerosol spray products by minors for the purposes of utilizing the contents to become under the influence of such aerosol substance; defines "inhalant"; provides that minors illegally possessing inhalants shall not come under the purview of the Youth Rehabilitation Act unless the court orders otherwise. The bill was signed by the Governor on March 26.

Florida

Container Safety—FL H. 1029 (Dennis) revises provisions relating to notification of accidents relating to and restrictions on use of unsafe containers. The bill passed the House on March 25 and was referred to the Senate Committee on Commerce.

Idaho

Inhalant Abuse—ID S. 1012 (Committee on Judiciary and Rules) relates to possession of inhalants by minors; prohibits the possession of inhalants or aerosol spray products by minors for the purposes of utilizing the contents to become under the influence of such aerosol substance; defines "inhalant"; provides that minors illegally possessing inhalants shall not come under the purview of the Youth Rehabilitation Act unless the court orders otherwise. The bill was signed by the Governor on March 26.

Illinois

Lead—IL H. 928 (M. Davis) requires that all students in grades 3, 6, and 8 receive lead poisoning screening. The bill was reported favorably as amended from the House Committee on Health Care and Human Services on April 1.

Paint Disposal—IL H. 2436 (Ryder) amends EPA Act by prohibiting a person from knowingly mixing a restricted municipal waste intended for collection and delivery to a restricted handling facility with any other municipal waste. Restricts liquid paint at sanitary landfills after January 1, 1996. The bill was referred to the House Committee on Environment and Energy on March 16.

Indiana

Workers' Rights—IN H. 1048 (Hayes) provides that statutory or common law remedies are available to an employee alleging an injury from sexual or racial harassment in the workplace. The bill passed the House in February and was referred to the Senate Committee on Corrections, Criminal and Civil Procedure on March 15.

Iowa

Lead—IA S. 333 (Committee on Environment and Energy Utilities) establishes a lead inspection and abatement program and provides a penalty. The bill was introduced on March 17.

IA S. 383 (Committee on Human Resources) relates to programs and services under the purview of the Iowa Department of Public Health, including programs and services relating to lead abatement and environmental toxins; provides criminal penalties. The bill was introduced on March 19.

Maine

Tributyltin (TBT)—ME H. 7 (Heino) repeals an increase in the restriction on the release of tributyltin from antifouling marine paints. States that the current standard of release of 4.0 micrograms per square centimeter per day remains in effect. The bill was signed by the Governor on March 29.

Packaging—ME H. 756 (Marsh) revises the toxics reduction in packaging laws to require that manufacturers furnish certificates of compliance to purchasers instead of to the agency; establishes a six-year exemption for packaging that cannot meet the statutory concentration levels because of the addition of post-consumer materials; amends the Voluntary Waste Reduction and Recycling Labeling Program to include the terms "compostable" and "biodegradable"; specifies other changes. The bill was introduced to the Joint Committee on Energy and Natural Resources.

Maryland

Containers—MD H. 1055 (Pitkin) requires a manufacturer of rigid plastic containers sold, offered for sale, or used in association with the sale or offer for sale of a product in the state to ensure that the container meets designated requirements by July 1, 1994; specifies exemptions; requires each manufacturer to submit a specified certification to the Department of the Environment by October 1, 1994, and by March 1 of each year thereafter. On March 15, the bill was reported unfavorably from the House Committee on Environmental Matters.

Lead—MD H. 1325 (Thomas) increases the membership of the Lead Paint Poisoning Commission. The bill passed the House on March 11 and was referred to the Senate Committee on Judicial Proceedings.

Massachusetts

Lead—MA H. 2798 (Jehlen) authorizes the use of market share liability in civil actions brought against certain producers or manufacturers of lead-based paints. On March 22, the bill was withdrawn from the Joint Committee on Health Care and referred to the Joint Committee on Judiciary. A public hearing by that committee was held on April 12.

MA H. 1823 (Buell) is an omnibus bill that amends the lead paint statutes. The bill is in the Joint Committee on Health Care.

Hazardous Products—MA H. 2543 (Cahir) establishes a Retail Hazardous Products Identification Program which would require special labeling of paint products. The bill is in the Joint Committee on Commerce and Labor.

Minnesota

Lead—MN H. 961 (Clark) relates to lead abatement programs; specifies that residence assessments must be con-

ducted regardless of availability of state or federal funding, and must be conducted by lead inspectors licensed by the Commissioner of Health; requires contractor to give notice before beginning work at each lead abatement worksite; imposes surety bond requirements on lead abatement contractors; allows local boards of health to adopt more stringent standards; requires contractor analysis in older residence. On April 1, the bill was passed as amended from the House Committee on Health and Human Services and referred to the House Committee on Judiciary.

MN H. 1099 (Simoneau) relates to Health Department programs; extends advisory councils and committees for four years (to June 30, 1997); includes modifications to lead abatement program provisions. The bill was introduced on March 15 and referred to the House Committee on Health and Human Services.

MN H. 1569 (Clark) specifies allowable disposal of residential lead paint waste, as defined, to exclude waste removed with chemical strippers or waste mixed with water and containing free liquid; allows disposal in a variety of land disposal facilities meeting state requirements; makes persons whose activities produce such wastes responsible for them; requires non-residents who produce such waste to remove them and retain responsibility. The bill is in the House Committee on Environment and Natural Resources. An identical bill (S. 1551—Berglin) was introduced in the Senate on April 5 and was referred to the Senate Committee on Environment and Natural Resources.

Packaging—MN S. 1217 (Johnson) amends Minnesota Statutes; relates to solid waste management; defines packaging (including transport cushioning materials); requires Office of Waste Management to expand its public education campaign to cover packaging reduction and disposal issues and to set up a Packaging Advisory Council; authorizes annual Governor's awards for excellence in environmentally sound packaging; specifies council duties, including advising on packaging materials classification system. The bill was introduced on March 22 and referred to the Senate Committee on Environment and Natural Resources.

MN S. 1245 (Mondale) proposes new statute adopting packaging waste reduction requirements on companies as of January 1, 1996; requires a 15% reduction by weight from 1990 or a minimum packaging efficiency ratio of 90% product to 10% package by weight or volume; provides options for companies by using a company wide approach or a specific package approach; requires new products to meet specific package approach; specifies exclusions for inconsequential packages or insubstantial packaging parts. The bill was introduced on March 22 and referred to the House Committee on Environment and Natural Resources.

Missouri

Toxics-in-Packaging—MO S. 100 (Good) prohibits the selling of packages, packing material, or components containing specified concentrations of lead, cadmium, mercury, or hexavalent chromium. The bill passed the Senate on March 11 and was referred to the House Committee on Energy and Environment.

Nebraska

Containers—NE L.B. 63 (Elmer) creates the Plastic Container Coding Act to prohibit the manufacture or distribution of any plastic bottle or rigid plastic container unless it is imprinted with a code identifying the appropriate resin type used to produce the structure of the container. The bill was signed by the Governor on March 9.

New York

Lead—NY A. 3357 (Silver) enacts the Public Works Environmental Lead Abatement Act; provides for regulation of activities relating to the removal of lead-based paint from public works facilities. On March 23, the bill was amended and returned to the Assembly Committee on Health.

NY A. 5974 (Eve) makes various provisions regarding lead inspection and abatement including licensing and certification requirements; makes lead testing requirements; requires coverage for screening and diagnosis for lead by certain insurers and imposes a special surcharge on sale of paint. On March 23, the bill was amended and returned to the Assembly Committee on Health.

NY A. 6804 (Grannis) prohibits the exclusion of coverage for exposure to lead-based paint with regard to a policy insuring against the liability for injury. The bill was introduced on March 30 and referred to the Assembly Committee on Insurance.

NY A. 6805 (Eve) provides that policies insuring against the liability for injury shall not exclude coverage for exposure to lead-based paint. The bill was introduced on March 30 and referred to the Assembly Committee on Insurance.

Household Hazardous Waste—NY A. 4518 (Tokasz) regulates the sale at retail of household hazardous products and provides for management and disposal of such products; empowers the Environmental Facilities Corporation to finance and site household waste facilities statewide, on a regional basis. The bill was released from the Assembly Committee on Environmental Conservation on March 30 and referred to the Committee on Codes.

Ohio

Environmental Marketing—OH H. 202 (Campbell) requires persons who make environmental impact claims relative to a consumer good or its package or container to maintain and provide upon request documentation supporting the claims. The bill was referred to the House Committee on Energy and Environment on March 16.

Oregon

Containers—OR S. 1011 (Committee on Agriculture and Natural Resources) imposes a fee on manufacturers of glass, metal, paper and plastic containers; requires certification for manufacturing and wholesale of glass, metal, paper and plastic containers; requires manufacturers to keep certain records for examination by the Department of Revenue; directs the Department of Revenue to administer a fee program and deposit monies into the State Treasury to be credited to the Oregon Recycling Markets Development Fund; imposes civil penalties of up to 100% of the deficiency for failure to pay the fee. The bill was introduced on March 22 and sequentially referred to the Senate Committee on Agriculture and Natural Resources and the Committee on Revenue and School Finance.

Pennsylvania

Toxics-in-Packaging—PA H. 337 (Vitali) regulates toxic materials used in packaging and components thereof; prohibits packaging that includes inks, dyes, pigments, adhesives, stabilizers, or any other additives containing lead, cadmium, mercury, or hexavalent chromium which has been intentionally introduced as an element during manufacturing. The bill was amended and released from the House

Committee on Conservation on March 23 and referred to the Committee on Appropriations.

Spray Paint Restriction—PA H. 861 (Keller) amends the Crimes and Offenses Code by prohibiting the sale of aerosol spray-paint cans and broad-tipped markers to minors; regulates the display of such cans and markers; provides a penalty. The bill was referred to the House Committee on Judiciary on March 23.

Rhode Island

Lead—RI S. 767 (Izzo) prohibits the Department of Administration from purchasing paint products or contracting for the painting of public buildings using lead-based paints. The bill passed the Senate on March 31 and was sent to the House.

Texas

Lead—TX H. 1901 (Martin) relates to the registration of certain painting contractors. The bill was referred to the House Committee on Public Health on March 15.

TX S. 1278 (Truan) relates to the regulation of lead-related activities. The bill was referred to the Senate Committee on Health and Human Services on March 18.

Toxic Household Products—TX H. 2536 (Van de Putte) relates to safety requirements regarding the use of certain toxic household products; requires that toxic household products containing certain substances, manufactured on or after January 1, 1994, include in the product a bittering agent that is nontoxic, in a concentration that renders the product adversely bitter, unless the product is packaged with child-resistant safety closures; prohibits a person from distributing or selling such a product if the product does not meet these requirements. The bill was introduced on March 18 and referred to the House Committee on Higher Education.

Vermont

Solid Waste—VT H. 216 (Weidman) repeals provisions of law that provide financial assistance for the management of solid waste; converts the state's role in the management of solid waste to one in which the state cooperates with municipalities and the private sector in developing an integrated, market-driven, waste management program; removes the requirement that priority be given to reducing the waste stream through recycling and through the reduction of non-biodegradable and hazardous ingredients. The bill passed the House on March 26 and was referred to the Senate Committee on Natural Resources and Energy.

Lead—VT S. 30 (Ready) proposes to establish a program for training and certification of lead hazard abatement workers and to make blood lead screening and testing of children available upon request of parents; provides for inspection and testing of child care facilities; ensures that lead-based paint hazards are disclosed in the sale or lease of pre-1978 housing. The bill passed the Senate on March 24 and was referred to the House Committee on Health and Welfare.

Preparation and Properties of Blocked Dimethyl m-Isopropenyl Benzyl Isocyanate for One-Component Coatings

Howard R. Lucas and Kuang-Jong Wu
American Cyanamid Company*

A unique characteristic of dimethyl m-isopropenyl benzyl isocyanate is the flexibility it offers to the coatings industry. Copolymerization through its double bond with a variety of acrylic monomers offers the chemist broad possibilities in polymer design. By blocking the isocyanate moiety of the monomer, one can theoretically incorporate a hydroxy functional acrylic monomer within the same polymer.

Aimed at automotive OEM, industrial, and coil coating applications, the coatings are true one-component systems. However, preparation of these polymers is not without difficulties. A methyl ethyl ketoximine blocked monomer is an ideal candidate in a one-component coating system, with onset deblocking at 50°C. However, the unsaturated isocyanate as well as its blocked monomer requires a minimum polymerization of 80°C for complete conversion of monomer to polymer. With onset deblocking of the isocyanate moiety at 50°C, premature crosslinking takes place with the acrylic hydroxyls located in the growing polymer chains before polymerization is complete. To prevent this, we have discovered that certain solvents used in the polymerization prevent this premature gelation without affecting the coatings cure response and properties.

INTRODUCTION

With greater emphasis being placed on coating manufacturers by mandates from regulatory agencies and governments for high performance and environmentally safe coating systems, paint manufacturers have responded with systems such as low VOC acrylic-melamines, epoxies, and isocyanates. One such system which satisfies these demands is a blocked isocyanate polymer. Long known for their durability and reactivity, aliphatic isocyanate polymers are leading candidates for automotive OEM, industrial, and coil coating applications. By incorporating blocked aliphatic isocyanate polymers, resin manufacturers have dramatically improved the industrial hygiene properties and shelf stability of these resin systems. Because the blocked isocyanate resins are pre-blended with hydroxy containing polymers by the resin manufacturer for optimum properties, the end user has little concern for proper stoichiometry and needs only to add catalyst to the system. The chemistry of one-package urethane coatings has been well reviewed by Wicks.[1,2]

Recently, a series of blocked isocyanates based on dimethyl m-isopropenyl benzyl isocyanate* (shown in *Figure* 1) was examined. Based on initial studies, these copolymers could offer a solution to the newest issue facing producers of automotive coatings—that of environmental etch.

A one-component coating system has been prepared from these blocked monomers by copolymerization with hydroxyl functional acrylic monomers. These coatings typically cure in 30 min at 110-145°C.
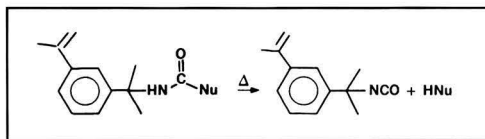
POLYMER DESIGN AND BLOCKING AGENTS

Since dimethyl meta-isopropenyl benzyl blocked isocyanate can be polymerized with hydroxyl acrylics through its double bond, it allows a greater latitude of

*TMI® (META) unsaturated aliphatic isocyanate.

Figure 1—Blocked dimethyl m-isopropenyl benzyl isocyanate and deblocking, generating free NCO. HNu = nucleophilic agent (isocyanate blocking agent)

Table 2—Typical Polymerization Charge	
Charge A	**Wt in Grams**
Toluene	6.7
Charge B	
Toluene	60.0
Blocked isocyanate[a]	77.4
Butylacrylate (BA)	53.4
Methyl methacrylate (MMA)	40.2
Hydroxy ethyl acrylate (HEA)	31.0
t-Butyl peroctoate	8.4
n-Dodecanethiol	4.2

(a) MEKO blocked TMI (meta) unsaturated aliphatic isocyanate.

polymer design. With the hydroxyl as an integral part of the polymer backbone, there is little concern for compatibility. This is in contrast to the traditional one-component resin systems, which require the blending of compatible resins capable of crosslinking with each other, and still maintain this compatibility during the transition from the thermoplastic to the crosslinked state.[3] To emphasize the compatibility of this type of system (*Figure* 2A) vs the traditional one-component system (*Figure* 2B), it can be seen that a potential problem exists during crosslinking. If not designed properly, "loose ends" of both the isocyanate and the hydroxyl are a potential source of incompatibility and degradation to the coating.

A series of blocked isocyanate monomers were prepared at our laboratories for internal evaluation. The deblocking rates (*Table* 1) were determined by following FTIR traces from 29-202°C at a 1°C/min temperature ramp, to the point of initial appearance of NCO followed by rapid generation of NCO.[4] From the data, a methyl ethyl ketone oxime (MEKO) blocked isocyanate monomer was selected for continuing studies due to its low deblocking temperature. Preparation of the copolymers proved difficult, as deblocking occurs close to the temperature required for complete conversion to polymer. Polymerizations of the unblocked isocyanate monomer run at temperatures of 42 and 62°C as shown in *Figure* 3

Table 1—FTIR Deblocking Temperatures			
		Deblocking Temp. (°C, Via FTIR[a])	
Isocyanate	**Blocking Group**	**NCO Onset**	**Rapid NCO**
Dimethyl m-isopropenyl			
benzyl isocyanate	Methanol	155	190
	n-Butanol	165[b]	190[b]
	n-Pentanol	165	190
	n-Hexanol	155	175
	E-caprolactam		
	(mp = 59-63°C)	90[b]	125[b]
	Methyl ethyl		
	ketoxime	50[b]	65[b]
	Acetone oxime	50[b]	80[b]
	Cyclohexyl oxime	65[b]	100[b]
	BD mono oxime	105[b]	120[b]
	n-Hydroxy		
	succinimide	115[b]	140[b]
	Propylene glycol	>180[b]	—

(a) Temp ramp from 29-202°C @ 5°C/min. IR re-measured every 5°C.
(b) Temp ramp from 29-202°C @ 1°C/min. IR re-measured every 5°C.
Note: NCO Onset is clearly defined while the Rapid Onset is mostly judgmental.

contain bimodal species and only conversions of 20-60%, respectively.[5]

RESULTS AND DISCUSSION

Solution polymers containing methyl ethyl ketoxime blocked unsaturated isocyanate monomer were prepared with a variety of commercial acrylic monomers, including acrylic monomers with a pendant primary hydroxyl group. The typical charge for the free radical polymerization and reactor schematic is shown in *Table* 2 and *Figure* 4, respectively. The polymerization is accomplished by feeding a nitrogen sparged monomer solution containing charge B to a preheated solvent, toluene (charge A), at 80°C over a four-hour period under nitrogen, followed by an additional four hours of heating at 80°C. The procedure, developed for the polymerization of unblocked unsaturated isocyanate, does not take into account acrylic monomer-containing hydroxyl groups that can react with the NCO moiety of the isocyanate monomer. By using the charges outlined in *Table* 2 and following the previously described procedure, the polymer solution gelled shortly after the four-hour monomer addition time. Other solvents typically used in preparing solution polymers for coatings such as methyl amyl ketone, xylene, and butyl acetate resulted in the same premature gelation.

Kinetic studies on blocked isocyanates have shown strong evidence for the elimination-addition reaction.[2] This supports the reason for the premature gelation encountered, i.e., once the isocyanate is liberated from the blocking agent (K_1) the isocyanate is free to react with the hydroxyl monomer in the growing polymer or react with the freed blocking agent (*Figure* 5). According to Carter and Pappas,[6] the back reaction (K_{-1}), the reaction of isocyanates to the ketoximine, is approximately 30 times greater than the reaction to a primary hydroxyl (K_2). However, only a few crosslinks are necessary in a polymer to make it unprocessible.

In the past we have experienced that some solvents, particularly those with large dipole moments, retard the reaction of dimethyl m-isopropenyl benzyl isocyanate polymers with difunctional primary hydroxyl compounds,[5] as shown in *Figure* 6. This is contrary to the literature[2,7] with this isocyanate. One possible explanation for this anomaly is addressed by Jensen.[8] In his overview of Lewis acid-base classification of solvents
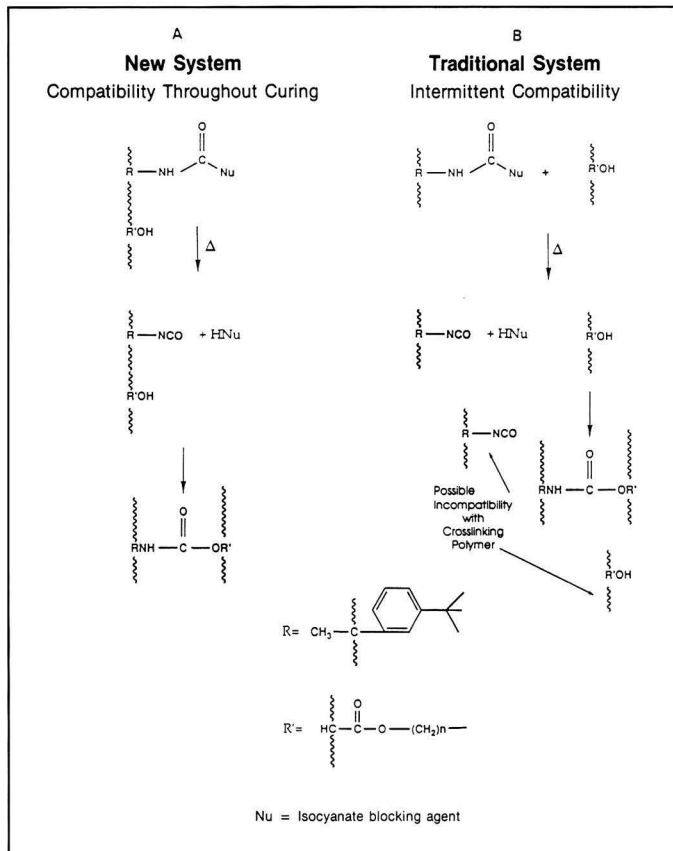
Figure 2—A: New system with compatibility throughout curing. B: Traditional system with intermittent compatibility. Nu = isocyanate blocking agent

and their ability to act as electron donors (DN) or acceptors (AN), solvent electrons form potential solvated complexes of both the oxime blocked and unblocked isocyanate monomer. This retarding action of the solvent in the deblocking mechanism results in the reduction of the reaction rates of the free isocyanate with the hydroxyl moiety (*Figure* 7) and is accomplished by supplying or accepting electrons from the solvent. An example of solvent complexing resulting in infrared shifts with both our isocyanate moiety and the MEKO blocked isocyanate monomers in a variety of polar and nonpolar solvents is shown in *Table* 3.

Since the kinetics of deblocking and blocking in polar solvents are critical in the preparation of MEKO blocked unsaturated isocyanate-acrylic polymers, a model study of the blocked monomer in solvent was run to determine the kinetic values on K_1 and K_{-1} in solvents with varying degrees of polarity (hexane $\mu = 0.0$,* butyl acetate $\mu = 1.9$, and acetonitrile $\mu = 3.9$). The experiments
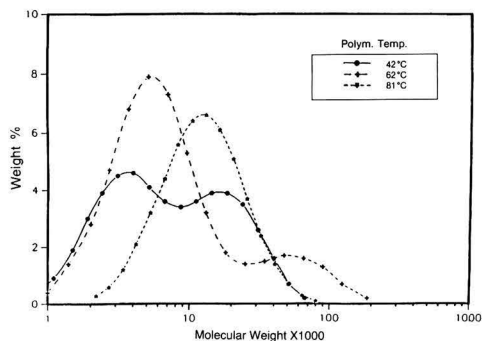

Figure 3—Molecular weight of dimethyl m-isopropenyl benzyl isocyanate copolymer. MMA 45/BA 40/unsaturated isocyanate, 15 mole % in toluene at various temperatures

*Toluene $\mu = 0.4$ could not be used in these experiments, since it elutes at the same time as the unblocked isocyanate monomer.

Chemicals Used

| Generic or Chemical Nomenclature	Tradename	Manufacturer
Dimethyltindilaurate	Fomrez® UL-28	Witco Chemical
Benzotriazole UV stabilizer	Cyagard® UV-2337	American Cyanamid Company
HALS radical scavenger	Sanduvor® 3058	Sandoz Chemical Company
Methylated melamine	Cymel® 330	American Cyanamid Company
Acrylic resin with hydroxyl number of 125	Doresco®	Dock Resin Corporation
Dimethyl m-isopropenyl benzyl isocyanate.........	TMI® (META) unsaturated aliphatic isocyanate	American Cyanamid Company
Diisocyanate adduct...........................	Cythane® 3160 aliphatic polyisocyanate resin	American Cyanamid Company
Acrylic resin................................	Joncryl® 500 resin	S.C. Johnson Company
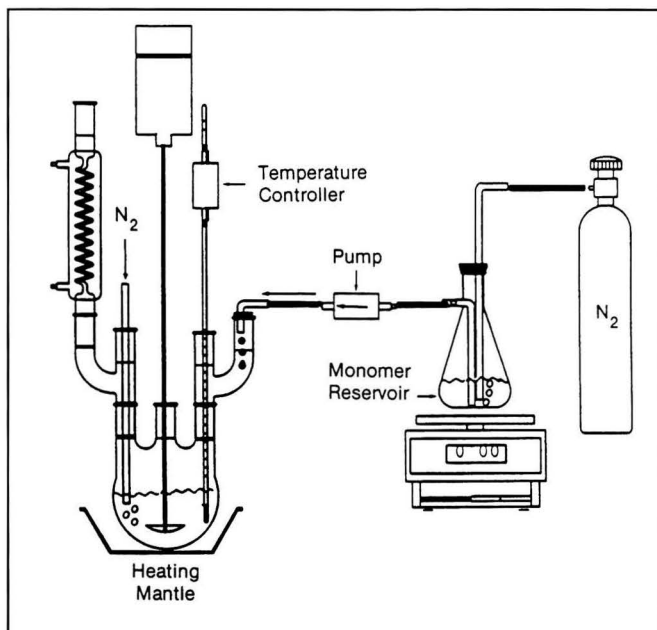
Figure 4—Polymerization schematic

Table 3—IR Shifts Due to Complexing of Solvents with Both Blocked Dimethyl m-Isopropenyl Benzyl Isocyanate and the Unblocked Groups

Solvent	μ	AN	DN	Peak[a](C=O)	Shift	(N=C=O)	Shift
None	—	—	—	1754	0	2258	0
Heptane......................	0	0	0	1754	0	2258	0
Acetonitrile...................	3.9	18.9	14.1	1750	−4	2262	+4
MAK.........................	2.7	12.5	17.0	1749	−5	2256	−2
Nitromethane	3.4	20.5	2.7	1740	−14	2263	+4
Butyl acetate..................	1.9	—	16.5	1750	−4	2246	−12
Toluene......................	0.4	8.2	0.1	1750	−4	2243	−15
DMSO........................	4.0	19.3	29.8	1748	−6	2240	−18
Benzonitrile	4.0	15.5	4.0	1750	−4	2243	−15
Methylene chloride	1.5	23.1	0	1744	−10	2243	−15

(a) Peak denotes the urethane C=O of MEKO blocked adducts of dimethyl m-isopropenyl benzyl isocyanate adduct.

were conducted by preparing solutions of our MEKO blocked isocyanate monomer in each of the aforementioned solvents. The ratio of solvent to blocked monomer was such that they simulated the ratios used in the solvent polymerization. Each solution was placed in six separate vials and sealed with a teflon septum and placed in an 80°C ± 0.5°C water bath. Samples of approximately 60 mg were extracted (one extraction per vial to prevent loss of blocking agent) at various intervals of time, diluted with methylene chloride in a 50 ml volumetric flask, followed by injection into the HPLC. The results shown in *Figure* 8 indicate that the deblocking (K_1) and the blocking (K_{-1}) reach equilibrium in all of the solvents in one-half hour. However, in the higher dipole monomer solvent, acetonitrile, the blocked reaction is favored by approximately 2/1 (K_{-1}/K_1) as compared to the low dipole solvent, hexane (*Table* 4).

The effect of solvent polarity on the retardation of premature gelation during polymerization was exemplified in another HPLC model experiment. Using the same technique described previously, equal molar ratios of our MEKO blocked monomer and n-butanol, to simulate the acrylic hydroxyl groups in the polymerization, were prepared.

They were dissolved in the same solvents used in the deblocking kinetic experiments with one exception, toluene was substituted for hexane, since we were only interested in the amount of urethane product (TMB) that was formed.
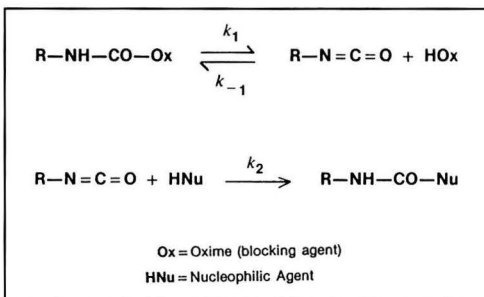
The results show (*Figure* 9) that, in eight hours at 80°C, 3% of the urethane product (TMB) was formed in toluene, and only 1.5% in acetonitrile after the same period. These model experiments correlate well with premature gelation in the selected solvent shown in *Table* 5. We believe that the effectiveness of acetonitrile in both the retardation of deblocking and urethane formation may not only be a result of its electrostatic properties, but its geometry as well. Being a small planar molecule, acetonitrile can more closely approach the blocked isocyanate monomer and the unblocked isocyanate moieties, as depicted in *Figure* 10, allowing for a more effective complexation, thus impeding the attack of the nucleophilic agent, which in this case is the hydroxyacrylic.[8] From these experiments it became clear that the choice of solvent was critical in the prevention of premature gelation during the preparation of our one-component polymer and why, in our earlier observations, polar solvent had retarded the NCO-hydroxyl reaction. By substituting acetonitrile for toluene in the charges in *Table* 2, copolymerization of the MEKO blocked isocyanate monomer and the hydroxyl acrylic was prepared without premature gelation.

POLYMER PREPARATION FOR COATING EVALUATION

A series of acrylic copolymers at various molar ratios at 75% concentration in acetonitrile was polymerized for eight hours, using the earlier described polymerization technique. In addition to MEKO blocked monomer, the unsaturated isocyanate, blocked with four other blocking agents, e-caprolactam, n-hydroxysuccinimide, methanol,

Table 4—Deblocking Kinetics K_1 and K_{-1} and the Effect of Solvents Based on their Polarity

Solvent	μ	K_{-1}/K_1
Hexane	0	40.0
Butyl acetate	1.9	50.0
Acetonitrile	3.9	70.0

$$R-NH-CO-Ox \underset{k_{-1}}{\overset{k_1}{\rightleftharpoons}} R-N=C=O + HOx$$

$$R-N=C=O + HNu \overset{k_2}{\longrightarrow} R-NH-CO-Nu$$

Ox = Oxime (blocking agent)
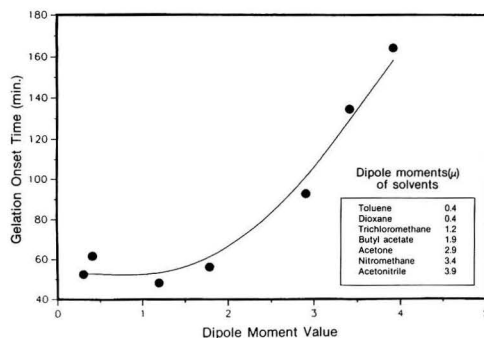
HNu = Nucleophilic Agent

Figure 5—Reaction kinetics of blocked isocyanate. Ox = oxime (blocking agent) and HNu = nucleophilic agent

Table 5—Polymerization of MEKO Blocked Dimethyl m-Isopropenyl Benzyl Isocyanate with Acrylic Monomers

MEKO blocked monomer 15/BA 40/MMA 30/HEA 15 polymerized at 75% solids at 80°C, four-hour addition, followed by an additional four hours heating

Solvent	Time to Gelation	μ	AN	DN
Toluene	~5 hr	0.5	8.2	0.1
Butyl acetate	6-7 hr	1.9	—	16.5
Acetonitrile	>8 hr	3.9	18.9	14.1

Dipole moments(μ) of solvents	
Toluene	0.4
Dioxane	0.4
Trichloromethane	1.2
Butyl acetate	1.9
Acetone	2.9
Nitromethane	3.4
Acetonitrile	3.9

Figure 6—Effect of solvent dipole on gelation of dimethyl m-isopropenyl benzyl isocyanate copolymer with 1,6-hexanediol at 40% solids at 40°C using 0.5% T-12 catalyst

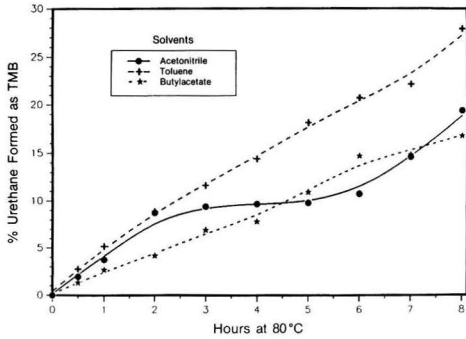Figure 7—Reaction rates of dimethyl m-isopropenyl benzyl isocyanate monomer and butanol. NCO/OH ratio 1/1 in various solvents

Table 7—MEKO Blocked TMI® Unsaturated Aliphatic Isocyanate Self Crosslinking Copolymers

Characteristics of Blocked TMI Monomer Copolymers

	Polymers					
	A	B	C	D	E	F
MEKO blocked TMI monomer, mole %	16	20	25	20	20	15
HEA, mole%	19	20	25	20	20	0
BA, mole%	43	50	50	20	30	35
HEMA, mole%	0	0	0	0	0	15
MMA, mole%	22	10	0	40	30	35
Theo. T_g, °C	12	10	0	40	30	35
DMA T_g, °C	n/a	51	n/a	85	80	n/a
NV (foil)................	75.7	76.3	75.8	72.2	74.3	70.7

Table 6—Blocked TMI® (Meta) Unsaturated Aliphatic Isocyanate Self Crosslinking Copolymers

Cure Temperature of Blocked TMI Monomer Copolymers

Blocking Agents	Temperature, °C
Methyl ethyl ketone oxime	≤120
E-caprolactam....................................	130
N-hydroxysuccinimide.............................	130
Methanol	>145
n-Butanol.......................................	>145

Figure 9—Crosslinking kinetics during polymerization of MEKO blocked dimethyl m-isopropenyl benzyl isocyanate monomer. NCO/OH 1/1 in various solvents
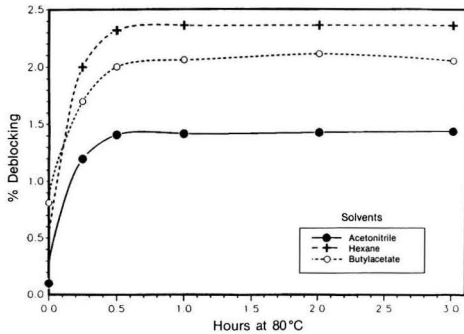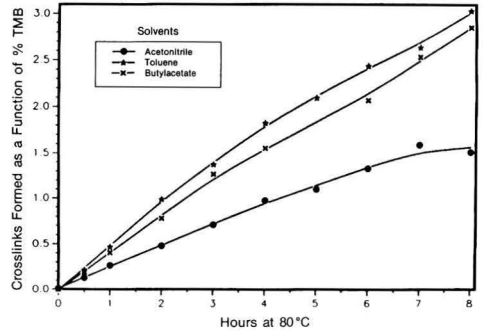

Figure 8—Deblocking of MEKO blocked dimethyl m-isopropenyl benzyl isocyanate monomer at 80°C in various solvents

Table 8—MEKO Blocked Dimethyl m-Isopropenyl Benzyl Isocyanate Self Crosslinking Copolymers

Effect of MEKO Blocked Isocyanate Monomer Level on Coating Performance

	Polymers			Control Acrylic-melamine[a]
	A	B	C	
Blocked isocyanate monomer, mole %	16	20	25	n/a
Thickness, mils	1.5	1.5	1.5	1.4
Hardness, KHN$_{25}$............	7.0	7.1	8.6	6.3
MEK rubs	200+	200+	200+	200+
Urethane sites[b].............	1.1	1.3	1.5	n/a
Stamford etch resistance[c] (0 = the best; 10 = the worst)				
2 weeks	1	1	1	9
10 weeks..................	2	2	3	10

(a) Joncryl® 500 Resin/Cymel® 1168 Resin/DDBSA = 75/25/0.7.
(b) The unit of the potential urethane sites: mEq/g.
(c) Etch resistance is subjective rating scale developed by General Motors based on observation by the viewer on the size and density of spots.
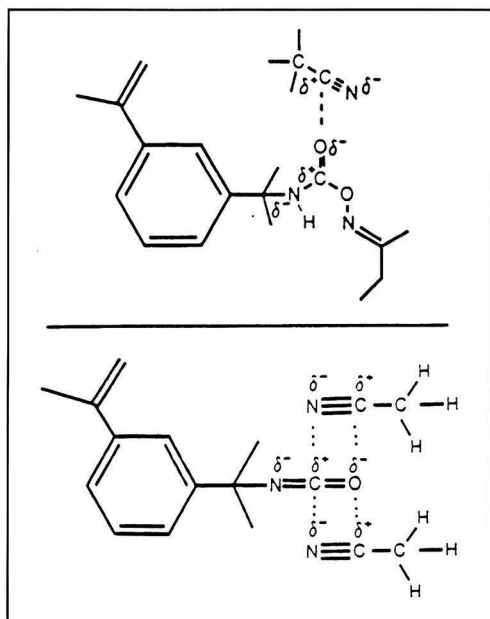
Figure 10—Proposed dipole-dipole interaction of acetonitrile with MEKO blocked dimethyl m-isopropenyl benzyl isocyanate and the free NCO

Table 9—MEKO Blocked Dimethyl m-Isopropenyl Benzyl Isocyanate Self Crosslinking Copolymers

Effect of T_g on Coating Performance

	Polymers			Control[a]
	D	E	B	TMP-TMXDI[b]
Thickness, mils	1.8	1.7	1.6	1.7
Hardness, KHN$_{25}$............	11.9	8.4	5.5	7.9
MEK rubs 200 + ...	200 +	200 +	200 +	200 +
Urethane sites[c]...............	1.4	1.3	1.3	2.5
Theo. T_g, °C of polymer......	47	32	7	n/a
T_g, °C of crosslinked coating (DMA)............	85	80	51	56

Stamford etch resistance (0 = the best; 10 = the worst)

1 week.....................	0	0	1	5
3 weeks...................	0	0	2	5
5 weeks	0	0	2	7

(a) Doresco® TA39-14 Resin/MEKO blocked trimethylol propane-TMXDI[2] adduct = 56/3/43.7.
(b) TMXDI = TMXDI® (META) aliphatic diisocyanate.
(c) The unit of the potential urethane sites: mEq/g.

and butanol were evaluated to determine the minimum cure temperature of the coating copolymers.

All of the isocyanate blocked monomers were incorporated at 15 mole %. The remaining monomers were butyl acrylate (BA) at 35 mole %, methyl methacrylate (MMA) at 35 mole %, and hydroxy ethyl acrylate (HEA) at 15 mole % for this thermal screening process. All of the blocked coatings were formulated with 0.5% dimethyl tin dilaurate catalyst and baked for 30 min. The minimum cure temperature of the selected blocked copolymer is the temperature where sufficient crosslinking takes place in a clear coating to the point where it can withstand more than 200 double MEK rubs without noticeable attack. Since the MEKO blocked isocyanate copolymer (*Table 6*) had the lowest cure temperature, it was selected for further study.

Studies focused on some of the factors which could affect the etch resistance of a cured coating, including the isocyanate levels and glass transition temperature (T_g) of cured coatings. The molar ratios and T_gs of MEKO blocked isocyanate copolymers are shown in *Table 7*.

MEKO BLOCKED DIMETHYL m-ISOPROPENYL BENZYL ISOCYANATE MONOMER LEVEL AND CROSSLINKING DENSITY

Experiments were carried out to define the effects of the urethane crosslinking density of the coating on etch resistance. The level of MEKO blocked isocyanate was varied from 15 to 25 mole % for this study. All of the

coatings were formulated with 2% benzotriazole UV stabilizer, 1% Hals radical scavenger, and 0.5% dimethyl tin dilaurate catalyst. The cure schedule used was 125°C for 30 min. Comparisons were made to an existing acrylic melamine automotive system as the control. The results of the experiment are shown in *Table 8*.

The etch resistance rating of the baked coatings was excellent and much better than the acrylic melamine control. The ratings ranged from 2 to 3 vs the melamine system's 10 after 10 weeks. The coatings with higher levels of isocyanate were expected to have better etch resistance due to their higher crosslinking density. However, there were no significant differences noticed among the three tested isocyanate coatings. It has been hypothesized that the hydrolysis of urethane groups in the coating by the acid rain may play an important factor, but this does not seem to be the case.

GLASS TRANSITION TEMPERATURE

It was also hypothesized that the higher the T_g of a coating, the better the etch resistance if the rest of the parameters are the same. A series of the self crosslinking MEKO blocked isocyanate copolymers with various T_gs

Table 10—MEKO Blocked Dimethyl m-Isopropenyl Benzyl Isocyanate Self Crosslinking Copolymers

Formulation for Xenon Accelerated Weathering

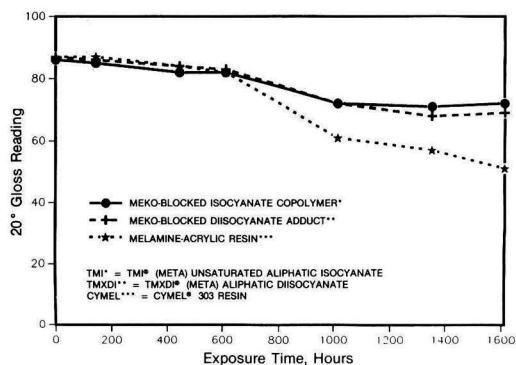|	Polymer		
	A	B	C
Copolymer F..................	100.0	0.0	0.0
TA39-14	0.0	53.4	65.0
MEKO blocked diisocyanate adduct	0.0	46.6	0.0
Methylated melamine resin.......	0.0	0.0	35.0
Tin catalyst...................	0.5	0.5	0.0
p-TSA	0.0	0.0	0.3
UV-2337 light stabilizer	2.0	2.0	2.0
3058 HALS	1.0	1.0	1.0

Figure 11—Xenon accelerated weathering

were prepared and compared to a multifunctional MEKO blocked diisocyanate adduct. The results of this experiment are shown in *Table 9*. As expected, better etch resistance was observed for coatings with higher T_gs. The T_g of coatings D and E is approximately 80°C, whereas the T_g of coating B and the control polymer is in the 50s. It is also illustrated in *Table 9* that the control, with a higher T_g than that of coating B, has considerably worse etch resistance. This suggests some hydrolysis of the many urethane groups in the control coating. In addition, the control is considered a traditional one-component system and, as pointed out earlier in the Polymer Design Section and in *Figure* 2B, it would be surprising that unreacted NCO and hydroxyl do not exist, which should also contribute to the poor etch resistance.

ACCELERATED WEATHERING (XENON EXPOSURE)

In addition to etch resistance, the MEKO blocked unsaturated monomer copolymers (*Figure* 11) have demonstrated excellent maintenance of gloss in Xenon exposure, as compared to typical coatings presently used in the automotive industry.

The MEKO blocked coating F was compared to a MEKO blocked TMP adduct and Cymel® 303 resin in the Xenon accelerated weathering test. The melamine-acrylic coating used was a Doresco® TA-39 resin (Polymer C of *Table* 10) which is composed of only acrylic monomers. The formulation is shown in *Table* 10. There is no significant difference noticed on the 60° gloss data. The 20° gloss retention data is shown in *Figure* 10.

SUMMARY

Dimethyl m-isopropenyl benzyl isocyanate monomer offers a unique opportunity in coatings. Through its double bond, it can be copolymerized with acrylic monomers, long known for their stability under the hard environmental conditions of weathering. By blocking the isocyanate moiety of the monomer and incorporating it into acrylic polymers containing hydroxyl groups, a one-component coating system has been successfully produced. The monomer blocked with MEKO can be polymerized at temperatures above its deblocking temperature in the presence of monomers containing hydroxyl functionality. Although contrary to the literature which suggests that polar solvents aid in the deblocking of isocyanates, the opposite has been experienced in certain high dipole moment solvents. Although not commonly used in polymerization, acetonitrile both retards the deblocking mechanism and inhibits the urethane formation, thus preventing premature crosslinking during the copolymerization stage. Curing at low temperatures, the coating produces excellent properties in the areas of solvent resistance, hardness, etch resistance, and weatherability.

ACKNOWLEDGMENTS

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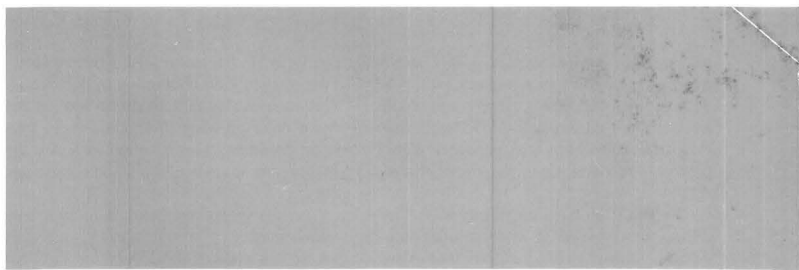
Titanium Dioxide Pigments Industry In the 1990s—Problems and Profits

James R. Fisher

International Business Management Associates*

The titanium dioxide (TiO$_2$) pigments industry is a large and vital one, with over 30 producers having production facilities in 25 countries on six continents. TiO$_2$ annual sales peaked in 1989 with three million tons of pigment valued at approximately $6 billion U.S., while approximately $1 billion of titanium-containing minerals were consumed. Sixty percent of the preferred white pigment is supplied for surface coatings, 15% for plastics, 13% for the paper industry, and the remaining 11% sold to a variety of smaller consuming industries such as synthetic fibers, ceramics, and rubber.

TiO$_2$ pigments are broadly categorized as a quality of life product whose consumption follows changes in GDP and style trends such as recent preferences for white or bright colored surfaces or products.

Production of TiO$_2$ pigments is accomplished by two processes—the sulfate process and the chloride process—in facilities costing as much as $3,500 U.S. per metric ton of installed capacity in a green field site. These facilities have much in common with facilities which produce large volume chemical products, with economies of scale, low cost raw materials, process automation, and minimization of low valued co-products or waste materials important to realizing low production costs. Modern production facilities have annual capacities of 60,000+ metric tons per year with a total fixed invest-

ment in excess of $200 million U.S., and a direct payroll of 400+ people.

Despite the commodity aspects of TiO$_2$ pigment production facilities, characteristics of pigment product technology and customer service requirements are more akin to specialty chemicals. Considerable product technology is needed to manufacture high performance pigment products for their diverse end use markets, where different properties are preferred for each application. For example, a surface coating pigment is markedly different in its performance properties than a paper or fiber grade pigment. Distinctions also exist in pigments used in surface coatings for interior and exterior applications.

The pigment industry is cyclical in nature, following the performance of global economies which drive the demand for its products. The industry enjoyed considerable success in the mid to late 1980s and added new capacity which is now coming on stream. However, the industry presently is having a difficult time. Profit margins are being reduced as pigment selling prices decline with reduced demand because developed global economies are in recession. Margins are further pressured due to recent capacity increases. Pigment production costs for some producers also are increasing, as a portion of the industry invests to meet government regulations controlling effluent discharges.

Predicting profits for this industry is difficult. There is a good correlation between pigment consumption (demand) and GDP

in major geographic markets, and some correlation exists between capacity utilization and pigment selling prices. It also is possible to estimate cash production costs for pigment producers, leading to a computation of typical profit margins for producers in the industry. As with all forecasting techniques, the results are only as good as the input data—the supply demand balance, the price forecast, and estimates of pigment production costs.

As we look at this industry and try to predict its performance in the 1990s, it is worthwhile to look at its performance in the 1980s under similar conditions.

The 1980s

The first quarter of the 1980s saw the pigment industry suffering from over capacity and low selling prices. This followed a period of growth and expansion in the early 1970s, when there was little excess capacity—described by some experienced producers as the golden age of titanium pigments.

	1973	1980
Supply (000T)[a]	2,116	2,552
Demand (000T)	2,058	2,010
Capacity utilization	97%	79%
U.S. pigment price ($/T)	775	1,350

(a) Metric tons.

Despite the rapid increase in selling prices during this period, margins were impacted by the run up in energy and raw

material costs. In the early 1980s, profit margins averaged approximately \$400 U.S. per ton. However, a low cost producer would have enjoyed a margin 300 \$/T higher than this, whereas a high cost producer would have experienced margins approximately 200 \$/T lower.

Very little production capacity was added during the early 1980s, as the pigment production industry consolidated, shut down some older inefficient facilities, and began seriously grappling with new government regulations controlling effluent discharges from pigment plants.

Plant Shutdowns

1981 .... 30,000 TPY Tioxide, in Billingham, U.K.
1882 .... 75,000 TPY NL Industries, in Sayerville, NJ
1984 ...... 40,000 TPY Gulf & Western, in Gloucester City, NJ
1985 .... 30,000 TPY Dow Chemical, in Balboa, Spain

Consolidations

1984 .... SCM acquires NJ Zinc (Gulf & Western) pigment business
1984 .... Kemira Oy acquires American Cyanamid pigment business
1984 .... Tioxide acquires Montedison pigment business
1984 .... SCM acquires LaPorte's pigment business

Effluent Discharges Affected by Regulations

Liquids .... Discharge of acidic wastes and those containing soluble heavy metals into surface waters. Some regulations controlling underground injection wells
Gaseous ... Acid mist, particulates, SO_2 and SO_3
Solids ...... Solid waste landfills and toxic substances, particularly soluble heavy metal compounds

During this period of consolidation and shrinking production capacity, pigment products were readily available in all markets and the major global pigment producers began looking to Third World emerging markets for growth opportunities, particularly in the Asia Pacific market where Japan had established a strong industrial economy, and Malaysia, Thailand, Singapore, South Korea, and Taiwan were showing signs of developing strong, globally focused industrial economies. All pigment consuming markets were beginning to demand improved pigment properties such as durability, gloss, and dispersibility.

During this period in the early 1980s, mineral suppliers to the pigment production industry were also suffering from over capacity and low mineral selling prices. However, higher TiO_2 containing mineral products such as slag and synthetic and natural rutile were growing in demand since pigment producers viewed them as a cost effective way to reduce liquid and solid effluents.

As the global economy entered a sustained period of growth and expansion in the second half of the 1980s, the pigment industry entered its second golden age. Rapid growth in demand far exceeded growth in supply during this period since little pigment production capacity was added from 1980 to 1988. Pigment demand was driven not only by growth in GDP, but also by a strong preference for whiter and brighter colored surfaces and products using more of the improved pigment products being introduced. Pigment selling prices and profit margins dramatically improved from earlier in the decade.

Pigment producers who had achieved a low cost position early in the decade realized very high margins during this period, as much as 350 \$/T above the average. Pigment selling prices also were high enough to provide acceptable profits even to high cost producers, who realized a margin of 450 \$/T, which represented to many of them reinvestment quality profits. During this period of growth and high profits, some producers' attention was diverted from concern with government regulations on effluent discharges and rigorous cost control.

The mineral industry, at this time, also began experiencing acceptable profit margins and quickly expanded its capacity to supply high TiO_2-containing raw materials. Margins justified investing in high capital and high fixed cost manufacturing facilities to produce slag and synthetic rutile. The increased availability and acceptance by the pigment producing industry of manufactured versus mined products dramatically changed the characteristics of this industry—the results of which are now being clearly felt.

As pigment producers began anticipating and planning new green field facilities during the second half of the decade, the preferred pigment production technology was the chloride process. Its advantages over the sulfate process have been well documented. Briefly, they are:

• Reduced liquid and solid effluents—approximately one ton of waste per ton of pigment versus approximately five tons with a sulfate process plant using slag feed.

• Reduced air emissions—chloride process air emissions can be incinerated and scrubbed to meet air quality standards, whereas existing scrubbing technology may not adequately control sulfate plant air emissions.

• Higher performing coating and plastics grade pigment products not available via the sulfate process.

• Favorable production economics typical of a continuous (chloride) process versus a batch (sulfate) process.

Difficulties associated with chloride process technology are less frequently discussed but are experienced by all chloride process producers. They are:

	1984	1985	1986	1987	1988	1989
Supply (000T)	2,582	2,633	2,772	2,849	3,002	3,171
Demand (000T)	2,364	2,491	2,605	2,743	2,910	2,933
Capacity utilization	92%	95%	94%	96%	97%	92%
U.S. pigment price (\$/T)	1,450	1,575	1,660	1,750	1,950	2,270
German pigment price (DM/T)	3,170	3,770	4,080	4,140	4,340	4,660
Average margin (\$/T)	450	550	600	630	800	1,000

• Complex unit operations linked closely together, all of which must operate satisfactorily to achieve an acceptable operating, or on-stream, factor. This operating factor generally determines whether a chloride process pigment plant can achieve acceptable production economies and is a good measure of a producers experience.

• Hazardous materials such as chlorine and titanium tetrachloride being reacted, recovered, and handled at elevated temperatures.

• Complex machinery involving exotic materials of construction and control systems all requiring careful monitoring and continuous maintenance with skilled personnel.

The prosperity enjoyed by the industry in the second half of the 1980s masked some fundamental, pervasive problems which remain largely unresolved and are exacerbated by current economic and industry conditions.

(1) The inability of most sulfate process pigment producers to comply with government regulations controlling effluent discharges in a cost effective way. This is not a universal condition as some sulfate process plants enjoy low production costs such that additional effluent treatment costs do not

result in uncompetitive production economics.

(2) Chloride process technology capable of being implemented in large scale facilities with low operating cost is available only to a few pigment producers. The economics of operating a chloride process pigment plant clearly follow the experience curve. This, coupled with its intrinsic technical and design problems, restrict its successful economic operation.

(3) High quality end use applications such as automotive coatings demand high performance pigment products. Chloride process producers find it easier to meet these performance standards than sulfate process producers.

(4) Some high cost producers face the dilemma of having to invest in cost increasing assets needed to comply with government regulations controlling effluent discharges. These can be truly nonproductive investments if the pigment producer is unable to economically recycle or sell the recovered, former waste materials, or remains constrained to produce pigment products which are inferior to newer product offerings.

The 1990s

The pigment production industry has entered the 1990s facing many of the same problems of a decade ago: overcapacity, soft pigment demand and weak pigment selling prices, and cost effective compliance with government regulations.

	1989	1991
Supply (000T)	3,171	3,284
Demand (000T)	2,933	2,785
Capacity utilization	92%	85%
U.S. pigment price ($/T)	$2,270	$1,980
German pigment price (DM/T)	4,660	3,900

During the 1989 to 1991 period, 113,000 tons of new capacity began operating. During this same period, demand declined 148,000 tons. The industry was anticipating a reduction in demand following the 1985 to 1989 growth period, but did not foresee how deep and broad the economic recession driving demand down would be. Pigment demand declined 2.5% per year in North America from 1989 to 1991, 7.5% per year in Western Europe, while growing 2% per year in Asia/Pacific and the rest of the world markets. The 15% excess capacity currently experienced by the industry is exacerbated by the resistance of higher cost producers to permanently shut down older inefficient facilities. Continued poor economic conditions may change this.

Economic forecasts for global economies in the 1990s include deflation in North America and West Europe with continued growth in Asia/Pacific and Third World markets having western-style quality of life consumption. Producers will adjust to modest demand growth in North American and Western European markets by concentrating on cost reduction programs, compliance with government regulations, and attempting to penetrate Asia/Pacific and Third World markets in competition with new production capacity in these areas. Growth in demand during this period will not consume excess capacity, consequently the industry cannot expect to grow its way out of its excess capacity condition.

In line with this excess capacity and soft demand, pigment prices fell 13% in the U.S. from 1989 to 1991 and 16% in Germany.

Cost effective compliance with effluent discharge regulations is difficult and in some cases not possible. Cleaning up liquid and solid effluent from a sulfate process plant is more costly than from a chloride process plant because there is more of it. Chloride process plants typically use higher TiO_2-containing minerals and the reduced quantities of metallic salts or hydroxides, mainly iron, can be landfilled. Recovering and recycling sulfate process plant wastes has received considerable attention and careful scrutiny. Processes which recover iron sulfate and sulfuric acid are expensive, with capital costs as high as $1,500 per ton of pigment capacity and can add as much as $160 per ton to operating costs. Selling iron salts into secondary markets such as waste water treatment, iron pigments, animal feed, or fertilizer takes considerable time and expense in an effort to develop commercial and technical organizations, specialized products, and channels of distribution.

Issues which have not been addressed but will capture the attention of the pigment industry in the 1990s are regulatory, economic, and technical.

Regulatory issues include: the economic treatment of weak acid streams from both sulfate and chloride process pigment plants; obtaining suitable solid waste disposal sites; and meeting point source standards on air emissions from sulfate process pigment plants.

Economic questions concern increased production cost differences between high cost and low cost producers.

Technical problems include improved pigment product performance and quality standards.

Briefly stated, low cost pigment producers with large plant facilities and greater purchasing power have been better able to manage and control their costs, whereas higher cost sulfate process pigment producers have not been as successful and are now realizing the effects of increased effluent disposal costs.

Improved pigment products are being utilized by some pigment producers to penetrate new markets or defend share in established markets. This has increased expectations of pigment consumers such that they now demand, and receive these improved products from a large number of producers. Some pigment producers have successfully embraced quality as a marketing tool and are attempting to create a higher valued quality image with their customers.

Current conditions in the Asia/Pacific pigment market are worth reviewing as this is a market where the major, global producers are focusing their efforts, and there could be some interesting parallels to other markets in the 1990s.

The Asia/Pacific market has enjoyed good growth in pigment demand, growing at a compound annual growth rate of 6% per year from 1985 to 1991. It is a large market which consumed approximately 600,000 tons of pigment in 1990, and it contains some dynamic, innovative, and aggressive industrial manufacturers whose consumption of pigments will continue growing at a greater rate than other markets.

Because of the attractive opportunities, numerous producers constructed capacity in this region to facilitate penetrating these markets: Ishihara, Singapore, 40,000 TPY chloride, 1990; SCM, Australia, 70,000 TPY chloride, 1990; Tiwest, Australia, 56,000 TPY chloride, 1991; Tioxide, Malaysia, 50,000 TPY sulfate, 1992; and other Far East producers, 35,000 TPY sulfate, 1988-1991.

In addition, Du Pont and Tioxide have increased exports to this market in anticipation of their new planned capacity: Du Pont, Taiwan, chloride 60,000 TPY; and Tioxide, Australia, chloride 60,000 TPY.

With this new capacity either in place or being constructed, the leading local Asia/Pacific producers are defending their position by forcing new entrants to match high quality products at lower prices. Aside from benefitting the Asia/Pacific pigment consumers, this is forcing new entrants to provide higher quality, improved products, and carefully control pigment production costs. A side effect is the impact Asia/Pacific conditions are having on Western Europe, where European producers who traditionally exported to the Asia/Pacific market are being forced out, exacerbating problems of excess capacity and stagnant or declining demand in European markets. The net result is that economic pressure is focusing on some older, high cost sulfate capacity in Western Europe.

In developing a forecast for the remainder of this decade, one must start with a set of assumptions. The following assumptions are a composite of what professional economists are saying, and what the industry's performance in the 1980s portends for the 1990s.

• Deflation in developed economies is a real possibility, meaning that demand growth

will be stagnant or at best erratic in the early to mid-1990s. Sustainable, strong demand growth in developed economies will not be realized until the middle of the decade, at the earliest. Driving this deflation is continued problems in the U.S. with its deficit and liquidity of funds. In Europe, Central and Eastern European problems are more difficult and costly than originally perceived, diverting funds from financial institutions (mainly German) which provide financing for much of Europe's industrial growth.

• Technological barriers are real in the pigment producing industry and their significance will be more widely felt as producers attempt to reduce costs.

• Niche pigment markets will be difficult to defend as low cost producers penetrate established, growing markets.

• The pigment producing industry will not grow out of its present overcapacity problems—closures and consolidations will occur in the 1990s.

From this, supply demand forecasts can be constructed. Two forecasts have been prepared. The optimistic forecast sees modest growth returning in the developed economies in 1992 and 1993, and some capacity shut down in the same period (*Table* 1).

A pessimistic forecast was prepared where stagnant demand growth and large excess capacity continues through the middle of the decade (*Table* 2).

Table 1— Optimistic Forecast	1989	1991	1995	2000
Supply (000T)	3,171	3,286	3,555	3,887
Demand (000T)	2,933	2,785	3,242	3,683
Capacity utilization	93%	85%	91%	95%

Table 2— Pessimistic Forecast	1989	1991	1995	2000
Supply	3,171	3,286	3,784	3,887
Demand	2,933	2,785	3,078	3,491
Capacity utilization	93%	85%	81%	90%

Table 3— Price Forecast							
Optimistic Scenario	1991	1995	2000	Pessimistic Scenario	1991	1995	2000
Capacity utilization	85%	91%	95%	Capacity utilization	85%	81%	90%
U.S. price per ton	$1,980	$2,300	$2,600	U.S. price per ton	$1,980	$1,790	$2,300

Attaching a pigment price forecast to these scenarios is difficult because of the large number of variables impacting pricing. However, assuming the industry will react to capacity utilization and general economic conditions as it has in the past, price forecasts can be made (*Table* 3).

Despite the poor economic conditions we are experiencing today, we believe the optimistic scenario will prevail, prices and margins will improve, and the 1990s will provide opportunities for the imaginative, aggressive producers to expand their businesses. This industry has demonstrated a remarkable ability to solve its problems while generating substantial profits.

January 1993 Subcommittee Reports of ASTM Committee D-1

The January meeting of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications was held on January 17-20 at the Crown Sterling in Fort Lauderdale, FL. In the three and one-half days preceding the final session and general meeting of Committee D-1, 240 members and guests met in about 200 scheduled meetings of D-1 and working task groups. The current membership of Committee D-1 is 600.

Highlights

D01.08—Increasing focus at the U.S. Environmental Protection Agency on the reduction of hazardous air pollutants highlights the need for increasing attention in D-1 on the analysis of these materials in paints and coatings.

D01.21—The second edition of the ASTM Technical Manual "Determination of the Volatile Organic Compounds (VOC) in Paints, Ink and Related Coating Products," is scheduled for release mid-year. Two workshops on measuring VOC content in paints are planned for 1993: May 5-6 in Chicago, IL, and November 3-4 in El Monte, CA. Information on the workshops can be obtained by contacting Matt Lieff, of ASTM.

D01.24—Work to develop a guide on detection, identification, and characterization of odors of paints, inks, and related materials is commencing. The committee is searching the literature for information and inviting interested people to join the task group.

D01.42—A new task group was formed to develop a guide on exterior stains. Interested people are invited to participate.

D01.46—A task group has been formed to develop a practice for field sampling samples of coatings films for heavy-metal analysis.

D01.55—In a task group on scratch resistance of automotive topcoats, a mechanical "brush test" with image analysis of defect formation is being investigated.

D01.57—A new task group on colored pencils was established to develop a specification for artists' colored pencils.

New Standards

New standards approved by the ASTM Committee on Standards since the June 1992 meeting are (subcommittee jurisdiction):

D 5326—Standard Test Method for Color Development in Tinted Latex Paints (D01.42);

D 5328—Standard Test Method for Volatile Organic Compound (VOC40) Content of Non-Heatset Paste Printing Ink Systems at 40°C (D01.56);

D 5327—Standard Practice for Evaluating and Comparing Transfer Efficiency Under General Laboratory Conditions (D01.55); and

D 5235—Standard Test Method for Microscopical Measurement of Dry Film Thickness of Coatings on Wood Products (D01.52)

Future Meetings

June 27-30, 1993—Franklin Wyndham, Philadelphia, PA.
January 23-26, 1994—Crown Sterling, Fort Lauderdale, FL.
June 26-29, 1994—Raleigh, NC.
January 22-25, 1995—Hyatt Regency, San Antonio, TX.

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DIVISION 1 ADMINISTRATION

SUBCOMMITTEE D01.08 ENVIRONMENTAL CONCERNS

J.J. Brezinski, Chairman

Topics discussed at the meeting include a progress report of the ongoing negotiated rulemaking regarding VOC control of architectural/industrial maintenance coatings (AIM); MSDS-related documents; EPA programs on VOC in consumer and commercial products; and VOC-related standards development in D-1.

Negotiated Rulemaking AIM—R. Klesper and M. Gaschke reported that data from an industry survey was used in the development of an initial industry proposal for the control of VOC content in AIM coatings and of recommended VOC levels for a number of specialty architectural coatings. Future meetings will review the industry response to questions presented by the facilitator related to the industry survey as well as discuss other proposals that may be presented. EPA plans to issue a proposed rule in late summer of 1993 with a final rule scheduled for January 15, 1994.

MSDS-Related Documents—Ongoing activity on MSDS-related documents includes:

• The American National Standards Institute (ANSI) is expected to recommend a standard format for preparing MSDSs in the spring of 1993.

• Committee E-34 on Occupational Health and Safety, in response to a request from the General Service Administration

(GSA), is nearing completion of the development of a "Practice for Preparing Material Safety Data, Transportation Data and Disposal Data Sheets for Hazardous Materials for the General Services Administration." The general practice includes as referenced documents the related ANSI standards, NPCA's Hazardous Materials Identification System (HMIS) and Canada's Hazardous Product Act (WHMIS).

No interest was expressed in the development of a guide in D-1 that would tabulate ASTM standards that could be used to develop physical property data on paints for inclusion on the MSDS.

VOC in Consumer and Commercial Products—Projects at EPA dealing with consumer and commercial products used in people's homes include the use of a source ranking data base to characterize (rank) 270 chemicals founds in these products according to the known or projected adverse health effects. Of these chemicals, all but 32 ranked of high to medium concern. Other projects at EPA include identifying the source categories that account for 80% of the VOC emissions from consumer/commercial products and dividing the categories into four groups for future regulatory attention.

The increasing focus at EPA on the reduction of hazardous air pollutants suggests the need for increasing attention in D-1 on the analysis of these materials in paint and coatings.

VOC-Related Standards—Standards that have advanced to the society letter ballot (from D01.55) include: "Volatile Content of Radiation Curable Materials" and the "Determination of Transfer Efficiency Under General Production Conditions for Spray Application of Paints." If approved and available in time, these standards will be included in the appendix of the second edition of the ASTM manual "Determination of Volatile Organic Compounds (VOC) in Paints, Inks, and Related Coating Products," expected to be published in mid-1993.

DIVISION 20
RESEARCH
AND
GENERAL MEETING

SUBCOMMITTEE D01.21
ANALYSIS OF WHOLE PAINTS
AND PAINT MATERIALS

K.H. Fujimoto, Chairman

D01.21.10—Lead Paint Policy—J.C. Weaver, Chairman, met with members and guests to discuss the greatly increased concern over reported excess blood lead levels in small children across the U.S.A.

The housing act passed by Congress in 1992 has many parts of which Title X is a complex and lengthy dictum on the abatement of lead in housing by HUD. HUD appropriated over $40 million to be spent by 10 large cities, conditional upon state/local matching funds and other stipulations. Fannie Mae has appropriated over $5 million to a new lead abatement foundation in Columbus, MD, with a stipulation that its Board of Directors and advisory committees be balanced for fairness. This implies representation by paint industry experienced technologists who know both historic paint formulations and modern sampling and analytical procedures.

Subcommittee E06.23 has held six meetings since their March 11, 1991 formation with M. McKnight as their chairperson. Most of E06.23's thirteen task groups met on January 14-15, 1993 to advance 10 or more draft standards. There is a glaring lack of commonality of membership between the mainly environmental advocacy bodies and the paint community technologists.

Task group D01.57.08, chaired by S. Stopford, is in the final stages of a subcommittee ballot on a gastric solubility concept of analysis for total lead extractable in artists' colors. This gastric method is adapted from the practice used in Europe.

As a result of EPA's study of lead in soils in the cities of Boston, Baltimore, and Cincinnati, a final draft report is to be released. This midterm 1991 report contains strong inferences that much of the lead in children's blood comes not from paint, but from soil, dirt, and dust; lead fallout from gasoline is suspected.

Many of the 50 states have lead abatement legislation in various stages of draft and passage. However, their great disparities call for the paint community to attempt to approach the legislators to rationalize these bills toward the Minnesota lead law concept of the "Swab-Team" as the most efficient, quick, and least costly control of lead in homes and environmental surroundings.

On July 25, 1993, in Boulder, CO, a broad program on diverse lead hazards and abatements will be sponsored, jointly, by Committees D-1, D-18, D-22 and E-06. With many speakers not confirmed, D-1's only contribution is a paper to be given by J.C. Weaver on the theme, "Tain't Paint, 'Tis Petrol that Taints Children's Blood With Excess Lead."

D01.21.13—Coordination of VOC Standards and Information—J.J. Brezinski, Chairman, used transparencies to present an overview of the major portions of the Clean Air Act of 1990 and highlighted those amendments which have affected and which will affect the coating industry today and in the near future. The information presented was timely, useful, and up-to-date.

The current status of the work being done by the EPA related to the preparation of Control Technique Guidelines and National Rules for coatings used on plastic automotive/transportation parts was discussed by E. Praschan.

The overview of the Clean Air Act of 1990 indicates that there will be increased regulatory action focused on the control of various paint solvents and those materials characterized as hazardous air pollutants. It is, therefore, important that D-1 develop test methods which will identify and quantitate these paint components.

D01.21.14—VOC New Publications and Workshops—J.J. Brezinski, Chairman, announced the ASTM peer review of the draft of the second edition of the ASTM technical manual, "Determination of the Volatile Organic Compounds (VOC) in Paint, Inks and Related Coating Products," has been completed, and appropriate changes have been made. The final revised document will be forwarded to ASTM headquarters by mid-February and publication is expected by mid-year.

A review of the analysis presented in the manuscript suggests the following needs:

—Development of precision data which uses the calculation for VOC content based on applied volume solids—after an acceptable consensus, an ASTM standard is developed for the determination of dry coating density and the related percent volume nonvolatile content.

—Mathematical analysis, which uses the minus water equation, showing the VOC content of water-reducible coatings as the VOC content decreases (and the water content increases), and as the values for volatile content, paint density, and water content vary within the range of the precision limits indicated in Federal Reference Method 24.

The 1993 Paint Volatile Organic Compound workshops are designed to instruct persons in the use of U.S. EPA Method 24 tests to determine if paints or coatings meet VOC requirements. Workshops are scheduled for: May 5-6, 1993—Chicago, IL; and November 3-4, 1993—El Monte, CA. Contact Matt Lieff, ASTM, (215) 299-5516.

D01.24.24—Revision of D 2369-92 Test Method for Volatile Content of Coatings—M.E. Sites, Chairperson, presented results on the latest revision of D 2369, which received two negative and seven affirmatives with comments on a D-1 ballot. Many excellent editorial comments were received and will be incorporated where appropriate. The negative received from M.A. Urnezis was editorial in nature and has already been taken care of.

K.H. Fujimoto cast a negative for the following reason: "Note 10 is not scientifically correct since no round-robin testing was done to verify whether an induction

period of 24 hours would give the same results as the one-hour induction period used in the test. The note should be placed in D 3960 and removed from D 2369."

Preliminary tests show that if longer induction periods are used, the surface of the film cures trapping solvents which lowers the VOC numbers. Even though a few members claim a 24-hour induction period is necessary for some multiple component system, no data has ever been submitted to substantiate this claim.

K.H. Fujimoto distributed a letter from M. Mahon which showed that in a limited trial conducted using one hour up to a 24-hour induction period, indicating that, "concern over possible solvent entrapment is valid."

After considerable discussion concerning "Note 10," three motions were passed:

(1) A motion to find K.H. Fujimoto's negative persuasive;

(2) A motion to change "Note 10" to make it acceptable in terms of the scope of the method and compatible with the precision statement which is based on round-robin studies using a one-hour induction period. There should be nothing said or implied about a 24-hour induction period.

(3) A motion to change "Note 10" to read: "Other induction periods are used. See U.S. EPA Reference Method 24."

The revised D 2369 will be withdrawn from ballot, and it will be reballoted concurrently on the next D01.21/D-1 ballot.

D01.21.24A—Ion Chromatography for Electrocoat Bath Samples—M. Mahon, Chairman, presented data from a recently completed ion chromatography (IC) round-robin study to determine the anions present in a cathodic electrocoat permeate bath sample. The permeate sample was spiked with 25 ppm of a nitrate anion and 50 ppm of a phosphate anion. None of the five participating laboratories could detect phosphate, but all could determine the nitrate anion with good quantitative accuracy.

Based on the close agreement of the data to the theoretical values for the nitrate anion, a standard guide for analyzing electrocoat permeate samples by IC was prepared and distributed for discussion. As a result of the comments received, some editorial revisions were made to this proposed method. J. Benga agreed to assist Mr. Mahon in re-evaluating the Precision and Bias section of the document by using ASTM E 691. Once this work is completed, the method will be submitted for concurrent D01.21/D-1 ballot.

D01.21.25A—Method to Determine the Amount of VOC Released from Automotive Waterborne Coatings—J. Komjathy, Chairman, reviewed the status of the method after a D-1 ballot. A comment received from the March D-1 ballot suggested that, if possible, the symbols and calculations used in this method should be made identical to those used in D 3960 and/or those symbols used in the two EPA methods. At the June 1992 meeting, task group D01.21.25A requested Mr. Komjathy to review the method and ascertain if any of the symbols could be changed. If the symbols and calculations are found not to be interchangeable, then a note should be added which states an effort was made to incorporate symbols and calculations used in other methods, e.g., D 3960. In addition, it should be stated that the symbols and calculations used in this method are unique to this test method. This request had been fulfilled as Section 1.4.

The revised method was sent out for D01.21 ballot in January 1993. To date, only one negative has been received, and this has been withdrawn. Task group D01.21.25A voted to place the method on a D-1 ballot.

Round-robin test results are expected by May 1993 and if possible, a precision statement will be generated for the next June 1993 meeting.

D01.21.26—Revision of D 2697, Percent Volume Nonvolatile Content Using the Helium Gas Pycnometer—B.L. Neff, Chairman, reviewed the actions taken during the previous meetings and the scope of this task group. The first item of discussion was a letter by the chairman to the U.S. EPA's Messrs. Jim Berry and Gary McAllister which discusses the justification for a sample preparation change in the current method. Since the chairman has not received any negative comments, it is assumed the sample preparation procedure is acceptable.

D. McCunn suggested a video be made to illustrate the procedure and technique used to obtain dried/baked free paint films. The chairman will look into the feasibility of preparing a demonstration video.

H. Stanley offered to send loaner pycnometer units to the round-robin collaborators. This will include set-up and use instructions along with a calibration sample. Mr. Neff extended his appreciation for Micromeretics' generosity and for making his task easier to inaugurate his round-robin work.

It was agreed that Mr. Neff will select three types of paint samples for the test and insure that the samples are sent to the collaborators with detailed instructions. Micromeretics will be notified and will supply helium gas pycnometer loaners. The chairman hopes to have the necessary data by the next meeting to ascertain the viability of the new sample preparation procedure.

D01.21.27A—VOC Content of Aerosols—R. Osterman, Chairman, reported that the "Determination of the Percent Weight Solids of Solvent Borne Aerosols" has passed society ballot, and it has been placed in the *ASTM Books of Standards* as D 5200.

Method D 5325, "Determination of the Percent Weight Solids of Waterborne Aerosols," will be on the March 1993, society ballot. Both D 5200 and D 5325 will be submitted to the U.S. EPA for their approval.

D01.21.27B—New Approach to VOC—R.K.M. Jayanty, Chairman. In the absence of Chairman Jayanty, K.H. Fujimoto chaired the meeting.

The objective of this task group, to develop a direct procedure to determine the VOC of water-reducible paints, was reviewed. It was hoped this direct method would eliminate or minimize the inherent calculation errors present in the U.S. EPA's Reference Method 24.

Based on the round-robin results of what appeared to be "apparently good data," Chairman Jayanty transposed the method into the ASTM format, recalculated the VOC data as "Weight of VOC/Volume of Paint Minus Water" instead of the standard deviation motif, and developed an ASTM precision statement. The revised method was sent out in August 1992 to D01.21 ballot. Nine negatives and three affirmatives with comments were received in the ballot.

Instead of addressing all of the negatives, the task group decided that if one of the negatives was found to be persuasive, the method would have to be withdrawn and revised. The other negatives and comments would be studied and handled at a later date.

K.H. Fujimoto's negative was chosen, discussed, and found persuasive. His negative stated: 1) Paragraph 9, Precision & Bias, was difficult to follow and understand; 2) repeatability of 50% was unacceptable; 3) the method does not collect methanol or low MW alcohols; 4) the apparatus is too complicated and difficult to run; 5) no comparable commercial apparatus is available; 6) the method does not duplicate D 2369 so it would give different VOC results; 7) more round-robins and improvement in equipment are needed; and 8) commercially available and light-in-weight charcoal tubes should be tried. The method will be withdrawn from letter ballot and further improved.

Other comments received were: 1) the method is still in the experimental stages; 2) too large a mass of carbon and moisture absorbent tubes used to measure the small differences in weight; 3) use other commercially available absorbent; 4) simplify equipment; 5) simplify leak checks; 6) too time consuming a method to be used routinely; and 7) the method redefines VOC.

The task group felt the work should be continued to improve this VOC measurement method.

D01.21.46—X-Ray Analysis of Pigment—M. Mahon, Chairman, discussed the comments which he received on the three items on D-1 ballots. Minor editorial cor-

rections were made to two documents: 1) "New Standard Test Method for the Identification of Crystalline Pigments and Extenders in Paint by X-Ray Diffraction Analysis; and 2) "New Standard Guide for X-Ray Fluorescence Spectroscopy of Pigments and Extenders." These two documents will move to society ballot.

Comments received concerning the reapproval of D 4764, "Standard Test Method for the Determination by X-Ray Fluorescence Spectroscopy of Titanium Dioxide Content in Paint," were discussed. It was agreed by all of the task group members that definitions of the symbols used in Section 10 were adequate, and, therefore, no revisions are necessary. This method will go on to society ballot.

Following this meeting, task group 46 will be placed on "inactive" status.

D01.21.49—Sample Preparation for Trace Metal Analysis—B. Neff, Chairman, discussed a proposed sample digestion method for trace metal analysis. In summary, this method is an enclosed (at atmospheric pressure) wet digestion scheme which uses commercially available equipment.

The results of a current round-robin were discussed. The round-robin samples consisted of an alkyd and acrylic resins inoculated with known amounts of lead and cobalt. Three laboratories (Du Pont, Glidden, and PPG) reported their data.

The data for cobalt shows excellent agreement among the laboratories.

Lead presented a problem as it formed a precipitate under the digestion conditions. The task group decided to continue with other metals which will be chosen after the meeting. It was agreed a caveat will be added to the procedure to check precipitates for lead content. Another round-robin is planned.

D01.21.51—Free Formaldehyde in Paints—D. McCunn, Chairman, reviewed the discussions which took place at the June 1992 meeting. S. A. Kail proposed the use of an OSHA approved method, #52, to measure the evolved formaldehyde. The task group indicated that the room temperature evolution of formaldehyde, collection by the air sampling tubes and measurement by gas chromatography connected to a nitrogen selective detector, should be both selective and sensitive. Hopefully, this approach will meet the task group's initial goal of measuring "free" formaldehyde in air-dried water-reducible paints.

Ms. Kail will provide sampling tubes to J. Benga and D. McCunn who agreed to evaluate the proposed method. Assuming success, the chairman will draft a method and a round-robin will be conducted prior to the next meeting. Currently five or six collaborators will analyze two or three samples which will be supplied by Glidden.

D01.21.52—Paint Solvent Analysis by Gas Chromatography—J. Lamberton, Chairman, reviewed the initial mission of this task group which is to utilize D 3271 methodology, as initially modified by the chairman, to quantitate a selected group of solvents found in water-reducible paints.

Two solvent standards and two paint samples are to be sent to the round-robin collaborators. Both polar (Carbowax 20M type) and non-polar (SE 54 type) fused silica capillary columns, 30 M x 0.32 mm I.D. with a 1.0 μ stationary phase coating are to be used by each participant.

At first, a flame ionization detector will be used. However, a mass spectrometric detector is to be considered as part of the final method. The results of the round-robin are to be considered at the next meeting in order to determine if the direction of the task group is correct.

D01.21.54—Revision of D 4017, Percent Weight by Karl Fischer Titration—M. Mahon, Chairman, led a discussion centered on a recent round-robin study of a proposed methodology for the Karl Fischer analysis of latex paints using methanol extraction. One laboratory reported difficulty in keeping the sample-resin system dispersed during the ultrasonification step. Another laboratory found handling methanol and keeping it "water-free" problematic, and they discovered that other alcohols such as isopropanol worked as well. The proposed procedure will incorporate the use of additional alcohols in the extraction steps.

After a lengthy discussion, the task group decided to rewrite the proposed extraction method into ASTM format, incorporate the procedure in D 4017, and utilize the data obtained thus far to develop a precision statement. Additionally, a note will be added to D 4017 allowing the use of Hydranal® reagent in place of the pyridine containing reagent currently specified in the method.

D01.21.56—Revision of D 3960-92. Standard Practice for Determining Volatile Organic Compounds (VOC) Content of Paints and Related Coatings—M. E. Sites, Chairman, discussed the latest revision of D 3960-92, which was balloted on a D-1 ballot. It received no negatives and one comment. This standard will be placed on a society ballot to become D 3960-93. Although this standard has undergone constant annual revisions since 1985 to keep it up to date, we anticipate more and constant changes.

The purpose of the latest revision was as follows:

—Delete under "Scope" the last phrase referencing VOS (volatile organic substance) since New Jersey has changed from VOS to VOC to be in compliance with the EPA terminology.

—Add D 5095 to the section of "Reference Documents" since the EPA has approved the use of D 5095-90 for the determination of VOC content of silane and siloxane coatings.

—Under Section 6.22, add reference to the D 5095 method.

—In "Appendix X3," replace "X3", regulatory definition of VOC with the revised definition published by the EPA under 40 CFR, Part 51, *Federal Register*, Vol. 57, No. 22, February 22, 1992, PP3941-3946.

—The comment received from M. Rossol concerned the lack of consistency between definitions of ozone depleting chemicals in 40 CFR 51 and in 40 CRF 82. She hoped all definitions that apply to CFCs could be made consistent. M. Rosso withdrew her comment when it was shown the two definitions referred to "good" and "bad" halohydrocarbons, i.e., those which react immediately and those which eventually drift to the stratosphere where they can destroy the protective layer of ozone.

D01.21.80—Exploratory Analytical Research—K.H. Fujimoto, Chairman, reviewed the purpose of task group 80 which is: 1) to review new or needed test methods; 2) to ascertain whether proposals made to D01.21 have merit and deserve further study and consideration; and 3) to recommend the formation of new task groups.

J.C. Weaver submitted two items: 1) use of a Parr bomb for the degradative analyses of reactive coating resins; and 2) the potential use of a portable laser spark surface mass analyzer.

Item 1 is based on an article by J.K. Haken in which he proposes the use of a modified Parr bomb to alkali and/or acid fuse resin into volatile components to be analyzed by gas chromatography or HPLC. The heavier components are derivatized to enhance their volatility and identification.

Since none of the task group members use the Parr bomb due to the fear of possible explosion, and since no interest was shown, the proposal was dropped.

Item 2 referred to a portable laser spark surface mass analyzer, which is used to obtain rapid measurements of surface contaminants, such as lead, at the 0.06 milligram/sq cm level. Other hazardous materials can also be determined. The instrument appears to be an emission spectograph. A laser is used to form a microplasma or spark to atomize and electronically excite the elements that make up the paint or other target materials.

The chairman will inquire to determine the availability of the equipment and to obtain any data to substantiate the sensitivity of the equipment.

Since there was no interest in developing an "OH" determination method for resins and polymers, no action was taken on this matter.

SUBCOMMITTEE D01.22
HEALTH AND SAFETY

J.J. Brezinski, Chairman

D01.22.01—Flammability and Combustibility—R. Osterman, Chairman, reported increased interest in promoting international acceptance of the use of ISO TR 9038, "Determination of the Ability of Liquid Paints to Sustain Combustion," as a means of discriminating between liquid products in the same nominal flash point range that do and do not sustain combustion. Future acceptance by the UN Committee of Experts on the Transport of Dangerous Goods could lead to revised flammability classification of those water-dilutable materials, including paint products, that meet specified test criteria.

A round-robin study has been organized to test the acceptability as an alternate to ISO 9038 of a modification of D 4206, "Sustained Burning of Liquid Mixtures by the Setaflash Tester (Open Cup)," using 55 and 75°C test temperatures. Materials to be studied include several water-dilutable paint products and selected water/alcohol mixtures previously tested in ISO TC 35 using ISO 9038. The closed cup flash points will be defined using D 3278.

ERDCO Engineering has offered to convert setaflash closed cup testers to open cup (and to reconvert) for round-robin participants. Persons wishing to join the study should contact R. Osterman, (414) 947-6857 or S. Orthey, ASTM, (215) 299-5400.

D01.22.02—Chromates, Health and Safety Standard—J. Brezinski, Ad hoc Chairman, reported that E 848, "Practice for Safety and Health Requirements Relating to Occupational Exposure to Water-Insoluble Chromates," will be included in the March society letter ballot showing the corrected placement of the lines and headings in Table 1. Suggested changes in the appendix of literature references will be addressed in the next revision.

SUBCOMMITTEE D01.23
PHYSICAL PROPERTIES OF
APPLIED PAINT FILMS

P.R. Guevin, Jr., Chairman

D01.23.10—Adhesion—G. Nelson, Chairman, reported the proposed addendum of D 3359-90, "Test Methods for Measuring Adhesion by Tape Test," completed D01.23 letter ballot. Several editorial comments were received which will be incorporated into the addendum and submitted to concurrent ballot before the next meeting.

The project to evaluate adhesion using tapes with varying adhesive strengths has been hindered by the inability to find a coating with consistent "mediocre" adhesion for testing. Issues relevant to D 5179, "Test Method for Measuring Adhesion of Organic Coatings to Plastic Substrates by Direct Tensile Testing," and D 3002, "Standard Practice for Evaluation of Coatings for Plastics," currently under revision, were discussed.

D01.23.12—Dry Film Thickness—S. Boocock, Chairman, reported two negatives were received on concurrent D01.23/D-1 ballot of revisions of D 1186-87, "Test Methods for Nondestructive Measurement of Dry-Film Thickness of Nonmagnetic Coatings Applied to Ferrous Base," and D 1400-87, "Test Methods for Nondestructive Measurement of Dry Film Thickness of Nonconductive Coatings Applied to a Nonferrous Base." One negative was found persuasive and the items were withdrawn from balloting for further revision.

P.R. Guevin, Jr. reported on work being conducted on the use of ultrasonic instrumentation to measure the dry film thickness of organic coatings. Further work will be conducted in task group D01.23.20 on Exploratory Research.

D01.23.14—Hardness, Abrasion and Mar Resistance—P.R. Guevin, Jr., Chairman. A. Rutkiewic reported for B. Gregorovich on the results of the second round-robin on the proposed "Test Method for Mar Resistance of High Gloss Coatings." The method uses a Taber Abraser to measure coating mar resistance as defined by a change in gloss. Variations in the results from three of the seven cooperating laboratories were discussed. The problem of the size of the port on most glossmeters in relation to the area being marred, as well as possible changes in the gloss calibration standards currently used to meet ISO standards, were discussed. Cooperators for the next round-robin will use new abrasive wheels, mechanical refacers, and a gloss-calibration ceramic tile.

A discussion on the proposed revision of D 2134, "Test Method for Determining the Hardness of Organic Coatings with a Sward-Type Rocker," centered on variations in the weight outlined in the method versus one of the currently marketed rockers. N. Bender will evaluate these variations to determine if weight affects test results. Three types of the Sward-Type Rocker with various weights will be used.

D01.23.15—Slip Resistance—P.R. Guevin, Jr., Chairman, read M. Morse's report on the status of round-robin activity to develop a precision statement for D 4518-91, "Test Methods for Measuring Static Friction of Coating Surfaces." Additional tests will be run using a calibrated spring horizontal pull slip tester and a method to calibrate the spring will be written.

The chairman reported on a survey conducted with subcommittee members to determine industry awareness of government legislation involving proposed OSHA and in effect laws, e.g., ADA (American with Disabilities Act), on specified coefficient of friction (COF) values, and test methods for walking surfaces. The small number of responses was disappointing.

The chairman reported on activities in other ASTM Committees and the workshop held at Bucknell University evaluating various static and dynamic COF slip testers.

D01.23.16—Water Vapor Transmission—T.J. Sliva, Chairman, reported the addendum to D 1653-85, "Test Method for Water Vapor Transmission of Organic Coating Films," is currently under D01.23 ballot. The chairman will revise the addendum to incorporate the editorial comments received and submit the item to concurrent ballot before the next meeting.

It was the decision of the task group not to introduce the terms "wet" and "dry" perms to the method, to make reference to the inverted cup method or, to define the term "moisture vapor barrier." H. Ashton will report on the results currently under study by the NRC on moisture vapor transmission methods at the next meeting.

D01.23.18—Flexibility—P.R. Guevin, Jr., Chairman, reported the revision of D 522-88, "Test Methods for Mandrel Bend Tests of Attached Organic Coatings," completed D-1 ballot and will proceed to society ballot if suggested additional comments are deemed editorial.

Editorial comments were suggested to revise the footnotes on equipment sources in D 2794, "Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)." With no further activity at this time, the task group has been made inactive. The revised test method will be submitted for concurrent ballot.

D01.23.19—Mechanical Drying Time—T.J. Sliva, Chairman. Subcommittee D01.23 has responsibility for D 1640, "Standard Test Methods for Drying, Curing or Film Formation of Organic Coatings at Room Temperature." The method will be reviewed for editorial and technical revisions and balloted before the next meeting.

The chairman distributed copies of the first draft of the proposed test method for determining drying characteristics of organic coatings using mechanical drying time recorders. Task group members will review the document and submit suggested changes. H. Ashton reported that data on the circular drying time recorder developed by Carlisle Chemical Works was not sufficient for a Precision and Bias statement, and the task group will need to run a round-robin using this device. At the present time, the task group will initiate a round-robin using only the straight line drying time recorder. Four cooperators agreed to participate. The group will evaluate the drying times obtained on

three coatings and compare them to drying times obtained with D 1640.

D01.23.20—Exploratory Research— P.R. Guevin, Jr., Chairman. This task group was set up to make members aware of new instrumentations and technologies that are being developed to determine the physical properties of applied films. Task group members will present and report relevant literature and articles.

At this meeting, the chairman reported on initial results on the determination of film thicknesses of coatings applied to concrete using ultrasonics. The use of X-ray fluorescence spectrometry to measure paint thickness on coil coatings was also discussed.

The chairman reported on tests run by Fischer Technologies using their dynamic hardness tester at the 1992 Paint Industries' Show. A. Rutkiewic indicated how useful it appears based on work at Du Pont and its ability to evaluate organic coatings. R. Bearhalter, a representative for a similar instrument from Shimadzu, offered to be a participant in the study. Samples of coatings on different substrates and with varying morphology will be evaluated. The chairman will report the results of these findings at the next meeting.

SUBCOMMITTEE D01.24 PHYSICAL PROPERTIES OF LIQUID PAINTS

C.K. Schoff, Chairman

D01.24.18—Dispersion Phenomena— R.K. Morrison, Chairman, reported on the revision of D 1210, "Fineness of Dispersion of Pigment-Vehicle Systems." A new step gauge (developed by SCM) for measuring cleanliness or texture will be included in the revised method. Provided proper arrangements can be made, round-robin tests of the step gauge will be conducted at the June 1993 D-1 meeting. The chairman will prepare a draft of the revised method for discussion at the meeting.

*D01.24.33—Odor Evaluation—*D.E. Darr, Chairman, discussed the preparation of a new guide titled, "A Guide to the Detection, Identification and Characterization of the Odors of Paints, Inks and Related Materials." The proposed scope for the guide will be publicized in both ASTM and trade publications soliciting input to determine what already is available and who is actively working on odor evaluation. A.F. Rutkiewic volunteered to search ASTM literature for applicable methods; the chairman will conduct a literature search of non-ASTM sources. The gathered information will be used at the June meeting to begin writing the guide.

SUBCOMMITTEE D01.26 OPTICAL PROPERTIES

R. Kumar, Chairman

*D01.26.02—Color Measurement—*R.T. Marcus, Chairman, reported that the results of the D-1 ballot on the revision of D 1544, "Test for Color of Transparent Liquids (Gardner Color Scale)," were discussed. The method will be revised by J. Peters and submitted for concurrent D01.26/D-1 ballot.

The results of the D01.26 ballot on a new "Standard Guide for the Preparation, Maintenance and Distribution of Physical Product Standards for Color and Geometric Appearance of Coatings," were discussed. There was one negative from J. Cave and several comments. The guide will be revised accordingly and submitted for concurrent D01.26/D-1 ballot.

The present edition of D 2244, "Standard Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates," contains an error in the CIELAB delta E equation. The square root (1/2 power) function is missing. C. Sherman will make the correction and submit it for concurrent D01.26/D-1 ballot.

*D01.26.06—Hiding Power—*L.E. Schaeffer, Chairman, reported on the results of the latest round-robin run by D01.51.02, "Hiding Power of Powder Coatings." This is the same round-robin for which partial results were discussed at the last meeting. At this time, it is considered complete with data from three cooperators. The results were calculated so as to present hiding as the film thickness at the contrast ratio of 0.98. Two values were obtained for each test application, one based on film weight converted to thickness by calculation and the other based on direct film thickness measurements. The weight derived values showed much better reproducibility. From these and the results of two previously reported round-robins, a weight basis method could be balloted. However, the consensus of the D01.51.02 was to continue its efforts based on direct film thickness measurement since this is a familiar and accepted procedure in powder coating technology. Task group D01.26.06 will continue to cooperate with D01.51.02 in these efforts.

D01.26.11—Gloss and Goniophotometry— A.F. Rutkiewic, Chairman, reported on the D01.26 ballot action on Draft #6 on "New Test Method for Instrumental Measurement of Distinctive of Image Gloss of Coating Surfaces." A negative was cast because Method B was removed in the latest draft. Method B employs the use of the Suga instrument and was removed because there was no description or reference of the instrument in any other ASTM method. However, it is used in an ISO method. It was the consensus of the group that this is valid and Method B will be reinstated. The revised method will be reballoted by D01.26.

*D01.26.24—Tinting Strength—*R. Morrison, Chairman, reported that two methods, D 387, "Test Method for Color and Strength of Color Pigments with a Mechanical Muller," and a new method, "Standard Test Method for Evaluating the Tint Undertone of Titanium Dioxide Pigments," were approved for concurrent D01.26/D-1 ballot.

The latest ballot of D 2745, "Standard Test Method for Relative Tinting Strength of White Pigments by Reflectance Measurements," to correct an error in the tinting strength equation, received a negative. The negative was withdrawn when the equation was explained and a comment was added to clarify it. The method now goes to society ballot.

As a future project for this task group, it was decided to investigate the development of a waterborne tinting strength method. Committee D-1 will be surveyed to determine if there is a need for such a method, and suggestions if indeed there is.

*D01.26—*R. Marcus, Vice Chairman of D01.26 and Chairman D01.26.02 on Color Measurement, has resigned because of changed responsibilities. F. O'Donnell has agreed to be Chairman of D01.26.02. Chairman Romesh will appoint a vice chairman by the next meeting.

SUBCOMMITTEE D01.27 ACCELERATED TESTS FOR PROTECTIVE COATINGS

D. Grossman, Chairman

*D01.27.02—Water Tests—*D. Grossman, Chairman. A D01.27 ballot will be made on a new Method B for D 2247, "100% Humidity." The practice now specifies that cabinets shall generate humidity by heating a bath of water in the chamber bottom. However, most of the cabinets being used for this type of humidity test generate humidity by bubbling compressed air through an unheated water bath, while maintaining chamber temperature by a heated water jacket around the walls. This design often fails to produce adequate condensation on the specimens, unless the water jacket is drained to below the level of the specimens. F. Lutze will draft a revision specifying the proper operation of this alternate design.

*D01.27.04—Light and Water Exposure Apparatus—*L.E. Thieben, Chairman, reported that D 3361, "Unfiltered Carbon Arc," received a negative on the D-1 ballot. Minor changes will be made and the revision will be sent to concurrent D01.27/D-1 ballot. A draft for the new standard on Xenon Arc received two negatives and several com-

ments at D01.27 ballot. J. Robbins will revise the draft for another D01.27 ballot.

D01.27.10—Accelerated Outdoor Weathering—J. Robbins, III, Chairman, discussed the D01.27 ballot of D 4141, "Standard Practice for Conducting Accelerated Outdoor Exposure Tests of Coatings." A decision was made to submit this practice to concurrent D01.27/D-1 ballot with a modification of Section 7.4.2.1. The frequency of calibration proposed for ultraviolet radiometers will be changed from every six months to every year.

D01.27.12—Detergent—F. Lutze, Chairman, reviewed the results of the D01.27 ballot on D 2248, "Detergent Resistance of Organic Finishes." Several editorial and technical comments were received and considered. Since no negative votes were cast, this will be sent to society ballot.

D01.27.17—Evaluation of Weathering Effects—J. Martin, Chairman, reported that six items were sent to D-1 ballot in November 1992. D 660, "Evaluation of Checking," and D 661, "Evaluating of Cracking," will be withdrawn from further balloting based upon a suggestion by M. Crewdson to combine the two. M. Crewdson will write the draft for the combined D 660. Method D 661 will be balloted for withdrawal as soon as the new combined standard is approved.

D 662, "Evaluation of Erosion," received no negatives and will be sent to society ballot.

D 772, "Evaluation of Flaking," will be reballoted in its entirety. The sections that were revised have been balloted. The main body of the document was never balloted.

D 1006, "Conducting Outdoor Weathering Tests on Wood Coatings," received one negative from R. Carlson, saying that reporting UV exposure should not be mandatory. Since the draft does not in fact make it mandatory, D01.27 voted the negative nonpersuasive, and with the concurrence of D-1, will proceed to society ballot.

The round-robin conducted for flaking and blistering last year will be incorporated into the methods. Another mini round-robin is tentatively scheduled for January 1994 for other test methods. J. Martin, M. Crewsdon, and D. Dietz will look for samples to use in this mini round-robin.

D01.27.28—Filiform Corrosion—F. Lutze, Chairman, reviewed the results of the D01.27 ballot on D 2803, "Filiform Corrosion Resistance of Organic Coatings on Metal." Several editorial and technical comments were reviewed and considered. The negative vote concerning a precision and bias statement for a guide was withdrawn. This document will be sent for society ballot.

D01.27.29—Test Substrates—D. Grossman, Chairman. F. Lutze noticed that in D 2201, "Zinc Coated Steel Panels for Paint Tests," Appendix X1, Characterization of Zinc Coating, many of the newer metric designations used in the auto industry are missing. He will draft a revision for concurrent D01.27/D-1 ballot.

D01.27.30—Corrosion Tests—Automotive—F. Lutze, Chairman. A revised draft of the "Standard Practice for Accelerated Outdoor Cosmetic Corrosion Exposure Testing of Organic Coatings on Metal," was reviewed and discussed. This draft will be submitted for D01.27 ballot.

A draft of the "Standard Practice for Cyclic Corrosion Testing of Painted Steel Parts" was reviewed. This document provides a listing of seven cyclic corrosion exposures used in various industries and is intended as a guide to aid the user in selection and performance of cyclic corrosion testing. This will be sent for D01.27 ballot.

A draft of a practice utilizing outdoor exposure on a 45° open rack with a twice weekly salt solution application was reviewed. The draft will be revised to reflect the results of the discussion and submitted for D01.27 ballot.

D01.27.31—Corrosion Tests—Non-Automotive—S. Boocock, Chairman, led a discussion of a round-robin to compare outdoor exposure with various cyclic corrosion tests. A proposal had previously been submitted to the Institute for Standards Research (ISR) which would result in underwriting certain fixed costs associated with the round-robin testing program. The task group questioned the time being taken to activate the round-robin testing program through the ISR. Furthermore, they questioned the value, both technical and financial, derived from continuing the proposal development. The task group voted that the round-robin program should be initiated immediately as a direct funding of D01.27.31 to save time and money. Funding required for activities related to the round-robin would be identified and reviewed. Exterior exposure sites will be finalized before the June meeting and samples preparation could begin immediately.

Two new draft standards for cyclic corrosion tests were reviewed: 1) cyclic salt fog alternating with UV/condensation, and 2) cyclic immersion/UV exposure. Both drafts will be sent to D01.27 ballot.

A mini round-robin intended to identify an ideal drying temperature for use with the cyclic salt fog component of the draft cyclic salt fog/UV/condensation standard was arranged. W. van der Linde will be distributing sample panels to participating volunteers.

SUBCOMMITTEE D01.28 BIODETERIORATION

M.C. McLaurin, Chairman

D01.28.01—Package Stability—M.C. McLaurin, Chairman. S. Orthey again prompted us to start a ballot action on the revised method, D 2574. The revised draft incorporating our various changes will be prepared by J. Hinkle and M. McLaurin, and circulated for D01.28 balloting February 5.

D01.28.02—Rapid Determination of Enzymes—G. Moore, Acting Chairman for C.W. Vanderslice. A very rough version of the drafted method was circulated for review. This will be put into final ASTM format and the round-robin agreed to last June will be conducted.

D01.28.03—Microbial Quality of Raw Materials—J. Hinkle, Chairman, circulated a final draft of the method prior to the meeting which incorporated changes proposed after June's meeting. A revision of the rating scale was agreed upon to more clearly define the differences between 1-4 ratings. After this section is revised, the method will be balloted with D01.28 in February.

D01.28.04—Resistance of Paint Films to Algae Attack—J. Hinkle, Chairman, circulated a proposed draft prior to the meeting which was discussed. Several minor changes were suggested, along with a consistent rating scale (0-4) based on other microbiological methods. This scale is consistent with D 2574, European Methods, and long-standing methods in the industry. These changes will be incorporated and the method used for round-robin testing. The choice of algal species to be used has not been finalized.

D01.28.05—Revision of D 3273 (Mildew Chamber)—J. Hinkle, Chairman. This new task group met and had a good start. Elimination of the circulating fan was discussed. B. Matta confirmed that, in his testing, elimination of the fan did not hurt, and in fact increased reproducibility of the samples. J. Hinkle and M. McLaurin indicated similar findings. The fan will be eliminated from the proposed revision. The chairman reported updated components for the chamber which will also be incorporated.

A revision to include the test's applicability for estimating relative performance in exterior coatings will be done separately since several complications may arise. It was generally agreed that even though survival of the exterior fungi *Aureobasidium pullulans* is minimal, this method gives comparable results to exterior exposure as long as: 1) the samples are subjected to prior "weathering" simulations such as leaching or accelerated weathering machine exposure; and 2) testing is for standard, pigmented film-forming coatings. M. McLaurin reported distinct changes with clear coatings and non-film forming stains. It was suggested that these be mentioned as exterior applications for which the method may not be representative. These two revisions will be drafted separately and balloted in D01.28.

D01.28.06—*Resistance of Paint Films to Fungal Attack*—J. Hinkle, Chairman, circulated a proposed draft prior to the meeting based on the algal method (D01.28.04). This method was discussed and appeared to have few problems (the rating system was made consistent to be 0-4). This method will be tested in a round-robin simultaneously with the algal method.

D01.28—*Biodeterioration*—The minutes from June's meeting were approved without comments or changes. It was noted that the meeting attendance was very good and contributed to valuable discussions in the various sessions.

B. Matta brought the original photographs used in D 3274 for rating fungal growth on test fence panels and discussed the concerns he and M. Crewdson have had. It was agreed there are problems with this rating scale and that most companies are not using it as a result. B. Matta will coordinate some proposed revisions and present them at the next meeting.

It was discovered that the changes to D 4610 proposed to address the negative in June of 1991 were never balloted. M. McLaurin thought he had previously done this and will check his records. Rough text was verbally agreed upon at the meeting and will be included in a revision for the D01.28 ballot to address the negative.

DIVISION 30
PAINT MATERIALS

SUBCOMMITTEE D01.31
PIGMENT SPECIFICATIONS

D.E. Kesatie, Chairman

D01.31.08—*Titanium Dioxide*—C.C. Tatman, Chairman, reviewed the current standard D 476, "Standard Specification for Titanium Dioxide Pigments," and compared it to the compositional requirements of BSI 851 and ISO 591. In preparation for the June 1993 meeting, the task group will poll the North American producers to determine: 1) whether the standard represents the current grades; 2) if the types should be expanded to include the "ultra-fine" grades and the new "multi-purpose" grades; and 3) should D 476 be aligned closer to ISO 591.

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

D01.31.15—*Micaceous Iron Oxide*—M. Hendry, Chairman, will submit a revised

method for a D01.31 ballot. If approved, it will be submitted for a D-1 ballot prior to the June 1993 meeting. It was decided that the color pictorial standards should be supplied by ASTM rather than the micaceous iron oxide suppliers. This will require an adjunct procedure by ASTM.

D01.31—Fourteen standards were placed on the D-1 ballot for reapproval or withdrawal. Nine standards were approved. Negative votes were received for D 867 "Pumice"; D 912, "Cuprous Oxide"; D 963, "Copper Phthalocyanine Blue"; and D 3021, "Phthalocyanine Green," when balloted for withdrawal. The negative votes were found to be persuasive. The four preceding methods will be placed on the D-1 ballot for reapproval.

Negative votes were also received on the reapproval of D 962, "Aluminum Powder and Paste Pigments for Paints." A minor revision was requested adding a reference document. The negative voters are being asked to withdraw their votes so that the method can be published "as is." Then a revised method will be written that will incorporate their request for the June 1993 meeting and submitted for a D01.31/D-1 ballot prior to the January 1994 meeting.

The method, D 1649, "Strontium Chromate," was discussed. The revised method will be submitted for a D01.31/D-1 ballot.

The method, D 212, "Chrome Green," will be reviewed by M. Denesha and will be discussed at the next meeting.

In new business, five standards are to be reviewed in 1993. The methods are: D 480, "Flaked Aluminum Powder," D 763, "Raw and Burnt Umber Pigments," D 768, "Yellow Iron Oxide," D 911, "Mercuric Oxide for Use in Antifouling Paints," and D 3724, "Brown Iron Oxide." All but D 911 will be placed on the D-1 ballot for reapproval with no revisions. D 911, "Mercuric Oxide for Use in Antifouling Paints," will be placed on the D-1 ballot for withdrawal.

SUBCOMMITTEE D01.33
POLYMERS AND RESINS

M.J. Mahon, Chairman

D01.33—M.J. Mahon, Chairman, reported results of the D-1 ballot. Most of the negatives and comments received concerning these ballot items were able to be addressed through editorial revisions. These changes were considered, approved, and the document sent for society ballot. D 1615, "Test Method for Glycerol, Ethylene Glycol, and Pentaerythritol in Alkyd Resins," and D 1641, "Test Method for Exterior Durability of Varnishes," were withdrawn from further balloting based on the negatives received. It was recommended that these meth-

ods and D 1642, "Test Methods for Elasticity or Toughness of Varnishes," be transferred to D01.42. Due to the absence of task group chairmen, or reports of task group activities, no further discussions of the urethane prepolymer isocyanate content or the D 563 round-robin were held.

SUBCOMMITTEE D01.34
NAVAL STORES

J. Russell, Chairman

D01.34.01—*Capillary Gas Chromatography of Rosin and Fatty Acids*—W. Trainor, Chairman, reviewed the status of the development of this new standard method. He stated that a draft of the method had been circulated prior to the meeting and that experimental work had been carried out by committee members to compare the use of diazomethane, tetramethylammonium hydroxide (TMAH), and trimethyl phenyl ammonium hydroxide (TMPAH) as reagents for converting rosin and fatty acids to their more volatile methyl esters. After considerable discussion, it was agreed that the latest draft should be reorganized to make it easier to follow. J. Bowers and J. Russell will work with the chairman on this reorganization and the new draft submitted to the D01.34 members prior to the next meeting.

The experimental work reviewed by the committee consisted of capillary GC analyses of a sample of distilled tall oil. Diazamethane, TMAH, and TMPAH were compared and the results obtained and confirmed that diazomethane and TMPAH gave identical results even when excess TMPAH was used. Excess TMAH however, was found to cause isomerization of the conjugated fatty acids, so extreme care is required when this reagent is used for fatty acid analysis.

D01.34.03—*Softening Point of Rosin and Rosin Derivatives by the Automatic Mettler Apparatus*—P. Zawislak, Chairman, reported on the results of the experimental work carried out since the last meeting to establish which heating rate on the Mettler apparatus would give the same softening point as the manual Ring and Ball Method (E 28). The desired heating rate appeared to be between 1.4 and 1.6°C/min. However, it was decided that the ball used in these trials, i.e., the standard ball used in the Ring & Ball determination, was slightly too large for the cup used in the Mettler equipment and so the results obtained were suspect.

It was agreed that W. Trainor would identify a source of stainless steel balls of the correct size and a round-robin will be carried out using three materials of differing softening points at heating rates of 1.4, 1.5, and 1.6°C/min. P. Zawislak will ask

Mettler to supply "loaner" instruments to laboratories that wish to participate in the round-robin but do not have the Mettler cup and ball apparatus.

D01.34.04—Iodine Value Using Solvents other than Carbon Tetrachloride—J. Bowers, Chairman, reported that all experimental work to-date indicated that iso-octane appeared to be a promising replacement for carbon tetrachloride in the determination of iodine value by Wijs solution and that a round-robin should be carried out either alone or jointly with D01.32 Drying Oils.

J. Russell, reported that D01.32, the subcommittee currently having responsibility for iodine value determination (standard D 1959), was essentially an inactive subcommittee and the changes of a joint round-robin were not good. It was agreed that D01.34 would offer to take over responsibility for D 1959 from D01.32. If that approach is not successful, D01.34 will prepare its own standard and carry out a round-robin on the new method. Interested parties would be invited to participate in this round-robin.

D 465—Acid Number—W. Trainor, Chairman, reported on the final results of the round-robin held on tall-oil fatty acids, rosin, and distilled tall oil. It was agreed by the subcommittee that the results obtained in the round-robin reflected the precision to be expected from this standard method and that a research report should be prepared. Further, a precision statement should be written for inclusion in the standard method and the method submitted for appropriate balloting.

D 464—Saponification Number—S. Whitfield, Chairman, reported on the final results of the round-robin on tall-oil fatty acids, rosin, and distilled tall oil. The precision obtained with this complicated method was disappointing, especially the reproducibility between laboratories. It was suggested by R. Humphrey that the precision could be improved if a small amount of water is present during the final titration. Consequently, it was agreed that another round-robin, this time with the added water, be run on tall-oil fatty acid. The results of this round-robin will be discussed at the next subcommittee meeting.

D 1065—Unsaponifiable Matter—J. Bowers, Chairman, reported on the final results of the round-robin analysis carried out on tall-oil fatty acid, rosin, and distilled tall oil. He concluded that the repeatability within a laboratory was at the expected level, but the reproducibility between laboratories was poor. Apparently, there are enough opportunities for individual variations in technique in this rather complex method that laboratory to laboratory differences are inevitable. It was agreed by the subcommittee that this method does not have the precision required for setting product specifications.

It was also agreed that J. Bowers would prepare a research report and also a precision statement for inclusion in the standard method.

D 1240—Rosin Acid Content and D 1585, Fatty Acid Content—W. Mark, Chairman, was unable to attend, and in his absence J. Bowers reported on the results of the round-robins carried out on these two inter-related methods. Round-robins to measure rosin acid content had been run on tall-oil fatty acids, distilled tall oil, and rosin and in every case the repeatability within a laboratory had the precision expected. With tall-oil fatty acid and distilled tall oil, the reproducibility of the rosin, acid determination was also satisfactory. However, with rosin, the reproducibility of the rosin acid content was poor. It would appear that the method, as written, permitted too many variations and this affected the precision of the results. Similar conclusions were reached with the fatty acid content determination as this method is essentially a mathematical calculation based on D 1240, "Rosin Acid Content" and D 1065, "Unsaponifiable Matter." It was agreed that these findings needed further discussion before a research report could be written. Specifically, some of the options existing in the current method need to be eliminated.

E 28—Ring and Ball Softening Point—P. Zawislak, Chairman, reported that the experimental results obtained comparing the automated Herzog Ring and Ball method to the manual Ring and Ball method showed that the resultant softening point was always higher with the Herzog method. It was agreed that if the automated method was to be included in standard E 28, then some changes would be required in the existing equipment. It was agreed that the German manufacturer be contacted and informed of our findings. Herzog would be invited to participate in future round-robins and amend his equipment so that the results were the same as those found with the manual method. Another area of discussion on this standard was the use of an alternative heating medium especially for high-melting resins. Currently, the recommended heating medium is glycerol, but at high temperatures it darkens and decomposes. It was agreed that interested members of the committee would explore the use of Dow Corning 200, a silicone based fluid, and report back to the committee at the next meeting.

D01.34—J. Russell, reported that nine of the subcommittee's standard methods will be on the March 1993 society ballot. Assuming no negative votes, this will mean that all 22 of the subcommittee standards are now up-to-date. It was also agreed that D01.34 would not meet in Philadelphia in June, but hold task group meetings in Savannah, GA, in May and Atlanta, GA, in the fall.

SUBCOMMITTEE D01.35 SOLVENTS, PLASTICIZERS AND CHEMICAL INTERMEDIATES

R.G. Montemayor, Chairman

D01.35.10—Hydrocarbon Solvents and Ketones—S.A. Yuhas and R.L. Hinrichs, Co-Chairmen. A new method, D XXXX, "Boiling Paint Distribution of Hydrocarbon Solvents by GC," has passed the D-1 ballot and is now ready for society ballot.

D 0235, "Specification for Mineral Spirits," is being revised to expand the table of types of mineral spirits to include 10 types. This standard drew a negative from J. Lebeau. He requested that the minimum flash point of Type II Mineral Spirits (high flash point) be raised from 140°F to 142°F in view of the forthcoming DOT reclassification of "flammable liquids" to include those having flash points up to and including 141°F. In conjunction with this, the suggestion was made to raise the 50% distillation temperature from 385°F to 390°F max. The negative was ruled persuasive. J. Lebeau also cast a negative on the basis of the odor requirement in the specification and requested that it be deleted on the grounds that it might pose a health hazard to the person performing the test. After much discussion, Mr. Lebeau withdrew his negative if the odor specification was footnoted as follows:

"Optional: Test for odor only when agreed as necessary by supplier and customer."

The changes will be made and D 0235 will be reballoted at D01.35. There was considerable discussion on the potential health hazard associated with performing the odor test by D 1296 which is part of many ASTM solvent specifications. Many solvent suppliers are deleting the odor test requirements from their sales specs. D01.22 on Health and Safety will be consulted for advice.

A round-robin for a capillary GC method to be added to D 3257, "Aromatics in Mineral Spirits by GC," has been initiated and being coordinated by S. Kail. Five samples and potentially six participating laboratories have been identified.

D01.35.20—Reactive Monomers—J.E. Frugé, Chairman. There were a number of negatives received at the D-1 ballot for several standards under the responsibility of this task group relating to the bias statement on the standards. P. Guevin cast negatives saying that the bias statement on a number of standards did not confirm to the August 1992 *Form and Style Handbook*. E. Gutman (ASTM Staff) and P. Guevin were present during the discussion on the bias statement. Initially, E. Gutman agreed that the advice given to D01.35 by ASTM staff several years ago was correct, i.e., that a simple bias statement such as "Bias has not been deter-

mined for this method," would be adequate, as long as the subcommittee considered and addressed the matter. However, in the new Blue Book revision, further explanation and action will be necessary as standards come up again for reapproval. P. Guevin withdrew his negatives contingent on D01.35 doing something to provide more definitive bias statement in the future.

D 3362, "Purity of Acrylate Esters by GC," received a negative from R.P. Doshi. Mr. Doshi wanted a capillary column to replace the packed column required by the standard. The negative was found to be nonpersuasive since changing to capillary column will be a different method than the existing method (9-0-0). The proposed new procedure was forwarded to D01.35.60 (Method Development Task Group) for consideration.

D01.35.30—Chemical Intermediates— J. Morrison, Chairman. A number of standards under the responsibility of this task group did not make it into the recent D-1 ballot. They will be balloted in the March 1993 D-1 ballot.

A GC method for the determination of "Acetone in Diacetone" is being reviewed for evaluation.

*D01.35.40—Plasticizers and Ester Solvents—*J.E. Lawniczak, Chairman. A number of negatives were received on the withdrawal of standards relating to tricresyl phosphate (TCP) and methyl amyl acetate (MAA). The Department of Defence have the TCP Standards as part of their specs and S. Yuhas pointed out current producers and suppliers of TCP and MAA. These negatives were found to be persuasive. The standards were withdrawn from D-1 ballot and will be reballoted for reapproval. In the meantime, the Department of Defence will find out if it will still have TCP as one of their requirements, and the producers and suppliers of TCP will be contacted by the chairman of the task group as to their requirement of keeping the standards on TCP current.

*D01.35.50—Coordination—*L. Forrest, Chairman. A number of standards from D-2, D-16, and E-15 were discussed. Among these is the round-robin to determine improved precision on D 86 using a wider neck flask and a thermometer centering device. There is also a round-robin being planned by D-2 on the effect of viscosity of liquids on the flash points when determined by D-93, Procedure A or Procedure B. This round-robin activity was relayed to D01.22.01. A request was made to indicate any interest of participation by organizations which are D-1 members to R.G. Montemayor. S. Yuhas and R. Hinrichs reported that they noticed errors in the methods calculation in D 1555, "Calculation of Volume and Weight of Industrial Aromatic Hydrocarbons." These have been commu-

nicated to ASTM headquarters for referral to the appropriate subcommittee.

*D01.35.60—Method Development—*T. Nemchock, Chairman. R.G. Montemayor presided during the meeting due to the absence of the chairman. There are three new methods under development by the task group: 1) Acetone in Diacetone Alcohol; 2) Capillary GC Method for Acrylate Esters; and 3) Capillary GC Method for Total Aromatics in Mineral Spirits.

SUBCOMMITTEE D01.36 CELLULOSE AND CELLULOSE DERIVATIVES

G.Y. Moore, III, Chairman

*D01.36—*After five years as chairman of this committee, D. Martin resigned, but will continue to be a member of the committee. G. Moore is now Chairman, and J. de Wit is Secretary.

Two methods were submitted for main committee ballot and received no negatives, "New Standard Test Method for Hydroxypropylcellulose," and D 1343, "Standard Test Method for Viscosity of Cellulose Derivatives by the Ball Drop Method." D 4085, "Standard Test Method for Metals in Cellulose by Atomic Absorption Spectrophotometry," was updated with keywords only, and received one negative concerning the complexity of the precision statement. The negative was found persuasive. G. Moore will edit the precision statement to conform with the standard format, and will place the information in the current statement in a footnote. The method will be resubmitted for concurrent D01.361 ballot.

In new business, G. Shore, of Chemical Coatings Inc., IRPM, a visitor at this meeting, has indicated interest in becoming a member. He also indicated a need for ASTM methods for high-solids and water-based coatings.

D01.36 will continue recruitment efforts. A publicity request form has been obtained from D-1 to publicize the work this small but active committee is doing.

SUBCOMMITTEE D01.37 INK VEHICLES

A.N. Scarlatti, Chairman

*D01.37.01—Resin Solutions—*J.T. Daust, Chairman, reported that the proposed method for "The Preparation of Resin Solutions Using a Stirrer Hot Plate," received two negative votes in D-1 ballot. The method will be revised and resubmitted to D01.37 ballot. Another proposed method for "The Preparation of Resin Solutions Using a War-

ing Blender," will be edited and discussed at the June meeting. A. Scarlatti will write proposed test methods for "The Preparation of Resin Solutions Using a Hot Air Gun," and "The Preparation of Resin Solutions Using a Hot Oil Bath," for review at the June 1993 meeting.

*D01.37.03—Gelability of Resin Solutions—*A.N. Scarlatti, Chairman, reported that the proposed practice D 5165, "The Laboratory Preparation of Gelled Vehicles Using a Resin Kettle," has passed D-1 ballot and will be submitted for society ballot this spring. H. Brooks will provide a proposed method for "The Evaluation of Gelled Vehicles," for D01.37 in June. J. Daust and H. Brooks will review European data and methods for evaluating vehicle rheology using oscillatory cone and phase rheometers.

*D01.37.04—Standard Ink Oil—*J. T. Daust, Chairman, reported that attempts to formulate a standard ink oil from chemically pure components, to be used for resin Q.C., has been unsuccessful. Before the end of February 1993, both Pennzoil (Magie) and Exxon have agreed to submit proposals for offering a quantity of aliphatic oil in the boiling range of 465-530°F, which will be held for distribution as the standard oil for resin Q.C. The quantity of oil, the storage conditions, method of marketing, and provisions to replace the standard with like oil when the initial inventory is depleted will be outlined. The task group is looking for representation from vegetable oil suppliers so that efforts can be made to provide standards for common vegetable oils (i.e., linseed, soybean).

*D01.37.05—Quality Control of Ink Oils—*J.B. Sardisco, Chairman, reported on the results of the round-robin on the proposed method for "The Quality Control of Ink Oils." The ballot of this method received three negatives. The method will be revised so that users can easily distinguish between hydrotreated and non-hydrotreated oils. The method will also state that by using this method and viscosity, various grades of oils can be distinguished. The method will be reballoted after revision and approval by D01.37.05.

*D01.37.06—Pigment Dispersibility of Paste Ink Vehicles—*M.E. Fuchs, Chairman, reported that use of the Hoover Muller in reverse for analyzing pigment dispersibility was found ineffective. A literature search will be conducted to find methods that might be used to measure the pigment dispersion characteristics of vehicles. D01.37.06 will also investigate a quick mill method for vehicle and presscake dispersion.

*D01.37.07—Resin/Solvent Compatibility—*D.P. Frisch, Chairman, accepted the chairmanship of D01.37.07 after the resignation of T. Sayer. A.N. Scarlatti will write a proposed method for "Determining the

Compatibility of Resin in a Given Ink Solvent by Precipitation Temperature" and distribute for D01.37.07 review before the June 1993 meeting.

D01.37.08—New Membership—D.P. Frisch, Chairman, reported that three D-1 members, H. Brooks, A. Mercado, and G. Wouch have requested membership in D01.37. For business reasons, J.H. Thomas and T.J. Sayers have resigned from D01.37. D01.37 is planning a membership drive to recruit new members. Anyone associated with printing ink vehicle formulation, manufacture, or use is welcome to join.

D01.37—Ink Vehicles—A.N. Scarlatti, Chairman, accepted the chairmanship of D01.37 after the resignation of J. Thomas. Aside from the development of new ink vehicle test methods, a primary agenda item for D01.37 will be to increase membership with industry specialists. A.N. Scarlatti has asked current D01.37 members to assist in recruiting additional members for active ASTM participation.

DIVISION 40
PAINT PRODUCTS
APPLIED ON SITE

SUBCOMMITTEE D01.42
ARCHITECTURAL FINISHES

L. Schaeffer, Chairman

D01.42.03—Porosity of Paint Films—C. Tatman, Chairman, reviewed the task group objective, which is to develop a quantitative method for measuring the porosity of a paint film of any color or brightness level. (Present method D 3258 works only with light colors.) Data obtained thus far indicates that round-robin activity should be limited to testing latex paints formulated above the CPVC, using low viscosity mineral oil as the saturant. Based on these decisions and on group discussion, a new round-robin will be conducted by five cooperators using three latex paints formulated above the CPVC. TiO$_2$ and extender variations will be used to produce test paints with different levels of porosity. The use of a fluorescent dye retention technique will first be investigated, as possible alternative to the weighing of absorbed mineral oil. This technique was not tried previously and warrants consideration before the task group proceeds to the new round-robin.

D01.42.04—Wet Adhesion of Latex Paints—C.W. Vanderslice, Chairman, was unable to attend and the meeting was chaired by A.J. Desmarais. The original test method was discussed, which employs as its test surface, an air-dried gloss alkyd enamel applied on wood panels. A strip of cheese cloth is embedded in the wet test paint application, and after drying and subsequent soaking, the cheesecloth is pulled off with weights to determine the force required to peel the test paint from the alkyd enamel substrate. This method is now considered by the group as too tedious, and it is not known to be widely used. A new and simpler method was proposed involving the use of a scrub test machine, with the alkyd enamel applied on a plastic panel instead of wood, and the test film scored prior to scrubbing. Data was presented showing this test to give essentially the same adhesion differentiation as measured with the cheesecloth technique. Several suggestions for further improvement of the new method were discussed. Polyester panels were suggested to provide a more definitive test substrate, in view of the variability of alkyd enamel films due to aging and formulation. The chemical similarity of polyesters and alkyds was pointed out. Another suggestion was to score the film in the form of a grid pattern, instead of just parallel lines. Scrub media type and soak time were also considered to be critical parameters. Samples of polyester panels will be obtained, and these will be evaluated in addition to the other suggested variables prior to the June meeting.

D01.42.09—Color Development in Tinted Paints—L. Schaeffer, Chairman Pro-Tem, reported that the proposed test method, "Color Development in Tinted Latex Paints," had passed society ballot and would be published in the *1993 ASTM Book of Standards* as D 5326. Technical comments from H. Ashton and D. Rich that had not been previously addressed were discussed briefly. These are to be addressed in a revision of D 5326 that will be submitted to concurrent ballot during 1993. The next step for this task group is to develop specific methods for evaluating (1) colorant dispersability in tint bases, and (2) tint base receptivity for colorants. Although the basic experimental procedure, namely the test method of D 5326, is the same for both objectives, it was tentatively agreed that the two need to be studied separately so as to avoid the need for excessively large round-robin testing programs.

D01.42.20—Water Repellency of Wood—V. Scarborough, Chairman, reported that the D-1 ballot of the proposed test method received two negatives and several editorial comments. One negative questioned how the wood was positioned during immersion, and in response the word "fully" was inserted in the appropriate location. This negative was withdrawn. The second negative was also withdrawn after an explanation was given as to the location of all referenced documents in the method. The method will next move to society ballot.

D01.42.21—Project Planning—S. LeSota, Chairman, led the discussion on possible new ASTM test methods for architectural finishes, to be worked on by D01.42. A. Lethers suggested that a new task group be formed to evaluate and measure exterior stain properties, such as appearance, mode of deterioration, lap sheen uniformity, grain raising, and UV penetration. It was accordingly agreed to form Task Group D01.42.29, Guides and Tests for Exterior Stains, with A. Leathers as Chairman. Anyone else perceiving an unmet need for an ASTM standard test method relative to architectural finishes, should contact the task group or subcommittee chairman.

D01.42.22—Guide for Testing Architectural Coatings—H. Ashton, Chairman, reported that the Guide to Testing Waterborne Architectural Coatings which was on D-1 ballot last spring and discussed at the June meeting, had subsequently passed society ballot as ASTM D 5324. His review of the limited staff revision and the printer's proof copy had eliminated several errors, so that the document in the *1993 ASTM Book of Standards* should be in very good shape. In addition, the chairman had written to two voters explaining the disposition of their comments. It was agreed that the four standards covered by D 5324 would be balloted for withdrawal in due course. However, since two of them have just been revised and published in the *1992 ASTM Book of Standards,* it was further agreed to initially submit only the nowdue guide, D 3358-88.

The chairman next reported that editorial revisions had been submitted for the companion solvent-borne guide D 5146-90, which therefore now becomes D 5146-90 ε1. As agreed at the last meeting, the four standards incorporated in D 5146 will also be balloted for withdrawal, and a D01.42 ballot for that purpose will go forward shortly.

With regard to technical revisions to D 5146 and D 5324 mentioned at the past two meetings, the chairman said he would work on these shortly. He noted that of the many good comments from B. Schiller, one of them related to D 3928, "Test for Uniformity of Gloss or Sheen," was not in the jurisdiction of this task group and should be redirected to the subcommittee chairman.

Finally, the results of the D01.42 ballot on revising the related guide, D 3730-78 (1988) "Testing of High-Performance Interior Architectural Wall Coatings," was announced as 18 affirmative, 18 abstaining, and 0 negative. Since no one present felt qualified to comment on the present maximum free diisocyanate content of 0.5%, the chairman said that if he could not obtain sufficient information on this subject in time to meet the D-1 ballot deadline, he would send forward only the three revisions that were needed to make D 3730 agree with D 5146-93 and D 5324-93.

D01.42.24—Application Properties of Paints—J. Price, Chairman, reported that there had been no comment from the task group or subcommittee on the data from the second round-robin attempting to measure the "natural spreading rate" of paints. ICI high shear viscometer values of the test paints were reported by one cooperator. The chairman will evaluate his viscosity data for possible correlation with the round-robin brushout results, and will present an analysis at the next meeting. Based on that analysis, a decision will be made as to the possibility of developing a reproducible test method for "natural spreading rate."

D01.42.25—Scrub Resistance of Architectural Coatings—D. Licata, Chairman, discussed conclusions reached at the last meeting. It had been agreed that sufficient data was available to warrant rewriting D 2486 to include the use of calibration panels for normalizing cycles-to-failure values. An initial proposed revision for that purpose was passed around. Note was taken of the extremely wide cycle-to-failure variations between laboratories in the most recent round-robin. This occurred despite the fact that all of the cooperating laboratories used basically the same type of scrub tester. Possible variables that might have caused these large differences were considered, with the harshness of the scrub brush being particularly suspect. The chairman proposed that each cooperating laboratory should adjust its scrub test machine to a standardized level of abrasiveness as a regular part of the test procedure. After much discussion, it was agreed that an additional round-robin should be conducted which would include that feature. In this new series of tests, applicator, brush type, brush holder weight, brush preconditioning, and scrub medium will all be carefully controlled, with the objective of obtaining reasonably similar scrub cycle values among the cooperators. The scrubbing machines themselves, as well as operator technique, would of course remain as variables. Eleven members have volunteered for this round-robin. Results will be discussed at the June meeting.

D01.42.26—Burnish Resistance—W. Vanderslice, Chairman, was unable to attend and the meeting was chaired by A.J. Desmarais. He reviewed the proposed test method and presented new data showing differences in both dry and wet burnish resistance with an interior eggshell interior flat, an exterior low gloss house paint, and a flat wall paint. Dry burnishing gave a greater increase in 85° gloss than wet burnishing in all three cases. The question of the optimum number of rubbing cycles for this test was discussed, and the group decided on 5, 25, and 200 cycles for their next round-robin The use of a moleskin cloth was suggested as a substitute for cheesecloth to possibly make the procedure more reproducible. Since the perceived degree of gloss change

did not necessarily agree with the instrumental gloss measurements, the possible use of visual gloss standards was also discussed; but for the next round-robin, it was decided to continue using the 85° gloss change as the criterion of burnishing.

D01.42.28—Coating Removers—V. Scarborough, Chairman, discussed the first draft of a proposed method for testing coating removers. The draft method details the preparation of wood and metal panels, with subsequent application of several layers of a test coating. The coating remover is applied, allowed to work for a period of time, and then taken off. The efficiency of coating removal is then scored on a scale of 0-10, and damage to the underlying substrate is also noted. It was suggested that masonry should also be included as a substrate, and that the application of the coating remover should allow for a variety of techniques. These comments will be incorporated into a second draft. A round-robin will be initiated with at least three cooperators and the results will be reported at the next meeting.

D01.42—Two new methods passed D-1 ballot: D 5324, "Guide to Testing Waterborne Architectural Coatings," and D 5326, "Test Method for Color Development in Tinted Latex Paints." Reapproval was gained on D 3258-90, "Test Method for Porosity of Paint Films," and revisions were approved on D 4213-92, "Wet Abrasion Resistance of Interior Paints," and D 4828-92, "Test Method for Practical Washability." The proposed new "Test Method for Evaluating Clear Water Repellents on Wood" received two negatives, on a D-1 ballot and both were resolved. It will proceed to society ballot. The D01.42 ballot on the revision of D 3730-78 (1988), "Guide for Testing High-Performance Interior Architectural Wall Coatings," passed with no negatives. A motion was made and passed unanimously to ballot the individual test guides D 2931, D 3129, D 3358, and D 4540 for withdrawal in favor of the new combined guide D 5324 for waterborne coatings. D 3358 will be balloted first and the others subsequently, as recommended by D01.42.22. A new task group, D01.42.29, "Guides for Tests for Exterior Stains," was formed and will be chaired by new member A. Leathers. Anyone interested in participating is invited to contact the chairman. New member A. Leman has agreed to take over the chairmanship of Task Group D01.42.05, "Adhesion of Chalky Surfaces," with the cooperation of previous chairman B. Schiller.

SUBCOMMITTEE D01.45
MARINE COATINGS

R.P. Gangi, Chairman

D01.45.08—Antifoulant Release Rates—C.B. Bieter, Chairman. Interest has been

expressed in expanding the scope of the organotin test protocol to include cuprous oxide. New marine antifoulants can be added following review and approval of the analytical methods by the committee.

D01.45.11—Marine Coatings; Partially Immersed—C. Perez, Chairman. A draft of the "Standard Practice for Testing Biofouling Resistance of Marine Coatings Partially Immersed," was presented to the committee for review. Following the committee discussion, previously registered negative votes were withdrawn. A motion was passed to have the method, as designed by C. Perez and amended at the committee meeting, submitted for joint committee and subcommittee ballot.

D01.45.12—Intermittent Antifouling Immersion—K. Nichols, Chairman. K. Nichols was appointed chairman of D01.45.12 replacing R. Gangi. A name change was recommended and accepted for this task group to better describe the objective. The new name is "Standard Practice for Testing Antifouling Coatings in Cyclic (Air/Water) Exposure." No progress was made on a draft of the procedure. It was agreed that the bio and physical evaluations proposed in task groups 11 and 13 would be employed in rating the antifouling coatings in this method, in addition to the methods for submerged and water line exposures.

D01.45.13—Biofouling of Treated Surfaces—C. Perez, Chairman. J. Hinkle presented a draft of the "Standard Method for Rating Biofouling of Marine Coatings," which will be reviewed by this task group. It was agreed that the method should also include the rating of physical properties which can impact on the biofouling ratings. A previous decision to separate these subjects was reversed. C. Perez will continue as Chairman of the task group and will be assisted by J. Hinkle, G.W. Swain, and K. Nichols.

D01.45—A new task group, D01.45.14, has been established to study standard methods of measuring barnacle adhesion. A need has been identified for assessing the fouling resistance of a new generation of coatings which function by offering a poor surface for fouling attachment. G. Swain has been appointed Chairman of the group.

SUBCOMMITTEE D01.46
INDUSTRIAL PROTECTIVE
COATINGS

G.W. Gardner, Chairman

D01.46.02—Surface Preparation—K.A. Trimber, Chairman, reported that he voted negative on the August society ballot for D 4417, "Standard Test Methods for Field Measurement of Surface Profile of Blast

Cleaned Steel," to prevent publication of the technical errors discussed at our June 1992 meeting. D 4417 was withdrawn from ballot to correct these errors. Mr. Trimber corrected these technical errors in the precision statements and D 4417 was sent out on the concurrent D01.46/D-1 fall ballot. Two affirmative comments and no negatives were received on this ballot. Mr. Ashton's affirmative comment regarding using Dixon's test for discarding outliers to give improved precision and correlation was considered valid as long as the Dixon test is used for all test methods in this standard. However, all agreed that the present precision tests were technically correct and we should proceed with a society ballot. Further, all agreed to begin working immediately to incorporate Dixon's test in the next revision of D 4417. Mr. Ashton's second comment regarding deleting the word "qualitative" in 8.1.1 will be incorporated in the society ballot as an editorial change since the word "qualitative" had been removed from 8.2.1, 8.3.1, and 8.4.1 in the previous D-1 ballot.

The affirmative comment from Mr. Sellars regarding another suitable comparator and gage to be added to Notes 3 and 5 was accepted and will be incorporated in the next revision of D 4417. In addition, the Precision Section should be modified in the next revision of D 4417 to include information on the type of equipment used to generate the precision information.

D 2092, "Standard Practice for Preparation of Zinc-Coated (Galvanized) Steel Surfaces for Painting," is due for reapproval. This standard is significantly out-of-date when compared with current practices. D01.46 does not have the expertise to address many of the treatments and, specifically, those treatments used on zinc-coated (galvanized) steel sheets and coils.

K.A. Trimber and G.W. Gardner will approach N. Emily to see if he knows of someone who has the expertise to review and revise D 2092 with a change in scope to limit D 2092 to zinc-coated (galvanized) steel sheets and coils. This will most likely require a change in responsibility from D01.46 to another subcommittee. D01.46 then will concentrate on a new standard practice having a scope limited to zinc-coated (galvanized) structural type steel.

D01.46.03—Repainting—G.W. Gardner, Chairman, reported that D 4752, "Standard Test Method for Measuring MEK Resistance of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub," received two negatives on the D-1 ballot. K. Oschwald's negative stated a need to have a stronger safety statement on methyl ethyl ketone. D01.46.03 found Mr. Oschwald persuasive and agreed to change D 4752 to have the same type of safety statements as the new proposed "Standard Practice for Assessing the Solvent Resistance of Organic Coatings Using Solvent Rubs." D 4752 was with-

drawn from ballot to incorporate these changes.

W. Golton's negative pointed not the need for precision statements to validate this practice. D01.46.03 agreed that this is important and that it will be done. However, D01.46.03 unanimously agreed that we should proceed with revising this practice.

The new "Standard Practice for Assessing the Solvent Resistance of Organic Coatings Using Solvent Rubs" was on the D-1 ballot and received no negatives. Three affirmative with comments persuaded D01.46.03 to make editorial changes to the practice before it goes to society ballot.

D 1014, "Standard Test Method for Conducting Exterior Exposure Tests of Paints on Steel," is due for reapproval. After considerable discussion, D01.46.03 agreed that the scope of this method was outside D01.46 responsibilities. D01.27 on Accelerated Tests will be contacted to determine if they are willing to take over this method.

D01.46.04—Pull-Off Adhesion—M.E. McKnight, Chairman, reported that D 4541, "Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers," passed the D01.46/D-1 ballot with zero negatives and five affirmatives with comments. The Ketola affirmative comment proposing an editorial rewrite of the Annex notes referencing equipment availability convinced D01.46.04 to make the suggested changes.

The Clement affirmative comment suggesting editorial changes in the Scope 1.2 Section was found nonpersausive. However, Mr. Clement's comments on A2.1.4, pointing out the need for editorial clarification in the manner in which the device ranges are presented, was considered persuasive. Mr. Ashton's comments on stress rate in Section 5.1.5 and 7.5 are persuasive and will be added as Note 1.

Two technical errors in Footnote 7, Mr. Ashton's comment on adding "X100" to convert to percent and C.J. Ray's comment on dividing by 2, will necessitate reballoting. Since this standard passed D-1 ballot with no negatives, it will automatically go to society ballot. M.E. McKnight will vote negative on the society ballot in order to prevent publication. To proceed with this standard, Ms. McKnight will correct these technical errors and submit the revised standard to D-1 ballot.

Additional comments from Mr. Ashton on Bias and Table 1 will also be incorporated in the spring ballot, as well as P. Guevin's comments on correction to Lord Corporation's address.

D01.46.07—Inspection—G.W. Gardner reported that D 3276, "Standard Guide for Painting Inspectors (Metal Substrates)," received two negatives (W.S. Zimmt and H.R. Stoner) in the D-1 ballot. H.R. Stoner's negative stating the need to reword the second sentence in 5.4.4.4 to clarify the relation-

ship to organotin and antifoulant was persuasive. Mr. Stoner's recommendation to add to 6.2.6.1 the requirement to wipe dry around detected holidays to eliminate telegraphing was also persuasive.

Also, H.R. Stoner pointed out the incorrect metric in 9.5. After finding Mr. Stoner's negative persuasive, D01.46.07 agreed to remove this guide from ballot. W.S. Zimmt's negative had several parts, all of which were considered persuasive, except for the addition of the words "and commercial" to the sentence: "This guide is intended as a reference for those concerned with inspection of industrial 'and commercial' coating work." D01.46.07 felt that the addition of commercial coating work is outside the scope of this document, although D01.46.07 agrees that this guide could be used for some commercial work. The other eleven parts of W.S. Zimmt's negative will be incorporated into the next revision of this guide.

Comments from affirmative voters, D.S. Heinrich, F.M. Winkelman, and I.C. Sellars will also be added to the next revision.

Draft #4 of the "Standard Guide for Painting Inspectors (Concrete and Masonry Substrates)," was sent out to D01.46 for review on January 1, 1993. Comments will be incorporated into a draft to be sent out for spring D01.46 ballot.

D01.46.12—Sampling—K.A. Trimber, Chairman, reported that Draft #2 of the new "Standard Practice for Field Sampling of Coating Films for Analysis of Heavy Metals," was sent out for D01.46 ballot. One negative from J.C. Weaver and several affirmative comments were received. The Weaver negative pointed out the need for a cleaning method to remove soil, dirt, and dust from the top surface of the sampling area. D01.46.12 found this negative persuasive and this standard will be withdrawn from ballot to add a cleaning method to the Procedure section.

In addition, J.C. Weaver's second negative comment concerning reporting the condition of the substrate under the paint sample was also found persuasive. Condition of substrate will be added to the Report section. An affirmative comment from B. Wittenbaum regarding expanding the scope to include substrates other than steel was found persuasive. In addition, D 1400 will be added to the Referenced Documents list as a method to measure by film thickness on nonferrous substrates. B. Wittenbaum's second affirmative comment to broaden the scope to include sampling for purposes other than heavy metal analysis was found nonpersuasive. D01.46.12 wants to limit the scope to heavy metals at this time in order to provide a good standard practice in a timely manner. We will consider broadening the scope in the future once this standard has passed society ballot.

An affirmative comment from J. Raska regarding use of heat to aid sample removal

was found persuasive. K.A. Trimber will add a heat statement to the Procedure section. The revised standard will be sent to concurrent D01.46/D-1 ballot.

K.A. Trimber reported that no work had been done in D01.46.12 on a standard practice for sampling soil. However, ASTM E06.23 has a draft on "Standard on Soil Sampling." M.E. McKnight will send a copy of this draft to K.A. Trimber so that D01.46 can be a liaison to E06.23 for this new standard.

D01.46—G.W. Gardner, Chairman, welcomed two new members to the D01.46 roster: R. Max, of Zarco Industries, Chicago, IL, and D. Schwab, of Garrett Automotive, in Torrance, CA. K.A. Trimber reported that D01.46.02 has been unsuccessful in its attempt to find a subcommittee that has the expertise to review D 2092. D 2092 covers surface preparation of hot-dip and electroplating galvanized surfaces. S. Boocock will initiate a revised draft of D 2092 with a scope limited to surface preparation of hot-dip galvanized surfaces since D01.46 does not have the expertise to cover electroplating.

D01.46 reviewed the standards under its jurisdiction and assigned stewards for each one.

SUBCOMMITTEE D01.47 MASONRY TREATMENTS

F. Gale, Chairman

D01.47.01—*Water Repellency of Treated Masonry*—V. Scarborough, Chairman, reviewed the recently conducted round-robin using 2-inch mortar cubes as the test substrate. Initial reproducibility was improved. The cooperators were polled to determine if the procedures were properly followed.

Draft #3 of the "Proposed Test Method for the Determination of the Water Repellency of Treated Masonry," was distributed and reviewed in part by the group. It was the decision of the task group to initiate a round-robin of the method using three materials. A more extensive data form will be used for reporting results and the test specimens will be allowed to equilibrate for a longer period of time before beginning the test. V. Scarborough will report the results of the round-robin at the next meeting.

D01.47.05—*Water Vapor Transmission of Treated Masonry Substrates*—L.V. Mertz, Chairman, distributed Draft #3 of the proposed "Test Method for Water Vapor Transmission of Treated Masonry Substrates" and reviewed the results of a recent round-robin. Results indicate good correlation from lab to lab.

It was the decision of the task group to initiate another round-robin with the method.

The coatings to be used will include a siloxane, acrylic, and urethane to evaluate materials having a range from low to high WVT. A two-minute drainage period after the coated bricks are removed from the water will be specified. L.V. Mertz will work with several task group members to prepare the document for balloting and will report the results of round-robin testing at the June meeting.

D01.47.06—*Freeze/Thaw Resistance of Treated Masonry*—L. Stark-Kalsey, Chairman, distributed the results of the first round-robin conducted on the proposed "Test Method for the Evaluation of the Freeze/Thaw Resistance of Treated Masonry."

After a review and discussion of the data, it was the decision of the task group to initiate another round-robin. In this round-robin, the water will be changed and the water level measured weekly and metal containers will be used for testing. L. Stark-Kalsey will incorporate suggested editorial and technical changes into the method and distribute the revised method and the results of the round-robin testing at the June meeting.

D01.47.10—*Evaluation of Field Applied Treatments*—E. McGettigan, Chairman, distributed literature relating to current methods used to determine the effectiveness of field-applied treatments. Results from the proposed method conducted by E. McGettigan included discussion on the size of the test specimens, length of the immersion period, surface condition of the substrate, and hydrostatic head of water used during the immersion period.

E. McGettigan will work with the task group members in writing up the method and in setting up a round-robin of the method before the June meeting.

D01.47.11—*Research and Planning*—F. Gale, Chairman, discussed comments received since the last meeting. A request was submitted to D01.47 to establish a task group on textured coatings to develop a standard similar to TT-C-800. All interested parties should contact F. Gale.

V. Scarborough has expressed an interest in setting up a task group on bond breakers. Several task group members have agreed to assist her. A publicity release will be issued to find out how much interest there is in setting up this new task group.

J. Kreiger circulated a draft of a proposed test method for the "Qualitative Determinations of the Surface Beading Characteristics of Water-Repellent Treatments on Masonry Substrates." There was considerable interest and it was decided to create a new task group, D01.47.12, on water beading. Task group members will also look at quantitative methods using contact angle measurements of water beading to be presented at the June meeting. All interested parties should contact F. Gale.

D01.47—T. Sliva reported that the revision of D 1734, "Practice for Preparing Mortar Panels for Testing Paint Finishes," had completed D01.47 ballot without receiving any negatives. Several editorial comments were received and will be incorporated into the method and the item will be submitted to concurrent ballot.

The group was also informed that D 5095, "Determination of the Nonvolatile Content of Silanes, Siloxanes and Silane/Siloxane Blends Used in Masonry Water Repellent Treatments," has been accepted by the EPA as method to determine nonvolatile content. The method will be incorporated into D 3960.

DIVISION 50 PAINTS FOR FACTORY APPLICATION

SUBCOMMITTEE D01.51 POWDER COATINGS

N.D. Emily, Chairman

D01.51.02—*Hiding Power of Powder Coatings*—M.P. Sharma, Chairman. Round-robin test results, from four collaborators, were returned. Three sets of data were reviewed. One set was eliminated because the collaborator's spray-out developed craters. The standard deviation for weight determinations was consistently lower than for the "caliper" film thickness determination. This proposed method would utilize metal Leneta black and white contrasting substrates. There is still concern with the film uniformity of the test samples.

D01.51.03—*Spray Characteristics of Powder*—C. Merritt, Chairman. A draft version for powder transfer efficiency testing has been made available for comments by March 15, 1993, to the chairman. First-pass transfer efficiency of powder was recognized as an important application characteristic to the economical use of organic powder coatings.

D01.51.04—*Optical Properties of Powder Coatings*—H. Fairman, Chairman. A "Guide to Optical Properties of Powder Coatings" has been approved by D-1 ballot and will be submitted for society ballot in the spring of 1993.

D01.51.05—*Specific Gravity of Coating Powders*—D. Schneider, Chairman. The statistical results of an initial survey were discussed. The repeatability of micromeritic pycnometer results was +/-0.005 which was much better than some other methods screened. Two round-robin collaborations are scheduled for early 1993. One will use a

micromeritic pycnometer procedure while the second will be a modification of an existing industry method.

D01.51.06—Review of Particle Size Measuring Techniques—D. Pont, Chairman. Results of a powder particle size distribution round-robin were discussed. A urethane polyester system had been submitted to 21 collaborators in December 1992. Eleven sets of data were returned by January 15, 1993. The most frequently used technique was laser analysis. The final 11 round-robin results are expected to be returned before the June 1993 meeting in Philadelphia.

SUBCOMMITTEE D01.52 FACTORY COATED WOOD BUILDING PRODUCTS

S.B. Schroeder, Chairman

D01.52B—Hardboard—S.B. Schroeder, Chairman, reported that D 4144, "Standard Test Method for Estimating Package Stability of Coatings for Ultraviolet Curing," was again discussed. A negative on D01.52 ballot which suggested replacing the 60°C stability test temperature with 50°C was found persuasive, and it was recommended that it be reballoted accordingly.

A comment on a D-1 ballot suggesting a specification for the water quality to be used in D 2921, "Standard Test Method for Qualitative Tests for the Presence of Water Repellents and Preservatives in Wood Products," was discussed and found nonpersuasive for this rather imprecise field test.

More extensive comments were received on D 2793, "Standard Test Method for Block Resistance of Organic Coatings on Wood Panel Substrates." These were largely editorial in nature and were incorporated into the method with the exception of a suggestion to change the damage rating scale from an industry practice 0-5 to the more traditional 10-1. It was recommended that the revised method be submitted to concurrent D01.52/D-1 ballot.

It was also reported that the "Cobb Ring" water permeability method research report is being completed before submitting the method to D-1 ballot.

D01.52.13—Prefinished Hardboard—T. Rieth, Chairman, provided an overview of task group history and objective. This was followed by a discussion of Revision 4 of the proposed "Standard Guide for Performing Outdoor Weathering of Factory Coated Embossed Hardboard using Concentrated Natural Sunlight and a Soak-Freeze-Thaw Procedure." Several editorial changes were made, and one suggestion to scale edges and backs of samples was found nonpersuasive. It was recommended that the

method be submitted to concurrent D01.52/D-1 ballot.

Considerable testing was reported in progress in an attempt to extend the new method to primed hardboard. Much of this new data is expected to be available at the summer meeting.

Discussion of negatives and comments on the ballot of D 3719, "Standard Test Method for Quantifying Dirt Collection on Coated Exterior Panels," resulted in a recommendation to withdraw D 3719 from ballot for additional study.

D01.52.15—Film Thickness—R. Matejka, Chairman, reported little progress on the proposed round-robin pending submission of appropriate samples. These are anticipated and a round-robin design should be completed by the summer meeting.

D01.52—The minutes of the summer 1992 meeting were approved and all task group recommendations accepted by unanimous votes. The difficulty in resolution of negatives on the ballot of D 3719 was solved by withdrawal of D 3719 from ballot and formation of a new task group D01.52.17 on Dirt Collection, K. Oschwald, Chairman.

SUBCOMMITTEE D01.53 COIL-COATED METAL

R.J. Tucker, Chairman

D01.53.01—Pretreatment of Metal Substrates—H.E.G. Rommal, Chairman, discussed the recent draft of a proposed practice, "Determination of Chromium in Metal Surface Treatments by X-Ray Fluorescence," prepared by D. Steele. A third draft will be prepared by the next meeting.

D01.53.03—Accelerated Weathering—D.A. Cocuzzi, Chairman. Due to the lack of adequate samples, there was no need for a meeting. A complete set of samples is expected by the next meeting.

D01.53—In the absence of the chairman, C. Milburn, Vice-Chairman, conducted the meeting. He reported that D 3794, "Standard Practice for Testing Coil Coatings," will be reballoted shortly. D 3003, "Test Method for Pressure Mottling and Blocking Resistance," was reviewed. Minor changes are needed, thus this method will be balloted. D 4147, "Standard Practice for Applying Coil Coatings Using the Wire-Wound Drawdown Bar," was reviewed. With some minor changes, this too will be balloted. Some consideration is being made to writing a specification for the variable- gap bar, better known as a Damon bar. Milburn also led a discussion of the subcommittee's apprehensions regarding the writing of performance specifications, as suggested by members of A-5.

A new task group was formed to investigate and evaluate the various methods to measure the dry film thickness (DFT) of coil coatings. G.R. Pilcher will serve as task group Chairman. Particular attention will be paid to a new device (although an old concept) manufactured in Canada by DJH Designs. At the next meeting, a plan will be formulated to perform round-robin testing to evaluate all known devices suitable for measuring DFT in coil coatings.

SUBCOMMITTEE D01.55 FACTORY APPLIED COATINGS ON PREFORMED PRODUCTS

G.R. Pilcher, Chairman

D01.55.06—Coatings on Plastics—M.A. Lutterbach, Chairman, conducted a survey regarding revision of D 3002, "Practice for Evaluation of Coatings for Plastics." The survey generated an excellent (51%) response. Based upon these results, as well as additional comments submitted at the D01.55.06 and D01.55 meetings, M.A. Lutterbach will prepare a revised draft for a guide to be presented at the June meeting.

D01.55.08—Transfer Efficiency—Laboratory Conditions—R.J. Diem, Chairman, reported that the new, "Standard Practice for Evaluating and Comparing Transfer Efficiency under General Laboratory Conditions," received no negatives when it was sent out for society ballot. This method will be published in the *1993 ASTM Book of Standards* as D 5327. The task group was officially dissolved.

D01.55.09—Transfer Efficiency—Production Conditions—J.S. Komjathy, Chairman, indicated that the new, "Practice for the Determination of Transfer Efficiency Under General Production Conditions for Spray Application of Paints," received one negative on the recent society ballot, citing the need for precision data. This was unanimously found not persuasive by the members of D01.55, since it would not be possible to find two identical production sites which could generate such data. The task group will immediately begin producing a revised version of this practice; it is possible that D 5009 can be reviewed for repeatability data which may be of use in this practice.

D01.55.10—VOC of Radiation-Cured Coatings—J.J. Brezinski, Acting Chairman, reported that the negative cast in the D-1 ballot against the proposed new, "Standard Method for Volatile Content of Radiation-Curable Coatings," has been withdrawn. The method will advance to society ballot with only editorial changes. The EPA has been made fully aware of this method, and Chairman Carlblom will write EPA formally re-

questing that they adopt this method. It appears that Methods A and B do not apply to two-component catalyst cured systems, and T. Wood has been asked to organize a new task group to address this issue.

D01.55.11—Scratch Resistance of Automotive Topcoats—J.S. Komjathy, Chairman, advised that existing marring tests are too severe to deal with this subtle effect. Since manual operations involve too much operator error, a mechanical "brush test" was run by BASF and Du Pont. Preliminary conclusions based upon image analysis indicate that the test has validity. Round-robin testing will be initiated.

D01.55—Factory Applied Coatings—G.R. Pilcher, indicated that the six methods/practices requiring action in 1993 are being reviewed; D 1211 and D 3459 will be sent for D-1 reapproval ballot. Both D 1308 and D 3023 will be sent to society ballot with editorial changes. It was announced that AFMA involvement will not occur at this time, and will be dropped from future agendas. At the request of L. E. Thieben, D01.55 will schedule an organizational meeting in Philadelphia to explore the feasibility of writing a method for testing "conductivity of boom coatings under conditions of 60,000 volts." This was G.A. Pilcher's final meeting; he will be replaced in June by long-time D01.55 supporter E.A. Praschan, of the Motor Vehicle Manufacturers Association of the United States.

SUBCOMMITTEE D01.56 PRINTING INKS

J.M. Fetsko, Chairman

D01.56.02—Lightfastness of Prints—J. Robins, Chairman, reported that the revised D 3424, "Test Method for Lightfastness and Weatherability of Printed Matter," has been approved and will appear in the *1993 ASTM Book of Standards*. Data generated by "natural daylight under glass" is being statistically evaluated, and carbon-arc data must be assembled.

D01.56.11—Nonvolatile Content—B. Blom, Chairman, reported that the proposed title change of D 4713 to, "Test Methods for Nonvolatile Content of Heatset and Liquid Printing Ink Systems," passed the society ballot and will appear in the *1993 ASTM Book of Standards*.

D01.56.12—Viscosity of Liquid Inks—J. Daugherty, Chairman, reported that five of eight laboratories reported data from a round-robin designed to compare the precision of the Shell and Zahn cups. The study was conducted with prediluted inks and a standard oil.

D01.56.14—Setting of Heatset Inks—D. Ness, Chairman, reported that he ran Sinvatrol tests using an eight-path wedge plate on the Vandercook. It was suggested that he also try a two-path variable film thickness plate on the Little Joe.

D01.56.15—Density (wt/gal) of Paste Inks—P. Ford, Chairman, distributed round-robin results in which seven labs tested ink densities by the weight-per-gallon cup along with VOC40. The results will be analyzed statistically.

D01.56.17—Guide for Printing Inks—J. Fetsko, Chairman, reported that D 5010, "Guide for Testing Printing Inks and Related Materials," should be updated to include the revised title for Test Method D 4713, the new test method on VOC40, and method D 2090 for clarity of paint and ink liquids.

D01.56.19—Printing Strength and Opacity—G. Wouch, Chairman, reported that he conducted a study using a blue offset ink diluted with a white tinting base and an extender in order to obtain samples varying in opacity. Delta E was the only parameter that showed any promise. It was suggested that he try laying down an ink film on a glass plate or overprinting one ink on a dry ink film of a different color.

D01.56.20—Technical Coordination—B. Blom, Chairman, reported that EPA will support the use of method D 2369 with a one-hour waiting period; 24 hours is a concern due to possible skin formation. He also reported that the TAG and Label Committee has approved a new method for the Sutherland Rub Tester.

D01.56.21—Print Abrasion—G. Vandermeersche, Chairman, reported that he is developing a proposed standard covering the use of the GV-CAT for testing cans and other metal decorated products. He also described a new target system for evaluating smudging in nonimpact printing.

D01.56.22—Drying of Oxidizable Inks—B. Blom, Chairman, reported that he is ready to prepare in ASTM format a proposed test method by which the drying of printed oxidizable inks is evaluated using a modified Sutherland Rub Tester and a cotton wipe with squalene.

D01.56.23—Degree of Dispersion (Microscope)—A. Mercado, Chairman, reported that he is exploring methods for eliminating the human factor from preparation of slides for microscopic evaluation of degree of dispersion.

D01.56.25—Degree of Radiation Curing—E. Kobylarz, new Chairman, agreed to conduct a literature survey on methods

for evaluating degree of cure in radiation curing of printing inks.

D01.56.27—VOC40 on Non-Heatset Paste Inks—P. Ford, Chairman, reported that the new test method, "VOC40 of Non-Heatset Paste Inks at 40°C," has been assigned the number D 5328 and will appear in the *1993 ASTM Book of Standards*. Round-robin data discussed in D01.56.15 will be the basis for a revised precision statement.

D01.56.28—Tinting Strength of Liquid Inks—D. Ness, Chairman, reported that three labs returned round-robin data in which liquid inks were reduced with a tinting base and placed in a cuvette for spectrophotometric determination of tinting strength. One of the labs detected a shift in spectrophotometric response with time in the cuvette. It was also agreed that the tints should be based on a weight ratio rather than a specific set of weights.

D01.56.29—Membership—J. Daugherty, Chairman, reported that T. Sayres has resigned from ASTM due to a change in work assignment. New members of ASTM and D01.56 include E. Kobylarz and H. Brookes. R. Marcus and R. Schiller have also requested D01.56 membership.

D01.56—Having accepted the chairmanship of D01.37 Ink Vehicles, A. Scarlatti submitted his resignation as secretary of D01.56. H. Brookes was appointed as his replacement.

SUBCOMMITTEE D01.57 ARTISTS' PAINTS AND RELATED MATERIALS

M. Gottsegan, Chairman

D01.57.02—Lightfastness of Pigments—T. Vonderbrink, Chairman. A negative vote by W. Ketola on a D-1 ballot of a revision of D 4303 was discussed and ruled persuasive; a suggested revision was presented and will be submitted for concurrent D01.57/D-1 balloting.

W. Ketola was also concerned with other specifics of D 4303 regarding sections 7.3.2.2, 7.3.3.2, and 7.3.3.3 on measuring total radiant energy and the calculation of broadband radiant energy dose. W. Ketola agreed to join a group consisting of T. Vonderbrink, R. Kinmonth, and J. Luke to work up a revision of D 4303 to address these issues. T. Takigawa will also be asked to participate.

Further results from additional fluorescent lamp testing in the HPUV apparatus were distributed and discussed, in an ongoing effort to find a replacement for the discontinued Verilux bulb specified in D 4303.

Although the new results seem to confirm earlier data, another round of testing was agreed to before making a final recommendation for the revision of D 4303.

D01.57.04—Specification of Artists' Paints—A. Spizzo, Chairman. Changes in D 4302, D 4303, D 5067, and D 5098 were proposed to include recommending testing for pigments which may darken in masstone, as a result of a study reported on by B. Gavett, of Golden Artist Colors. Prior to initiating the changes, a further study will be conducted under the auspices of D01.57.07 on Physical Properties, to clarify the extent to which different binders and varied formulations affect darkening and to quantify the extent of the darkening. Six manufacturers expressed interest in participating in the study.

A change in D 5098 to require the artists' acrylic emulsion paints be evaluated for pigment bleeding was proposed and will be sent for D01.57 balloting.

A proposal to further amend D 5098, presented at the last meeting and which would have allowed the use of chemical description labels for pigment names, instead of the common name from Table I—a change which would have affected all the other paint specifications—was withdrawn after a compromise amendment was agreed upon. Under the new agreement, sections 5.1.5 of D 4302, D 5067, and D 5098 will be modified to read: "The common name of the significant pigment used shall be given in letters no less than 1/2 the type size of the title and be clearly legible." This proposed change will be sent for D01.57 ballot.

A new task group, D01.57.14 on Colored Pencils was established, with I. Seymour as Chairman. A preliminary rough draft of a proposed "Standard Specification for Artists' Colored Pencils" was distributed and lightfastness test methods were discussed. Mr. Seymour requested input from the Colored Pencil Society of America and other manufacturers of colored pencils in preparing the next draft.

D01.57.07—Physical Properties—R. Gamblin, Chairman, reported on a study by the Conservation Analytical Lab of the Smithsonian, using computer modeling instead of artificial aging techniques to examine the effect of temperature and relative humidity changes on the performance of paint films. He proposed that the results of such work could be used to strengthen existing performance standards and support the development of new standards. A survey of artists to determine their concerns in this area will be drafted for the next meeting.

D01.57.08—Toxicity Labeling—W. Stopford, Chairman. A phrase, "Not for use in Health Care Facilities," was proposed by W. Stopford to be added to the Annex of D

4246 and will be sent for D01.57 ballot. Removal from the Annex of the phrase, "Possible Cancer Agent Based on Tests with Laboratory Animals," was also proposed: the word "possible" is too vague, and the already-existing phrase, "Cancer Agent Based on Tests with Laboratory Animals," can be used. This proposed change will also be sent for D01.57 ballot.

D01.57.09—Watercolors—T. Vonderbrink, Chairman, stepped down as chair of this task group, and W. Upchurch and G. de Calan agreed to co-chair. There was no new business to report.

D01.57.10—Consumer Evaluation—J.T. Luke, Chairman, reported that both new standards dealing with the visual determination of the lightfastness of art materials passed D-1 balloting and will be balloted at the society level in March 1993. Some slight editorial revisions were proposed: if they do not inhibit society balloting they will be made.

D01.57.11—Gouache Paints—T. Takigawa, Chairman. In Mr. Tagikawa's absence, M. Gottsegen presided. Final results of Mr. Tagikawa's lightfastness tests and a third draft of the proposed "Standard Specification for Gouache Paints" were distributed and discussed. A change in the definition of "designers gouache" was requested, since the current one implies the use of less permanent pigments and lower quality—this is problematic for companies which have historically labeled their gouache paints as "designers," but which use permanent ingredients. Further comments were requested to be sent to M. Gottsegen.

D01.57.12—Determination of Toxicity—W. Stopford, Chairman. Three negatives were received on D01.57 ballot of "Standard Test Method for Determining Bioavailability of Metals in Art Materials." Two were withdrawn, one with the proviso that some editorial changes be made in the section on Scope. The third negative, by H. Fujimoto concerning statistical reporting procedures, was voted persuasive. There were several other editorial suggestions submitted. When the proposed standard has been modified to reflect the changes, it will be sent for D01.57/D-1 balloting.

W. Stopford reported that the proposed "Standard Test Method for Determining Bioequivalency of Nitroaromatic Amines in Art Materials," still needs intralaboratory precision testing, and that he will approach the Dry Color Manufacturers Association and the Art & Craft Materials Institute to manage the effort.

Validation studies and intralaboratory precision testing are complete on two other methods under consideration: "Determination of Respirable Fraction of Art Materials" and "Determination of Crystalline Silica in Art Materials." Discussion produced some

disagreement about the suitability of a water column test for determining respirable fractions of certain types of art materials, and W. Stopford agreed to look into it.

Dr. Stopford also presented a summary and brief description of other methods to be considered for adaptation in support of D 4236: Methods for the Determination of Free Formaldehyde, Acrylate Monomers, Ethylene Oxide, Acrylamide and Vinyl Acetate Monomers in Art Materials.

C. Jacobson distributed copies of the recently published final guidelines for "Determining Chronic Toxicity of Products Subject to FHSA," which also contains the final supplemental definition of "toxic," defining chronic toxicity under the FHSA (Federal Hazardous Substances Act). Mr. Jacobson also reported on the CPSC's enforcement activity regarding the Labeling of Hazardous Art Materials Act, which incorporates D 4236. He said that awareness of the law by fine arts materials producers is generally good, but other groups like importers of children's products (watercolors, craft materials, clays, and so on) usually don't consider themselves to be in the "art materials" business. It was suggested that the U.S. Customs Service might be of assistance regarding proper labeling of imports.

C. Jacobson also reported that the CPSC has requested that D 4236 be revised to eliminate the requirement that toxicologists using D 4236 be "board-certified," in order to make the standard consistent with the Commission's enforcement policy. Since a D01.57 member expressed substantial opposition to the idea of loosening the definition of "toxicologist" in the standard, no direct action was taken. However, a modification of a footnote in D 4236 referring to the law was moved and passed: "Under this law and its regulations (16 CFR 1500), manufacturers must submit to CPSC (Washington, DC 20207) written criteria used by the toxicologist to recommend labeling." This change to D 4236 will be sent for D01.57 balloting.

D01.57.13—Washability Testing—C. Sathre, Chairman, has agreed to draft a washability standard and requested input from the members.

D01.57—A vote to reapprove D 4838 with no changes passed. Votes determining persuasive negatives on the revisions of D 4303 and the proposed "Bioavailability: standard passed. The motions for changes in the Labeling Annex and a new footnote in D 4236, the addition of a requirement to test pigments for bleeding in D 5098, and to reduce the type size requirement of the common names in D 4302, D 5067, and D 5098, all passed.

The next D01.57 meeting will be in conjunction with the National Art Materials Trade Association in Miami, FL, April 30, 1993.

DIVISION 60
PAINT APPLICATION

SUBCOMMITTEE D01.61
PAINT APPLICATION TOOLS

F.B. Burns, Chairman

D01.61.01—Paint Brushes—T. Sliva, Chairman, reported that the proposed "Standard Practice for Physical Characterization of Paint Brushes" had successfully completed society ballot (92-2) and will be published as D 5301-92. Copies of the new standard were distributed and the chairman has agreed to serve as steward for the method.

T. Sliva distributed the results of the recently completed round-robin on leveling efficiency and ease of clean-up of paint brushes. The results of this round-robin looked promising. It was the decision of the group to repeat the round-robin. Six members have agreed to be cooperators. The leveling test will be altered to specify an application rate of 500 ± 50 ft^2/gal. The times required for the various steps in the clean-up procedure will be extended and the coated brush will be allowed to air dry for only 30 minutes before clean-up.

The chairman will determine the number of paint samples needed to develop a precision and bias statement, locate a source for panels for brushout, evaluate which paint would be best suited for this test.

J. Price will develop a statement as a guide for the type of paint to be recom-

mended for use with this method. G. Harsch will develop a statement as a guide for the type of paint to be used for brush clean-up when using bristling materials of various colors, and submit a procedure used in the industry for cleaning a brush after use.

T. Sliva will report the results of the next round-robin and present the first draft of the method for review by the task group at the June 1993 meeting.

D01.61.02—Paint Rollers—J.F. Price, Chairman, led a discussion on the method. Two members volunteered to become cooperators bringing the total to the desired five. The chairman agreed to send preliminary information to the new cooperators, including that for panel preparation. As soon as the method is revised, a new round-robin will be initiated.

D01.61.05—Bulk Density of Filaments and Bristle—W.B. Bond, Chairman, provided a letter summarizing data for level and tapered, hollow and solid filament. When data from T.S. Simms (hollow tapered) is supplied, a research report will be drafted with assessment of precision and accuracy for level filaments.

An insert of 1.45 in. diameter, 1 in. long and tapered conically for one-half that length was designed and tested to aid in measurement of bundle tip diameter as well as butt diameter. Use of this insert improved test reproducibility for two testers. W.B. Bond agreed to issue a revised draft for this test as well as a description of the derivation of the new equation.

H. Stanley, described a device Micromeritics is developing to measure bulk density and promised a report on the status for

the next meeting. The potential advantage of this technique is that an assumption of the shape of the bundle taper is not needed.

D01.61.06—Buckling Resistance of Filaments—W.B. Bond, Chairman, led a discussion concerning the Ketema test device status and filaments needed for testing. After explaining the reasons for the delay, Mr. Simms estimated delivery of equipment by mid-February.

Polymers (S. Nelson) agreed to supply one bundle each of 2 in. hollow polypropylene and polyester in .040 in. and .050 in. wall and 8, 10, and 12 mil thickness. Ketema (T. O'Brien) will supply the same sizes in one wall thickness. This will provide 18 bundles for testing. The chairman requested data from tests by April or May so he could analyze the data and prepare a report for the June meeting. Once the initial round of testing is complete, the method will be revised. The group agreed that a pass/fail test is desirable from both the supplier and user point of view.

D01.61—F. Burns commented that the newly issued 5301-92, "Standard Practice for Physical Characterization of Paint Brushes," was to be reviewed by E. Louder, as a model for a possible new task group on Physical Characterization of Woven Paint Roller Fabrics. T. Sliva circulated a report from the Paul N. Gardner Company, which related to testing bristle brush characteristics. Their model D12VFI paint film washability machine was modified so that brushes used in washability tests could be evaluated for bristle stiffness. Upon review, it was the consensus that this procedure is not applicable to paint brushes.

January 1993 Subcommittee Reports of ASTM Committee G-3

The January meeting of ASTM Committee G-3 on Durability of Nonmetallic Materials was held on January 17-20, 1993 at the Crown Sterling in Fort Lauderdale, FL. The present membership of G-3 is 90.

Future Meetings

June 27-30, 1993—Franklin Wyndam, Philadelphia, PA.
January 23-26, 1994—Crown Sterling, Fort Lauderdale, FL.
June 26-29, 1994—Marriott Crab Tree Valley, Raleigh, NC.
January 22-25, 1995—Hyatt Regency, San Antonio, TX.

SUBCOMMITTEE G03.01
EDITORIAL AND DEFINITIONS

M.J. Crewdson, Chairman

The G-3 General Terminology Standards has passed society ballot and will appear in the *1993 ASTM Book of Standards* as G113-92. The chairman advised that the immediate incorporation of the new accepted terminology into documents under revision was necessary. There was discussion concerning the procedures for updating the terminology standard, incorporating new terms, and how to correctly reference the new G113 document.

A draft of the proposed "Practice for Conditioning and Handling of Nonmetallic Materials" was presented. A discussion of proposed changes followed, and at the request of the subcommittee, the chairman will present this draft for immediate subcommittee ballot.

A draft of the proposed "Practice for Application of Statistics to Weathering Data" was presented. It was agreed that this document should be a guide. The chairman will make further revisions to the draft in order to make a more complete presentation.

As a result of the increased interest generated by the two new draft standards, it was decided that two new task groups be established: G03.01.02, Conditioning and Handling; and G03.01.03, Statistics. These task groups will have their own distinct time slot at the next meeting, as it is hoped to attract a wider participation.

SUBCOMMITTEE G03.02
NATURAL ENVIRONMENTAL TESTING

J.S. Robbins III, Chairman

Data was presented at this and previous meetings concerning the transmittance of glass used in G 24, "Conducting Exposures to Daylight Filtered Through Glass." At the January 1992 meeting of this subcommittee, A G03.02 ballot which would have required laboratories to measure and report glass transmittance received one negative vote. Discussion concerning this negative vote continues and a revision to G 24 will likely be balloted prior to the June 1993 meeting in Philadelphia.

The G03.02 ballot of G 90, "Performing Accelerated Outdoor Weathering of Nonmetallic Materials Using Concentrated Natural Sunlight," received one negative vote. The latest revision of G 90 requires the use of a collimating tube to measure direct beam ultraviolet radiation. A simpler method of making this measurement was proposed and will be investigated further. If this investigation is satisfactory, G 90 will be revised and reballoted prior to the June meeting.

Task group G03.02.05, which was assigned to develop standards concerning the measurement of short wavelength ultraviolet radiation, has been made a separate subcommittee (G03.09). The standards on solar radiation measurement formerly managed by subcommittee G03.02 will be handled by G03.09. This includes E 824, "Transfer of Calibration from Reference to Field Pyranometers," and E 816, "Calibration of Secondary Reference Pyrheliometers and Pyrheliometers for Field Use."

SUBCOMMITTEE G03.03
SIMULATED AND CONTROLLED ENVIRONMENTAL TESTING

R.M. Fischer, Chairman

G03.03.01—Round-Robin Studies—R. Fischer, Chairman. A copy of the G 26 round-robin final report will be attached to the G03.03 minutes. Group discussion centered on identifying sources of variability in G 23, G 26, and G 53 round-robins. Of particular interest was the outlier lab II of the G 26 round-robin. The range of gloss values for the ten round-robin test specimens was especially compressed in this lab. An action plan was developed which involves; (1) comparative sample analysis for surface contaminants between G 26 lab II and the other participants, (2) investigate protocol problems, and (3) prepare precision statements for these standard practices for discussion at the June 1993 meeting.

G03.03—A revision of G 53-91, "Standard Practice of Operating Light and Water Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Non-metallic Materials Lamp Application Annex," received no negatives on G-3 ballot and will go to society ballot. This particular revision describes typical fluorescent lamps that are used in fluorescent UV-condensation devices.

The G03.03 ballots for the revision of G 23-90, "Standard Practice for Operating Light Exposure Apparatus (Carbon Arc Type) With and Without Water for Exposure of Non-Metallic Materials," and of G 26-90, "Standard Practice for Operating Light Exposure Apparatus (Xenon Arc Type) With and Without Water for Exposure of Non-Metallic Minerals," passed with no negatives. These particular revisions set the specimen water spray temperature to 25±5°C. Previously, these standards recommended 16±5°C which often meant the supply water required special treatment to fall within this temperature range. Few labs complied with this requirement.

The G03.03 ballot of draft "Standard Practice for Conducting Light-Exposure Tests of Non-Metallic Materials using Horizontal Specimen Tray Xenon-Arc Exposure Apparatus," returned with several negatives that were found persuasive. J. Robbins III will redraft this proposed standard which will again be balloted at subcommittee level.

Data was presented comparing Suga Test Instruments xenon arc devices to G 26 devices. Spearman rank correlation coefficients between G 26 laboratories (for gloss loss of 10 vinyl tape samples) ranged from 0.82 to 0.99. In Trial #1, correlation coefficients from the Suga devices (to the G 26 devices) ranged from 0.32 to 0.81. Repeat exposures were conducted and the correlation coefficients compared to the G 26 devices had increased to 0.86 to 0.98. The Suga water-cooled devices in Trial #2 gave essentially equivalent results compared to the G 26 devices. The ensuing discussion involved whether Suga Test Instruments should be added to the G 26 footnote as a supplier or should footnotes referencing any equipment manufacturer be dropped from G03.03 standard practices. D. Van Court moved (second by K. Scott) that all device manufac-

turer footnotes by dropped from G 23, G 26, and G 53 standard practices. The subcommittee voted 14 affirmative and 6 opposed to ballot this proposed change at subcommittee level.

P. Brennan requested that time be set aside at the next meeting to discuss the transition of the current G03.03 "equipment-based" standards to a "performance" standard. Mr. Brennan agreed to prepare a proposal that will serve as a basis for this discussion.

SUBCOMMITTEE G3.05 REFERENCE MATERIALS FOR EXPOSURE TESTS

W.D. Ketola, Chairman

W. Ketola reported that all seven laboratories participating in the round-robin have completed exposures of polysulfone films in fluorescent UV/condensation devices. Results show that there is excellent agreement between two operators in one laboratory for determination of absorbance change at 330 nm. Components of variance analysis showed that 80% of the variability in 33 nm absorbance change is caused by differences between laboratories and that 20% of the variability is caused by differences between replicate specimens exposed in a single device. The laboratory variable includes variability caused by the exposure device and the spectrophotometer in each laboratory. The continued change in 330 nm absorbance after exposure makes it impossible to separate these variables. Based on analysis of data for all exposure times, the 95% confidence interval for 330 nm absorbance change for replicate specimens of polysulfone exposed in a single laboratory is ±0.03 absorbance units. The 95% confidence interval for 330 nm absorbance change is ± 0.06 absorbance units for polysulfone films exposed in any laboratory. The variability between laboratories is smaller for shorter exposure times. Additional analysis of data will be reported at the next meeting.

Six of seven laboratories have completed exposure of polystyrene in water-cooled xenon arcs and determination of CIELAB Δb* according to SAE J1960. Analysis of data obtained to date shows excellent agreement between operators in a single laboratory. The data for Δb* determined by transmission measurements shows less variability than for Δb* measured by reflectance. In addition, Δb* data determined using a single colorimeter shows significantly less variability, indicating the separation of laboratory variability into exposure device and colorimeter components may be possible. A complete report will be presented at the next meeting.

Only three of seven laboratories have completed carbon arc exposures and determination of carbonyl absorbance change of the polyethylene film. It is hoped that all laboratories will have completed exposures before the next meeting.

N. Searle reported results from preliminary measurements of activation spectra for the polyethylene films exposed in carbon arc devices. In all cases, radiation in the 290-340 nm region caused the most carbonyl absorbance increase. It also appears that there is significant variability in carbonyl absorbance increase within a single specimen of the polyethylene film used for the round-robin. A more complete summary of activation spectra determination for the polyethylene film will be presented at the next meeting.

SUBCOMMITTEE G.03.07 LIAISON AND HARMONIZATION

R.A. Kinmonth, Chairman

In the third meeting of this subcommittee, final task group assignments were made, with chairs as reported at last meeting. Liaison reports were given by several members covering their work on other ASTM committees as well as within ISO, DIN, and SAE.

jct Journal of Coatings Technology

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Society Meetings

BIRMINGHAM .... MARCH

"Rheology"

The meeting's technical presentation was given by Mark Power, of Carri-Med Ltd. Mr. Power's topic was "RHEOLOGY AND ITS IMPLICATIONS FOR THE COATINGS INDUSTRY."

The speaker defined rheology as the study of flow and deformation of matter. He said that over the last 5-10 years the interest in this fairly new science has increased.

Mr. Power reviewed the many different theoretical models of flow that have been in existence for many years, including: Bingham, plastic, pseudoplastic, Newtonian, and dilatent.

Constant shear equipment and constant stress equipment were discussed. The technicalities of yield stress, the amount of force needed to make it flow, were explained.

Mr. Power said that three ways to measure sample structure include: Hysteresis Loop Technique, oscillation (wobble), and creep. The Hysteresis Loop Technique destroys the structure, is not repeatable, and can be called shear thinning. Oscillation does not destroy the structure and is good for short times and fast processes. The creep technique can only be used on controlled stress equipment which enables materials to be looked at over a long time frame.

DAVID C. MORRIS, *Secretary*

CDIC .... FEB.

"ISO 9000"

The meeting's first speaker was Lisa Brown, of Boehringer-Mannheim Company, who discussed "ISO 9000."

The speaker described ISO 9000's various components and objectives, indicating that is analogous with Total Quality Management.

Ms. Brown stated that ISO 9000 requires a company to give its players strategy. She pointed out that the objective of the ISO Series of Standards is to describe the generic requirements of a set of quality systems that maximize the probability that a product or service organization will satisfy customer needs in a manner consistent with its organizational policies, goals, and objectives.

The speaker said that ISO 9000, an international standard that spells out guidelines, is based on British Standard 5750. Ms. Brown explained the different ISO 9000 standards, including: ISO 9001—a quality assurance model for design/development; ISO 9002—a quality assurance model for production and installations; ISO 9003—a

model for final inspections; and ISO 9004—guidelines for elements.

It was noted that EN 29000 to EN 29004 are regional European direct equivalents for the ISO 9000 series. The U.S. equivalent is ANSI/ASQC Q 90 to Q 94.

The advantages of ISO 9000, an opportunity for increased sales, maintained sales, and decreased costs, were discussed.

Q. In a recent issue of Business Week, an article listed disadvantages of ISO 9000. What comments can you make?

A. One disadvantage is to make the performance requirements too limiting. The document shouldn't be so rigid that it prevents the employee from doing a good job.

The second speaker was John R. Johnson, of Reilly Industries, whose topic was "NEW CATALYSTS FOR EPOXY COATINGS."

PAUL R. GUEVIN JR., *Acting Secretary*

CDIC .... MAR.

"Chemophobia and Ecohysteria Mongers"

The Nominating Committee's slate of officers for 1993-94 was announced as follows: President—Paul R. Guevin Jr., of P.R. Guevin Associates; Vice President—Alan L. Machek, of Dow Corning Corporation; Secretary—John C. Avery, of Cintech Industrial Coatings and Kenneth Pendleton, of K.A. Pendleton Company, Inc.; and Treasurer—Sherrill Henegar, of Reilly Industries and William Jelf III, of Akzo Coatings, Inc.

Joseph Reckers, retired, formerly of Perry & Derrick Company, Inc., was nominated for Society Honorary Membership. A vote will take place during the April Society meeting.

It was announced that Hugh Lowrey, of Perry & Derrick Company, Inc., has been appointed as the Society Representative to the Ohio Paint Council's Operating Committee.

A chairman is being sought to head up the planning for the Society's 75th Anniversary celebration in 1995. Interested people should contact President Alipio R. Rubin Jr., of Rubin Dispersions.

Golden Gate Society member Robert D. Athey Jr., of Athey Technologies, gave the meeting's technical presentation. His topic

was "CHEMOPHOBIA MONGERS AND ECOHYSTERIA MONGERS."

Dr. Athey summarized his activity regarding the environmental community over the last four years. Global warming was one of his topics of discussion. He said that carbon dioxide levels may be increasing and the reason why may not be fully understandable.

The speaker said that when scares arise, they are full of sound and fury but usually signify nothing. Each case has multiple levels of direction at each stage and each monger group is different. Each of these mongers has a unique driving force for their contribution of the hysteria.

Dr. Athey reviewed the five groups that are "staging" these scares—media, regulators, legislators, public interest groups, and labor/trade unions.

In conclusion, the speaker stated that people should take a pro-active approach and confront inappropriate sources of information.

The educational speaker was Mark Ballard, of Huntington National Bank, who discussed "BUSINESS ETIQUETTE IN THE 90s."

The speaker reviewed many of the common business *faux pas* according to Catalyst, a New York based nonprofit organization. Some of the more common mistakes include: calling business associates and clients by their first names, sending out sloppy business correspondence, smoking in the wrong places, and talking only about business at a function that is both business and social.

PAUL R. GUEVIN JR., *Acting Secretary*

VOC COMPLIANT COATINGS—Rich Johnson, of Cargill, Inc., addresses members of the Chicago Society during the February meeting

Constituent Society Meetings and Secretaries

BALTIMORE (Third Thursday—Martins West, Baltimore, MD). HELENE J. RANFONE, Duron, Inc.,10460 Tucker St., Beltsville, MD 20705.

BIRMINGHAM (First Thursday—Strathallan Hotel, Birmingham, England). D.C. MORRIS, PPG Industries (UK) Ltd., P.O. Box 359, Birmingham, B16 0AD, England.

CDIC (Second Monday—Location alternates between Columbus, Cincinnati, Dayton, and Indianapolis). JEFFERY I. BUCHMAN, Akzo Coatings, Inc, P.O. Box 147, Columbus, OH 43216-0147 .

CHICAGO (First Monday—Sharko's Restaurant, Villa Park, IL). NATU C. PATEL, Ace Hardware Corp., Paint Div., 21901 S. Central Ave., Matteson, IL 60443.

CLEVELAND (Third Tuesday—Brown Derby, Independence, OH in Nov., Mar., and Apr.; Cleveland Hilton, Cleveland, OH, in Feb.; Landerhaven, Mayfield Heights, OH, in May). CONSTANCE F. WILLIAMS, The Glidden Co., 801 Canterbury Rd., Westlake, OH 44145.

DALLAS (Thursday following second Wednesday—Holiday Inn Crowne Plaza, Dallas, TX). BENNY PUCKETT, Kelly-Moore Paint Co., Inc., 301 W. Hurst Blvd., Hurst, TX 76053.

DETROIT (Second Tuesday—meeting sites vary). JANE ALLEN, Reichhold Chemicals, Inc., 814 Commerce Dr., Oakbrook, IL 60521.

GOLDEN GATE (Monday before third Wednesday—alternates between Francesco's in Oakland, CA, and Holiday Inn in S. San Franscisco). RICHARD COOPER, Synergistic Performance Corp., 5801 Christie Ave., #590, Emeryville, CA 94608.

HOUSTON (Second Wednesday—Hobby Airport Hilton, Houston, TX). TOM FITZGERALD, Monarch Paint Co., P.O. Box 55604, Houston, TX 77255.

KANSAS CITY (Second Thursday—Cascone's Restaurant, Kansas City, MO). LAWRENCE J. MURPHY, Tnemec Co. Inc., 123 W. 23rd Ave., N. Kansas City, MO 64116.

LOS ANGELES (Second Wednesday—Steven's Steakhouse, Commerce, CA). JOHN C. KULNANE, Ameritone Paint Corp., 18414 S. Santa Fe Ave., P.O. Box 190, Long Beach, CA 90801.

LOUISVILLE (Third Wednesday—Executive West Motor Hotel, Louisville, KY). WILLIAM LEIGHTNER, C.L. McGuire & Co., 8134 Newlagrange Rd., Louisville, KY 40222.

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

MONTREAL (First Wednesday—Le Bifthèque Steakhouse, Ville St. Laurent, Quebec). ESTHER ROULEAU MCCARTHY, Stochem Inc., 1455 32nd Ave., Lachine, Que. H8T 3J1, Canada.

NEW ENGLAND (Third Thursday—Sheraton Lexington Hotel, Lexington, MA, and other locations in Massachusetts and Rhode Island). CHARLES SHEARER, ICI Resins US, 730 Main St., Wilmington, MA 01887.

NEW YORK (Second Tuesday—Landmark II, East Rutherford, NJ). GEORGE M. AMRICH JR., Benjamin Moore & Co., 134 Lister Ave., Newark, NJ 07105.

NORTHWESTERN (Jax Cafe, Minneapolis, MN). MICHAEL GRIVNA, Hirshfield's Paint Mfg., Inc., 4450 Lyndale Ave., N., Minneapolis, MN 55412.

PACIFIC NORTHWEST (PORTLAND SECTION—Third Tuesday; PUGET SOUND SECTION—Third Wednesday; VANCOUVER SECTION— Third Thursday). JOHN WESTENDORF, Lipscomb Chemical Northwest, 2627 NW Nicolai, Portland, OR 97210.

PHILADELPHIA (Second Thursday—Williamson's Restaurant, GSB Bldg., Bala Cynwyd, PA). ROBERT D. THOMAS, M.A. Bruder & Sons, Inc., 52nd & Grays Ave., Philadelphia, PA 19143.

PIEDMONT (Third Wednesday—Ramada Inn Airport, Greensboro, NC). DALE BAKER, Kohl Marketing, Inc., 3859 Battleground Ave., Ste. 203, Greensboro, NC 27410.

PITTSBURGH (Second Monday—Montemurro's Restaurant, Sharpsburg, PA). MARK A. HARLEY JR., PPG Industries, Inc., 4325 Rosanna Dr., P.O. Box 9, Allison Park, PA 15101.

ROCKY MOUNTAIN (Monday following first Wednesday—Zangs Brewery, Denver, CO). CHRISTINE LESCAMELA, Luzenac America, Inc., 8985 E. Nichols Ave., Englewood CO 80112.

ST. LOUIS (Third Tuesday—Salad Bowl Cafeteria, St. Louis, MO). CHUCK REITTER, American Paint Journal Co., 2911 Washington Ave., St. Louis, MO 63103.

SOUTHERN (GULF COAST SECTION—third Thursday; CENTRAL FLORIDA SECTION—third Thursday after first Monday; ATLANTA SECTION—third Thursday; MEMPHIS SECTION—bimonthly on second Tuesday; and MIAMI SECTION—Tuesday prior to Central Florida Section). JEFF SHUBERT, Shubert Paints, Inc., 2157 Mountain Industrial Blvd., Tucker, GA 30084.

TORONTO (Second Monday—Cambridge Motor Hotel, Toronto). BOB C. NG, Hoechst Canada Inc., 100 Tempo Ave., Willowdale, Ont. M2H 2N8, Canada.

WESTERN NEW YORK (Third Tuesday—meeting sites vary). MARKO MARKOFF, 182 Farmingdale Rd., Cheektowaga, NY 14225.

CHICAGO .... FEB.

"VOC Compliant Coatings"

Rich Johnson, of Cargill, Inc., spoke about "NEW DEVELOPMENTS IN VOC COMPLIANT COATINGS FOR CONSUMER PAINTS." Mr. Johnson is a member of the Northwestern Society.

The spreading restrictions on VOCs in architectural and industrial maintenance coatings under the Clean Air Act were reviewed. According to the speaker, these restrictions have incited renewed interest in high solids and waterborne systems. Also, the need to meet hazardous air pollutant regulations has prompted new research into waterborne dispersions and water-reducible resins.

A chart displayed the industry usage of various types of resins and their normal end usage. The degree of difficulty of replacing their usage with alternative resins to achieve the required properties also was presented. According to Mr. Johnson, this might explain why the sales of higher solids alkyds increased 37% in the month following California's reduction in allowable VOCs in architectural paints, whereas the use of waterborne alternatives increased only by 10%. He explained that the customers had to be educated to brush out the alternative high solids alkyd products to achieve the same drying speed and spreading rate per gallon as the lower solids products.

In addition, Mr. Johnson said that most industrial maintenance coatings required newer products. Also, new water-reducible polyolefins are being successfully used as penetrating stain vehicles.

Mr. Johnson reviewed the progress in the design of new acrylic waterborne dispersion resins of much lower VOCs than water-reducible solutions.

CLIFFORD O. SCHWAHN, *Publicity*

CHICAGO .... MAR.

"Associative Thickeners"

Edward J. Schaller, of Rohm and Haas Company, was the meeting's technical speaker. His talk was entitled "SELECTING AND USING ASSOCIATIVE THICKENERS."

An associative thickener was defined as a high molecular weight multi-functional surfactant with hydrophobe modifiers at each end and sometimes within the molecule. Mr. Schaller said associative thickeners give a lot more thickening than expected from their molecular weight due to their associations with other materials, the latex, pigments, and other thickener molecules.

The three basic types of associative thickeners were described: HEUR, hydrophobe modified ethylene oxide urethanes; HASE, hydrophobe modified alkali soluble emulsions; and HMHEC,

hydrophobe modified hydroxy ethyl celluloses.

According to Mr. Schaller, because of the many types and grades of associative thickeners available, and the relative sensitivity of this type of thickener to other formulation components, it is often difficult to select the appropriate grade for the best balance of performance and cost. He stressed the need to consider the inherent properties of the various types in considering the best choices for the formulator for each of the formulas.

The advantages and disadvantages of the different associative thickeners were discussed.

In conclusion, Mr. Schaller stated that most people like combinations of thickeners for optimum properties rather than changing their basic surfactant system to accommodate the thickeners themselves.

CLIFFORD O. SCHWAHN, *Publicity*

CLEVELAND .... FEB.

"Responsible Care"

It was announced that the Society Board of Directors has approved a Scholarship Fund for the membership.

The technical presentation was delivered by Bernard Silverstein, of ARCO Chemical Company. His topic was "RESPONSIBLE CARE."

The speaker said the Responsible Care concept was developed by the Chemical Manufacturers Association to restore a positive image of the chemical industry in the public's eye.

Mr. Silverstein presented on overview of ARCO Chemical and its involvement with Responsible Care.

He explained the goals of Responsible Care including continuous improvement in health, safety, and the environment, which will be driven by the demands of the public.

Also, the principles of the Responsible Care program, safe operation guidelines at all plants, research, and resolving past waste problems were discussed.

According to Mr. Silverstein, a new trend of the program is developing whereby notifications of hazards are not just reported to regulatory agencies, but to the public, suppliers, industry, and the competition.

The speaker stated that the industry has to study the public's opinion of the chemical industry and determine where the public feels comfortable with the industry.

He emphasized that regulations are only a nuisance when bureaucracy plays a part. The chemical industry can reduce the necessity of government regulations by showing Responsible Care through self-evaluation, and therefore, self-regulation of the industry.

Employee safety, community awareness, emergency response, pollution prevention, process safety, and distribution of products—the six codes of the program were discussed.

Mr. Silverstein explained the codes of the program. More health and environmental personnel are being located in the plants to get more input for plant designs and more research on products. The community awareness code involves working with the community to prepare them for a possible emergency, to respond to their concerns by meeting with them to find out what the industry is doing right or wrong. Pollution prevention means a manufacturer should examine the processes involved in making a product and try to prevent or minimize the amount of waste generated versus looking at treatment of waste. Process safety is where the company examines the outer boundaries where products are stored, in addition to inside the manufacturing facility. The Distribution Safety Code promotes safety over the road.

According to the speaker, product stewardship is considered the umbrella of all the other codes, dictating that the company go outside to suppliers, customers, contractors, and tool manufacturers and work with them on products through their entire life cycle.

In conclusion, Mr. Silverstein stated that the Responsible Care program is international, and those involved in the chemical industry will become much more involved. He said self-regulation will be the trend of the future, because if the chemical industry does not clean up its act to meet the public's concern, the industry will gradually fade out of existence.
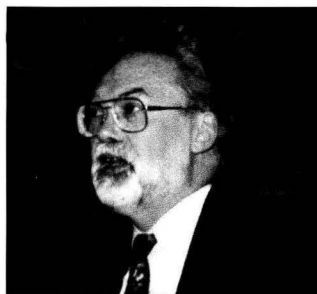
CONSTANCE F. WILLIAMS, *Secretary*

LOS ANGELES .... MAR.

"Silicones"

The Society Past-Presidents in attendance were greeted with a "formal" introduction. In addition, each was presented with a complimentary three-day pass to the Western Coatings Societies' Symposium and Show.

The 14 honorees included: Leonard S. Feldman, Society Honorary Member (1960-61); Alan U. Hershey, retired (1961-62); Robert A. McNeill, RAM Consulting (1974-75); Kenneth J. O'Morrow, retired (1975-76); Robert F. Koperek, retired (1977-78); Fred Croad, retired (1978-79); Albert Seneker, retired (1979-80); Donald I. Jordan, Society Honorary Member (1980-81); Jan Van Zelm, BYK-Chemie USA (1981-82); Romer E. Johnson, Dorsett & Jackson (1982-83); Michael Gildon, retired (1985-86); Ray DiMaio, of Kop-Coat Inc. (1987-88); Melinda Rutledge, of RHEOX, Inc. (1989-90); and James D. Hall, of Sinclair Paint Company (1991-92).

MARCH TECHNICAL SPEAKER—Edward J. Schaller, of Rohm and Haas Company, explains associative thickeners to the members of the Chicago Society

It was noted during the presentation of the Environmental Committee Report that the South Coast Air Quality Management District has proposed Rule 2013 for "Measuring and Reporting Emissions of Reactive Organic Compounds (ROC)." It was announced that many changes have been made from the original version of the rule.

In addition, bills from the State Board of Equalization regarding the Childhood Lead Poisoning Prevention Fee for Architectural Coatings distributors have arrived. A $.02388 fee per gallon based on 1991 production is the amount arrived at.

Matt Dustin, of the California Paint Council, suggests preparing the form and check but not sending it immediately. The Chemical Manufacturers Association is attempting to file legal action to block the billing on two counts. First, the fee appears to be a tax. Second, the regulation fails to assess other lead sources. The State Department of Health Services definition of an architectural coating appears to be different from that of the South Coast Air Quality Management District.

Also, Bert Osen, of Fine Line Paint Corporation, advised the membership to investigate the application of the "fee" for the Childhood Lead Poisoning Prevention Fee for Architectural Coatings. He commented on the fact that the fee does not apply to lacquers, varnishes, etc. Mr. Osen said that product types manufactured should be clarified, because some of the products may not have to be reported for the fee.

New York Society member Edward W. Orr, of BYK-Chemie USA, was the meeting's technical speaker. His topic was "SILICONES IN THE COATINGS INDUSTRY: THE INFLUENCE OF CHEMICAL STRUCTURE UPON PROPERTIES."

The speaker discussed silicone additives and their use in coatings. He stated that with the proper silicone additive, a coating can be made to coat a grease coated steel.

Mr. Orr explained that a new structure has been developed to provide very low surface tensions to match those of fluoro type products, minus the side effects of foam.

However, he said that silicones are thought to cause problems with intercoat adhesion. It was noted that the longer the alkyl group, the less influence on surface tension reduction.

The speaker stated that the silicone molecule can be modified by changing the molecular weight. He said the molecular weight must correspond to the molecular weight of the relatively low or moderately low areas.

The general structures of the polysiloxane molecule were reviewed.

In conclusion, Mr. Orr stated that many surface defects are caused by differences in surface tension. By proper selection of silicone additives, many of these defects can be eliminated.

Q. What is the fastest way to find the optimum amount of silicone additive?

A. Match the surface tension of the components of the formulation with the silicone additive and avoid use of silicone additives which contain pendant oxygen groups.

Q. Are silicone additives as effective in water as they are in solvent coatings?

A. Yes. The mechanism of matching the effect of fluoro type compounds actually is more effective in water than solvent coatings.

JOHN C. KULNANE, *Secretary*

MONTREAL .... FEB.

"Waterbased Coatings: Low VOCs" Symposium

The Society's Annual Symposium was conducted during this month's meeting. The theme of this year's event was "WATERBASED COATINGS: LOW VOCs."

The first speaker was Northwestern Society member Rich Johnson, of Cargill, Inc. His topic was "WATERBASED TECHNOLOGIES FOR LOWER VOC."

Mr. Johnson presented an outline of the driving forces behind the new waterborne technologies. According to a coatings industry survey, 78% of the respondents stated that the primary reason for this new waterborne technology was legislative regulations, including the Title I Clean Air Act, Title III 190 Hazardous Air Pollutants, and the Environmental Protection Agency's 33/50 project.

According to the speaker, the ultimate result of this legislation will be the elimination of many raw materials now used, and eventually zero VOC limits.

Mr. Johnson reviewed the major technologies available.

In conclusion, the speaker stated that major changes are taking place and, to survive, successful raw material suppliers and coating producers will change. He said that many technologies have been developed in the last 5-10 years which "common wisdom at the time thought impossible." Mr. Johnson assured the membership that this change will continue to occur.

Q. Will a uniform standard be established in the U.S. for VOC requirements?

A. Yes, efforts are underway between industry and both national and state government bodies to establish a minimum uniform requirement. However, individual states will retain the right to set more stringent norms (i.e., California probably will still lead the way).

The next speaker was Krishan C. Sehgal, of Union Carbide Chemicals & Plastics Company Inc., whose talk was entitled, "SOLVING PRACTICAL ADHESION PROBLEMS IN TRADE PAINTS WITH FUNDAMENTAL STUDIES."

The various adhesion models and common methods used in the coatings industry were reviewed.

Dr. Krishan showed that adhesion failure can be divided into two types: interfacial, between coating and substrate; and bulk contribution, within the coating itself.

According to the speaker, the testing methods used presently are both destructive and nondestructive (x-ray) types. These methods measure different modes of failure (or potential failure). He noted that test results do not always correlate with the expectations of the coating, or from one test method to the next.

Dr. Krishan stated that most measurements are defined not by the formation of the bond, but by the breaking of the bond. To make full use of the test results, he said it is important to know the mode of failure.

Wettability studies and SEM results used to characterize the surface energy, composition, and morphology of naturally weathered surfaces were presented.

In conclusion, Dr. Sehgal explained that a combination of test results (i.e., tape pull, Instron, wet scrub, shower stall, 180 peel test) should be conducted to best compare adhesion performance of latex coatings. He stated that in many cases one test gave misleading results. Also, the morphology of the substrate (chalk paint), including roughness, surface characteristics, and the cross section of the substrate (chalk layer) are important.

Kent Wood, of Rohm and Haas Company, gave a talk on "WATERBASED COATING SYSTEMS FOR THE WOOD FINISHING INDUSTRY."

The speaker said that traditionally, nitrocellulose lacquers have been used by the furniture industry, resulting in over a 250,000,000 lbs of solvents (VOC) being release annually by this industry. Mr. Wood stated that waterborne resins offer lower VOCs and improved safety for the furniture industry.

Film properties, latex film formation, and UV curable coating systems were discussed.

In conclusion, Mr. Wood said molecular weights will increase, along with good early film properties, through improved latex design. Also, VOC will continue to drop, and formulation adjustments will continue to be critical for success.

Q. How good is the color retention of these waterborne coatings?

A. Very good, thermoplastics retain initial color very well.

Q. Has the grain raising problem been solved?

A. Not really, but it can be minimized by controlling the pH. When the pH is over 8, it increases; below 7 is the best. Hybrid systems (sealers) are perhaps still the best for difficult wood substrates.

The Society's Annual Symposium was concluded with a presentation by New York Society member Edward W. Orr, of BYK-Chemie USA. His topic was "ADDITIVE US-

LOS ANGELES SOCIETY PAST-PRESIDENTS—Attending the March Society meeting are, standing (from left): Fred Croad, Jan Van Zelm, Melinda Rutledge, Romer E. Johnson, Leonard S. Feldman, and Ray DiMaio. Seated: Alan U. Hershey and Michael Gildon

AGE IN ENVIRONMENTALLY FRIENDLY COATINGS."

Mr. Orr explained that the trend towards environmentally acceptable coatings poses special challenges to additive suppliers, including: flow, rheology, catalysis, substrate wetting, pigment wetting, and dispersing.

On overview of new additive technologies, with emphasis on wetting and dispersing, surface flow and leveling, and defoaming, was presented.

The speaker discussed the proper usage of silicone additives (flow and surface control) for environmental coatings. He explained that the increased understandability of silicone chemistry has allowed manufacturers to develop new classes of silicone compounds that are "trouble free."

In conclusion, Mr. Orr stated that environmentally acceptable coatings demand higher performance, so new additive structures have evolved to meet the new demands of advanced coatings components and application technologies.

Q. Are surfactants the primary cause of foaming problems?

A. Yes, but if the system does not have enough surfactants, the shear stability will be poor.

JASON G. HART

NORTHWESTERN .... FEB.

Spouses' Night

Environmental Committee Chairman Mark Uglem, of Hirshfield's Paint Mfg., Inc., reported on the activities of the Paint Council. Currently there are two items of concern: Bill H.F. 65, regarding recycling and packaging, which could have great ramifications to the industry; and proposed Environmental Protection Agency regulations for above ground storage tanks.

Technical Committee Chairman Edward Ferlauto, of The Valspar Corporation, gave an update on the Committee's activities, including a paper he is writing regarding lead abatement. Other active projects include work on acid rain, formaldehyde, and corrosion test methods.

The first speaker was Juergen H. Braun, of DuPont Chemicals, who spoke on "GLOSS OF PAINT FILMS AND THE MECHANISM OF PIGMENT INVOLVEMENT."

The speaker defined gloss as the intensity of reflected light. The optics of gloss include the physics of reflected light and the measurement of intensity. Also discussed was the psychology of gloss.

Dr. Braun said the drying process of a paint film is divided into two phases. During the wetter stage of drying, the surface of the film is smooth down to a molecule level and appears wet. As the film dries, a Surface

MORE LOS ANGELES SOCIETY PAST-PRESIDENTS—Also in attendance at the March Society meeting are, standing (from left): Robert A. McNeill and Albert Seneker. Seated: Donald I. Jordan, Robert F. Koperek, James D. Hall, and Kenneth J. O'Morrow

Critical Point is reached. According to the speaker, this is when the pigment structure forces are equal to the surface tension forces. Beyond this point the pigment structure forces become more dominant.

The second speaker was Doris Brashear, of Hirshfield's Paint Mfg., Inc. Ms. Brashear discussed "THE LATEST IN FABRIC AND WINDOW TREATMENTS."

The speaker demonstrated many new products. Current trends in window coverings, including shear/blind combinations, wood blinds, custom blinds, and headers, were reviewed.

In conclusion, Ms. Brashear stated that blinds can help conserve energy better than conventional drapery, even when the blinds are partially open.

MICHAEL GRIVNA, *Secretary*

PHILADELPHIA .... FEB.

"Adhesion to Chalky Surfaces"

The meeting featured a visit from FSCT President-Elect John A. Lanning, of Courtaulds Coatings, Inc., and staff members Michael Bell, Director of Educational Services, and Patricia Viola, Director of Publications.

Mr. Lanning spoke first and discussed the importance of quality improvement and how some of the FSCT's many programs and training aids can help contribute toward quality improvement.

Also, he talked about membership participation and membership growth in the FSCT.

In conclusion, Mr. Lanning outlined the cooperative efforts of the FSCT and NPCA.

Mr. Bell reviewed the attendance statistics of the 1992 Annual Meeting and Paint Industries' Show (AM&PS), held in Chicago, IL, and talked about future AM&PS sites.

Also discussed were Spring Week 1993, scheduled for May 16-19, in Houston, TX; the Coatings Industry Education Fund; and the FSCT budget.

Mr. Bell talked about the quantity and quality of the many FSCT publications, and reviewed the coatings MSDS system on CD-ROM project, which will provide a compilation of the Material Safety Data Sheets used by the coatings industry on new CD-ROM technology.

The technical speaker was Krishan C. Sehgal, of Union Carbide Chemicals and Plastics Company, Inc., who spoke on "ADHESION TO WEATHERED CHALKY SURFACES."

Several models of adhesion and their respective limits and benefits were discussed. Dr. Sehgal suggested that an adequate treatment of adhesion must deal with both the surface energy component and the viscoelastic component of adhesion. He stressed that the test data always should include the mode of failure as well as the data for the point of failure. It was noted that the mode of failure will indicate the direction of greater interest when seeking improvements.

The speaker commented that the characterization of the surface is very important when studying the coating-substrate interface and its integrity.

In conclusion, Dr. Sehgal said good adhesion properties depend on a proper balance of the surface energy and the viscoelastic contributions.

ROBERT D. THOMAS, *Secretary*

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Elections

BALTIMORE

Active

Frick, Jan P.—Farboil Co., Baltimore, MD.
Timmins, Jesse S.—W.R. Grace & Co., Baltimore.

Associate

Akers, Woodrow J.—Farboil Co., Baltimore, MD.
Boldt, Paul W.—E.W. Kaufmann Co., Ellicott City, MD.
Brophy, Terrence K.—The Thornley, Wilmington, DE.
Crowley, Colin D.—ChemCentral, Baltimore.

BIRMINGHAM

Active

Price, Andy—PermaBond, Eastleigh, Hampshire.
Yuen, Shu Wah—Manfield Coatings Co., Ltd., Fotan Shatzn, Hong Kong.

Associate

Costin, Colin—Synthopol Chemie, Rush Green, Romford, Essex.
Martin, John C.—Inchema, Orpington, Kent.
McGregor, Ronald J.—Siber Hegner Ltd., Beckenham, Kent.

CDIC

Active

Chihara, Kohji Y.—Gencorp., Wabash, IN.
Heater, Kenneth J.—Battelle Columbus Labs., Columbus, OH.
Humphrey, Burnice L.—Patriot Paint Co., Inc., Portland, IN.
McDonald, William F.—Battelle Columbus Labs., Columbus.
Metz, Barbara A.—Battelle Columbus Labs., Columbus.
Mitchell, Ben G.—Akzo Coatings Inc., Columbus.
Parsons, Alice B.—Battelle Columbus Labs., Columbus.
Russell, Robert E. III—Battelle Columbus Labs., Columbus.
Spangler, J. Michael—Battelle Columbus Labs., Columbus.
Wojtowicz, Jadwiga—Akzo Coatings Inc., Columbus.

Associate

O'Connor, J. Brian—McWhorter Inc., Carpentersville, IL.

CLEVELAND

Active

Allen, Karen S.—Lubrizol Corp., Wickliffe, OH.
Bourne, Robert G.—American Colors Inc., Sandusky, OH.
Brannon, Selby M.—Plasticolors, Inc., Ashtabulam, OH.
Davies, Oswald G.—The Sherwin-Williams Co., Cleveland, OH.
Raghavan, Vaikunt—Lemora Coatings, Cleveland.

Associate

Hand, Patrick F.—CPS Chemical, Strongsville, OH.

Educator/Student

Payer, Joe H.—Case Western Reserve University, Cleveland, OH.

Retired

Robinson, Peter V.—Medina, OH.

GOLDEN GATE

Active

Phillips, Dave S.—P & S Sales, Inc., Hayward, CA.
Shratter, Alan E.—Kelly-Moore Paint Co., Inc., San Carlos, CA.

Associate

Kercher, Doug G.—Harcros Pigments, Oakley, CA.
Witt, Monica E.—SCM Chemicals, Inc., Santa Fe Springs, CA.

LOS ANGELES

Active

Ellis, George E. Jr.—Ellis Engineering & Management Systems, San Pedro, CA.
Lorenz, Arthur W.—Sinclair Paint Co., Los Angeles, CA.
Schule, Martin—Ink Systems, Inc., Bell, CA.
Thompson, Carl V.—Superior Industries, Van Nuys, CA.

Associate

Gray, Daniel A. Jr.—John K. Bice Co., Inc., Los Angeles, CA.
Hutchins, Ward—P.T. Hutchins Co., Ltd., Los Angeles.
Roller, David—Alpha Consultants, Upland, CA.

Sherman, Roger F. Jr.—Sonoco Fibre Drum, Santa Fe Springs, CA.

MONTREAL

Active

Ades, David—Protech Chemicals Ltd., St. Laurent, Que.
Chantal, Jacques—Sico Inc., Outremont, Que.
Laurent, Edouard El.—Pemla Inc., St. Leonard, Que.

Associate

Pierre, Yves—Inortech Chimie Inc., Laval, Que.
Primeau, Guy D.—R.M. Ferguson & Co., Cornwall, Ont.

NEW YORK

Active

Calienni, James J.—William Zinsser Co., Somerset, NJ.
Kapilow, Lorraine—CIBA-GEIGY Corp., Hawthorne, NY.
Kuusisto, Eeva L.—Troy Corp., Newark, NJ.
Maniar, Kash N.—Insl/X Products Corp., Stony Point, NY.
Randolph, John H.—Randolph Products Co., Carlstadt, NJ.
Schaeufele, Peter J.—Troy Corp., Newark.
Swan, Archie G. Sr.—Hartin Paint & Filler, Carlstadt.

Associate

Aromando, Donald A.—Schweizerhall, Inc., Piscataway, NJ.
Chelak, William Jr.—Buckman Laboratories, Inc., Philadelphia, PA.
Happe, George W.—Mineral Pigments Co., Pompton Plains, NJ.
Morlino, Nancy M.—Lonza Inc., Fair Lawn, NJ.
Saul, Frank T.—Seegott Inc., West Orange, NJ.
Stotler, Kristin M.—Praxair Inc., Moorestown, NJ.

SITUATIONS WANTED

People

The appointment of **Maria Repici** as Marketing Services Manager has been announced by the Instrument Systems Division of Minolta Corp., Ramsey, NJ. She will manage and supervise all advertising, public relations, and trade show activities for the Instrument Systems Division. Additional responsibilities include the development and implementation of marketing programs in support of sales and the division's objectives.

M. Repici

Two new sales representatives have been appointed by Lomas International, Macon, GA. **James Harrell** will represent Lomas in the mid-central section of the U.S. and will work out of his Memphis, TN, office. Previously, he held a Senior Sales Management position with a Memphis-based specialty chemical supplier. He replaces former Lomas salesman for this region, **Jim Hunt,** who has been transferred to Tampa for sales work in northern and western Florida.

In addition, **Mandy Thomas,** a longtime Customer Service Representative at the Macon headquarters, will relocate to the regional office in Charlotte, NC, and be responsible for sales in North and South Carolina. **Lisa Lusk** fills the vacant customer service position left by Ms. Thomas.

Albert P. Stroucken has been named Executive Vice President of the Miles Industrial Chemicals Div., Pittsburgh, PA. Previously, Mr. Stroucken was the Vice President of the Pigments, Enamels and Ceramics business. He succeeds **Richard L. White,** who has been named Executive Vice President of the Miles Organic Products Division and Polysar Rubber Division, as well as President and CEO of the Polysar Rubber Corporation of Canada.

ICI Surfactants, Wilmington, DE, has appointed **Ronald E. McCoy** as Director of Production for ICI Surfactants in North America. He is responsible for four manufacturing sites in the U.S. and Canada. Prior to his recent appointment, Mr. McCoy served as Director of Corporate Safety for ICI Americas Inc. He is based at ICI's Atlas Point Site in New Castle, DE. Mr. McCoy joined ICI in 1988.

The Polymers Div. of Cook Composites and Polymers has announced organizational changes due to the acquisition of Cray Valley Products, Inc.

Eric Percy has been appointed to Field Sales Technical Director. He will provide technical support to CCP's field sales people, agents, and distributors.

Michael Foster has been promoted to Eastern Regional Sales Manager. He will be responsible for the division's sales for the eastern United States. Prior to this promotion, Mr. Foster was Marketing Manager.

Following a change in CCP's sales structure from a national to an eastern/western regional concept, **Edwin Bigelow** was named Western Regional Sales Manager. Previously, Mr. Bigelow was National Sales Manager.

John Bradley has been appointed Marketing Manager. He will retain responsibility for sales outside the U.S., and will coordinate toll manufacturing. Mr. Bradley previously held the position of National Accounts Manager. He is a member of the Kansas City Society.

Patricia Fricano was named Product Manager, and will oversee the coatings resins product line. Prior to her appointment, Ms. Fricano was Product Manager for CCP's Composites Division.

Buckman Laboratories International, Inc., Memphis, TN, has named **Mohan D. Karve** to the newly created position of Vice President—Corporate Alliances. He is to develop corporate partnerships on a global basis. Recently, Mr. Karve was instrumental in creating a Buckman associate company in Tokyo.

Troy Corp., East Hanover, NJ, has appointed **Kurt L. Hansen** as Manager, Microbiology within Troy's Applied Research and Technical Service Group. Mr. Hansen's responsibilities include developing new methods for testing the effectiveness of Troy biocides in a wide range of formulation and environmental conditions, and helping customers test specific formulations. Before joining Troy, he directed corporate microbiological research.

Witco Corp., New York, NY, has announced the formation of an international business development department. **David A. Kreckman** has been named Director of this department, which was created to develop new business for Witco's specialty chemical product lines concentrating in Asia and Latin America.

Obituary

Elias Singer, retired President of Troy Corp., East Hanover, NJ, died on January 23, 1993, in Debray Beach, FL. He was 81.

Mr. Singer received a B.S. Degree from the City College of New York. Shortly after receiving an M.S. Degree in chemistry in 1933 from Columbia University, Mr. Singer began working as a Chemist for the Joachim Research Laboratory. He joined the D.H. Litter Co. as a Chemist in the company's technical service laboratory in 1933. Mr. Singer was named the laboratory's Research Director in 1938 and Technical Director in 1947.

In 1952, Mr. Singer began his long association with Troy Chemical Corp. as the company's President and Technical Director. He remained the company's President until 1979, when the company was sold to its current owners. Mr. Singer continued working there as a consultant until his retirement in 1988.

In 1946, Mr. Singer began 19 years of service as an instructor of surface technology at New York University. In addition, he was an author of numerous technical articles on coatings.

As a member of the New York Society for Coatings Technology, Mr. Singer served on the Educational and Historical Committees. He was also a member of the National Paint and Coatings Association and the American Chemical Society.

In appreciation and recognition of Mr. Singer's long service to Troy, the company created the annual Elias Singer Award, which is given to authors of outstanding technical papers at the annual Waterborne, Higher-Solids and Powder Coatings Symposium in New Orleans.

A 50-year member of the Federation, Mr. Singer was also the 1982 recipient of the New York Society's PaVac Award.

A native of Zaleschicki, Austria-Hungary, Mr. Singer is survived by his wife, Sylvia; two daughters, Carol and Marion; and a son, Fred.

Technical Program and Exhibition Focus on VOC; Highlight 21st Western Coatings Societies' Event

On March 23-25, the Western Coatings Societies held their 21st Biennial Symposium and Show, at the Disneyland Hotel & Convention Center, in Anaheim, CA. Approximately 2,200 people attended the event which focused on the theme, "Visions-Opportunities-Challenges."

The Los Angeles Society hosted the conference and exhibition, and was supported by the Golden Gate, Pacific Northwest, and Rocky Mountain Societies.

Exemplifying the theme of the event were 24 technical presentations, centering around VOC regulations, formulations, and new technologies, and a trade show featuring 110 exhibitors in 15,400-sq. ft. of exhibit space.

The "Visions-Opportunities-Challenges" theme was best stated during the technical sessions. What follows is a brief synopsis of each speaker's presentation.

"NEW CATALYSTS FOR HIGH SOLIDS COATINGS"—Robert Middlemiss, of OMG Group, Inc. Mr. Middlemiss spoke on the optimum drier combinations for many new high-solids resins. Significant dry time reductions, and cost savings were discussed. In conclusion, he explained drier promoted polymerization, use of chelating agents to enhance dry, and driers for waterborne coatings.

"EXTERIOR STUDIES OF EXTERIOR LATEX HOUSE PAINTS CONTAINING KAOLIN CLAY PIGMENTS"—Thad T. Broome, of J.M. Huber Co. Mr. Broome reviewed the typical physical characteristics of kaolin clay pigments. His presentation focused on data from a four-year study which described the formulation, laboratory tests, and exposure tests performed on various kaolin clays. The exterior studies which were conducted compared scrub resistance with exterior durability. Characteristics such as opacity, brightness, and oil absorption were examined.

This event was dedicated to the "beautiful memory" of Geneva Wells, who died last year. Ms. Wells was an integral member of this symposium and show. Four years ago, she accepted the position of General Chairman and set the foundation for this successful event.

"PLANT HOUSEKEEPING CONSIDERATIONS WITH NONMERCURIAL BIOCIDES"—Jeffrey S. Hinkle, of Hüls America Inc. The speaker focused on the causes of spoilage from a microbiological standpoint. Describing the systems and components of paint formulations which are most susceptible to attack, Mr. Hinkle discussed possible sources of contamination in the plant/manufacturing environment. The effects of spoilage were explained.

"APPROACH TO FORMULATION OF ARCHITECTURAL & MAINTENANCE COATINGS"—John Ballard, of Burgess Pigment Co. According to Mr. Ballard, three factors which must be considered when formulating coatings are cost, hiding, and touch-up. He detailed factors which influence touch-up and those which affect mud cracking. Investigated were 10 key architectural maintenance large volume products, which in combination contribute significantly to profitability. Specific raw materials were evaluated.

"NPCA UPDATE"—J. Andrew Doyle, Executive Director, National Paint & Coatings Association; Sandra Skommesa, President, Southern California Paint & Coatings Association; and Mathew Dustin, Director, California Paint Council. National and local issues affecting the coatings industry were the focus of this session.

"OPTIMIZING LIGHTFASTNESS OF ORGANIC PIGMENTS THROUGH PARTICLE SIZE CONTROL"—Romesh Kumar, of Hoechst Celanese Corp. Dr. Kumar reviewed the influence of particle size distribution on lightfastness of organic pigments in various resin systems. Physical parameters influencing lightfastness, including crystal structure and specific surface area, were detailed.

"GLOSS: FACTORS INFLUENCING GLOSS DEVELOPMENT AND FORMULATING GUIDELINES TO ACHIEVE GLOSS IN PAINT"—Rebecca Craft-Tulloch, of E.I. du Pont de Nemours & Co. The speaker began with a discussion of surface roughness and some new techniques for measuring gloss. Paint properties that influence gloss development include flow and leveling, particle size dispersion and concentration, and film formation. Ms. Craft-Tulloch emphasized achieving the proper set of physical properties of a coating to create the desired appearance. She stated that in some coatings formulations the actual achievement of gloss is not a primary concern, while in others gloss must be high and is of primary importance not only in the initial dried film, but over an extended period of time of exposure to the elements.

"ION EXCHANGED ALTERNATIVES TO CHROMATE FOR CORROSION INHIBITION"—Michael Maule, of W.R. Grace Co. The properties and behavior of calcium exchanged silica gel were covered. Mr. Maule described the anticorrosive results obtained in comparison to chromate pigment(s). In addition, practical formulating tips for the paint chemist were provided. In conclusion, the speaker reviewed incorporation techniques, loading levels, and the benefits of synergistic pigments.
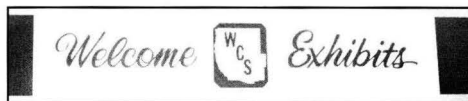
"HVLP SPRAY GUN TECHNOLOGY"—Glen L. Muir, of Graco Inc. The basic forms of atomization of airspray/high volume low pressure and airless were presented. Performance parameters of all types of tools and some of the benefits and problems of each method were reviewed.

"WATERBORNE EPOXY RESIN EMULSION"—Marcel Gaschke, of Ciba-Geigy Corp. The speaker described a stable aqueous emulsion free of organic solvents, based on a multifunctional epoxy resin of a relatively high molecular weight. Detailed were the stability of the emulsion, its application, and its per-

formance in ambient temperature systems cured with a polyamidoamine. Dr. Gaschke explained the properties of the coatings and the chemical resistance of the cured films.

"GRAFFITI ABATEMENT AND PREVENTION HANDBOOK"—V.C. "Bud" Jenkins, Consultant. Mr. Jenkins referenced a handbook prepared by the L.A. Society Technical Committee on ways to best fight particular graffiti problems. He said the handbook will be sent to cities throughout the world. In addition, experts in the graffiti eradication field were present to give their views on how the paint industry can help fight graffiti. The pros and

cons of many existing techniques for fighting graffiti were discussed.

"HIGH SOLIDS SOLVENT FREE BAKING ENAMELS"—Richard Farmer, of Cargill Resin Products Div. The objectives of high solids coatings—to reduce VOC and retain coating performance requirements—were covered. Explained was the feasibility of the application of higher viscosity, solvent-free formulations based on a polyester/melamine system. The speaker highlighted advantages and disadvantages of conventional polyesters and current high solid polyesters, and predicted the capabilities of the next generation of high solids polyesters.

"THE CAL POLY POLYMERS AND COATINGS PROGRAM: FROM DREAM TO REALITY"—Dane Jones, James D. Westover, and Max T. Wills, of California Polytechnic State University. Discussed was the Chemistry Department's undergraduate program in Polymers and Coatings. Projects undertaken by the program include: the California Paint Recycling Program, the use of glass as a filler in latex coatings, the effect of opaque polymers on color of latex coatings, the use of Raman scattering to analyze latex coatings, and development and testing of new coatings formulations.

"OPTICAL BRIGHTENERS—MORE THAN JUST CLEAN LAUNDRY"—Timothy Geran, of Ciba-Geigy Corp. The structure activity relationships of fluorescent whitening agents, including both waterborne and solvent soluble chemistries were detailed. Mr. Geran provided an introductory review of the photochemical physics.

"EL-RAP"—Representatives from the Environmental, Legislative, and Regulatory Advocacy Program of the Southern California Paint & Coatings Association discussed programs recommended by the EL RAP Action Committee.

"AQUEOUS SUSPENSIONS OF CELLULOSIC THICKENERS: FUNCTION AND MECHANISM IN WATER BASED COATINGS"—C.W. Vanderslice, H.F. Haag, and C.L. Burdick, of Aqualon Co. According to Mr. Vanderslice, recent advances in technology have allowed both conventional and newer hydrophobically-modified types of hydroethyl cellulose (HEC) to be produced in aqueous suspension form. These stable liquid suspensions do not contribute to VOC since organic diluents are not required to prevent polymer hydration during storage. Explained were the processing and production advantages of using these types of HEC during paint manufacture.

"SYSTEM DESIGN CONSIDERATIONS FOR POWDER COATINGS OPERATIONS"—Michael W. Cravens, of Powdercoats Services. The powder coating process as it relates to system design considerations was reviewed. Information ranging from the most basic batch systems, to the more elaborate semi-automated conveyorized systems, was covered. In addition, the speaker discussed plant layout, racking system design, pretreatment and rinse water systems, application and powder recovery equipment, oven design, and fire protection.

"ULTRAFINE TITANIUM DIOXIDE: ITS PROPERTIES AND APPLICATIONS IN COATINGS"—John Clayton, of Tioxide Specialties, Inc. The speaker explained the differences between ultrafine titanium dioxide and traditional TiO_2. By drastically reducing its crystal size and controlled application of inorganic coatings, one can obtain a material that has dramatically different properties than pigmentary TiO_2. Mr. Clayton stated that ultrafine titanium dioxide is being used to produce pearlescent metallic finishes and displays potential as a transparent UV absorber, particularly in wood finishes.

"INVESTIGATION OF LONG-TERM YELLOWING OF ARCHITECTURAL ENAMELS AS A FUNCTION OF COATING COMPOSITION"—Jeffrey H. Danneman and Arthur C. Smith, of Reichhold Chemicals, Inc. An investigation of the film yellowing phenomenon of high solids air dry alkyds was presented. The influence of drier combination, additives, storage conditions, and resin composition on long-term yellowing of architectural enamels was described.

"THE UTILITY OF GLYCOL ETHERS AS COSOLVENTS IN WATER-BASED COATINGS"—Tom D. Boyce, Doug K. Pollock, and Randy LaTulip, of The Dow Chemical Co. Reviewed was the accelerating trend of the industry to move from conventional solvent-based coatings systems to water-based (emulsions and dispersions) coatings systems. It was pointed out that paint formulators are working towards developing systems that comply with continually changing, more stringent regulations, while not sacrificing coating quality.

"DEVELOPMENTS IN THE EUROPEAN AQUEOUS LOW VOC ARCHITECTURAL COATINGS MARKET"—David Sykes, of Rohm and Haas Co. Concerns about environmental protection and health and safety caused by solvent emissions during paint application exist. These issues were discussed on a country-by-country basis, with details of the relevant government legislation or voluntary agreements in place.

"RISK MANAGEMENT IN PAINT BIOCIDE USE"—Judith Ross and Angela Downey, of Rohm and Haas Co. Reviewed were the safety advantages of the nonmetallic biocides currently used in the paint industry. Safe handling procedures for these concentrated biocides in the work place were examined, including improved delivery systems. The environmental fate profile, another aspect of modern biocides, and formaldehyde release, a property exhibited by some in-can preservatives, were discussed. Ms. Ross recommended training employees to understand MSDSs; improving plant hygiene techniques; and using protective clothing and equipment as recommended by biocide suppliers.

"ECONOMIC OUTLOOK FOR 1993-1994"—Richard Stuckey, of E.I. du Pont de Nemours & Company. The speaker predicted moderate growth for 1993 and 1994, and offered a cautiously optimistic outlook for the future of the industry.

In addition, the Industry Luncheon had Darryl F. Gates, retired Chief of Police of the Los Angeles Police Department, as its guest speaker. Chief Gates addressed the luncheon crowd on "How to Fight Crime in the Streets."

The three-day event was concluded with a Banquet and Show, featuring entertainment by Danny Gans, Marie Osmond, and the Joe Diamond Orchestra.

The following members of the L.A. Society were responsible for the success of the 21st Biennial Western Coatings Societies' Symposium and Show: General Chairman—Donald I. Jordan, of Cargill, Inc.; Co-Chairmen: Melinda Rutledge, of RHEOX, Inc., and Sandra Dickinson, of Synergistic Performance Corp.; Advisor—Andrew Ellis, of KRONOS, Inc.; Entertainment—Bruce Cotton, of Pluess-Staufer; Exhibits Committee Chairman—William Zimmerman, of Sinclair Paint Company; Publications—Ronald R. Elliott, of J.R. Elliott, Inc.; Publicity—Samuel

A. Rumfola, of TCR Industries; Registration Committee Chairman—James Hall, of Sinclair Paint Co.; Secretary—Richard Sutherland II, of E.T. Horn Co.; Technical Committee Chairman—John Kulnane, of Ameritone Paint Corp.; and Treasurer—James Calkin, of E.T. Horn Company.

Piedmont Society Mini-Symposium and Trade Show Focuses on "Compliance: Plain and Simple"

The Piedmont Society for Coatings Technology sponsored a Mini-Technical Symposium and Exhibit Trade Show geared to the theme, "Compliance: Plain and Simple," on March 10, at the Holiday Inn, in High Point, NC.

The Technical Symposium consisted of four presentations, with each session being attended by approximately 70 people. The following topics were discussed:

"Solvents and the Clean Air Act of 1990"—Tom Boyce, of Dow Chemical Company;

"Low-VOC Waterborne Coatings for Wood Based on Nitrocellulose-Acrylic Latex"—Harold Haag, of Aqualon Company;

"Compliance and Performance of Waterborne UV Curable Polymer Resins for the Wood Finishing Industry"—Paul Stenson, of Zeneca Resins; and

"Compliant Waterborne Acrylic Latexes for Wood Coatings"—Linda Smith, of Rohm and Haas Company.

The Exhibit/Trade Show featured a total of 33 exhibitors. The following vendors attracted nearly 200 people to the Show: Aqualon Company; Buckman Laboratories, Inc.; Burks, Inc.; BYK-Chemie USA; BYK-Gardner; Chemarco, Inc.; Ciba-Geigy Corporation; Cytec Industries; Degussa Corporation; Du Pont Company; Engelhard Corporation; Epworth; Etna Products; Florida Steel Drum Company; W.R. Grace & Company; HeucoTech Ltd.; Hoover Color Corporation; J.M. Huber Corporation; Hüls America, Inc.; King Industries, Inc.; Kohl Marketing, Inc.; Lomas International; McCullough & Benton, Inc.; Mineral Pigments Company; Penn Color, Inc.; RHEOX, Inc.; Rohm and Haas Company; Seegott, Inc.; Silberline Manufacturing Company, Inc.; Tego Chemie Service; U.S. Borax; Van Horn, Metz & Company, Inc.; and Zeneca Resins.

Highlighting the Piedmont Paint and Coatings Association sponsored luncheon was a presentation by Daniel Cox, of Cargill, Inc. Mr. Cox addressed a luncheon crowd of 150 people on the "Status and Future of the Coatings Industry."

PIEDMONT SYMPOSIUM—Technical sessions on "Compliance" are well attended

Golden Gate Society Manufacturing Committee To Host "Process and Compliance" Conference

The Manufacturing Committee of the Golden Gate Society for Coatings Technology will sponsor a conference on "Process and Compliance," on June 14, at the Ramada Inn (formerly Holiday Inn), in South San Francisco, CA.

The all-day program was designed to help manufacturers and paint contractors understand and help each other in dealing with both community and government officials. The conference will feature what standards are being applied for quality control, both locally and internationally.

The following topics are scheduled for presentation:

"International Standard Organization (ISO)-9000 Series"—speaker to be announced, Clorox Company;

Piedmont Society members who helped make the event a success include the following: Exhibits Committee—Richard C. Chodnicki, of Van Horn, Metz & Company, Inc., Sara Robinson, of HeucoTech Ltd., and Fred L. Tolerico, of Chemarco, Inc.; Technical Program Chairman—Robert C. Matejka, of Akzo Coatings, Inc.; and Luncheon Chairman—David L. Reynolds, of Eastman Chemical Products, Inc.

"Application of ISO Standards"—speaker to be announced, E.I. du Pont de Nemours & Company, Inc.;

"Computer Systems for Small Business/Paint Contractors"—speaker to be announced, Pacific Micro Engineering;

"Why Material Safety Data Sheets (MSDS)"—speaker to be announced, OSHA;

"Basis for Raw Material MSDS"—Mike Larson, of Minerals Technologies;

"Basis for Coatings MSDS"—Chris West, Co-Chairman of PARLE;

"Paint Contractor/Consumer Opinion of MSDS"—Clifford Burg, Executive Director, of California Paint and Decorating Contractors of America; and

"What are Government and Industry Doing to Help Business"—Michael Allen, of A-K Associates.

In addition, a panel discussion, on "Safety and MSDS-Program Participants," has been planned.

The registration fee for the conference is $70.00, and includes lunch and dinner. Registration for the all-day symposium is limited.

Members of the Golden Gate Society Conference Committee include: Chairman Louie F. Sanguinetti, of Jasco Chemical Corporation; Co-Chairman Ronald Hughes, of Ashland Chemical Company; Ernest "Bud" Harmon, consultant; Adrian Adkins, of Schoofs, Inc.; Raymond J. Benedetti and Ken E. Trautwein, of Triangle Coatings, Inc.; Jack Duis, of Pacific Coast Chemicals Company; Lawrence Graubart, consultant; Don Mazzone, of The O'Brien Corporation; Tina Onderbeke, of Dowd & Guild, Inc.; Kevin Porterfield, of Minerals Technologies; A. Gordon Rook, consultant; Leo Schinasi, consultant; and Rocky Williams, of Napa Valley Paint Company.

For more information, contact Mr. Hughes, Ashland Chemical, Inc., 8600 Enterprise Dr., Newark, CA 94560.

PIEDMONT TRADE SHOW—Exhibitors are kept busy by inquisitive attendees

Literature

Spectrometer

The introduction of a new, fully optimized system spectrometer has been made through a product release. By combining the complementary techniques of Raman and infrared spectroscopy, the new spectrometer is designed to enhance routine analysis of solids, liquids, powders and gels, including stressed polymers, pharmaceutical tablets, and pharmaceutical polymorphs. For more information on the System 2000R Near-IR FT-Raman Spectrometer, write The Perkin-Elmer Corp., 761 Main Ave., Norwalk, CT 06859-0012.

Circle No. 200 on Reader Service Card

Colorants

Information highlighting a line of industrial colorants that are based on an acrylic resin vehicle intended to minimize the need for surfactants and additives has been released. Chroma-Chem 844® colorants are designed specifically for use in high performance, non-aqueous, industrial and maintenance coatings. For more information on Hüls industrial colorants, contact M. Feldman, Hüls America Inc., 80 Centennial Ave., Piscataway, NJ 08855-0365.

Circle No. 201 on Reader Service Card

Coating Thickness Gage

A digital coating thickness gage that combines four gages in one, as it measures non-magnetic coatings on ferrous and non-ferrous metals, is introduced in a product release. This gage also measures both low range and high range thicknesses with a gage range of 0-200 mils (0-5000 microns). For further information on the QuaNix 1500, contact KTA, 115 Technology Dr., Pittsburgh, PA 15275.

Circle No. 202 on Reader Service Card

Microwave Furnace

A new microwave high temperature furnace has been introduced through literature. The furnace reportedly harnesses the power of microwave energy to heat a variety of materials at temperatures up to 1200°C, which reduces typical analysis of ash and LOI to minutes instead of hours. For more information on the MAS-7000 Microwave Muffle Furnace, contact Vickie Kiker, CEM Corp., 3100 Smith Farm Rd., P.O. Box 200, Matthews, NC 28106-0200.

Circle No. 203 on Reader Service Card

Waste Disposal

A new product designed to remove paint solids from water wash spray booths, allowing the user to dispose of dry, solid waste material instead of water filled barrels, is the topic of a literature. Available in different sizes and configurations, the Paint Pig Plus is incorporated as a side stream filtration loop into the booth's water wash system. Contact Aaron Vermilion, US Centrifuge, 1435 Brookville Way, Ste. G, Indianapolis, IN 46239 for more information.

Circle No. 204 on Reader Service Card

Media Mill

The availability of a line of heavy duty, horizontal media mills for continuous production in paint, coatings, specialty chemical, food, pharmaceutical, and other applications has been announced. The units are designed with a high-energy cooling system for maximum heat transfer on heat sensitive products as well as a self-cleaning, rotating screen. To obtain more information on the Supermill® Wet Processing Media Mills, contact Premier Mill Corporation, One Birchmont Dr., Reading, PA 19606-3298.

Circle No. 205 on Reader Service Card

Coatings' Inspection Instrumentation

The 73-page handbook on coatings' inspection instruments use has been published. The publication provides pictures, descriptions, step by step calibration and use, and helpful hints for over 30 coatings inspection instruments and standards manufactured or written by leading companies and organizations in the coatings industry. Contact KTA, 115 Technology Drive, Pittsburgh, PA 15275, for more information on how to obtain a copy of Volume II of this handbook.

Circle No. 206 on Reader Service Card

Carbon Dioxide

Ultra-pure grade carbon dioxide developed specifically for use in supercritical fluid chromatography (SFC) and supercritical fluid extraction (SFE) is described in literature. The data sheet explains the manufacturing, packaging, and delivery of the two supercritical grades of carbon dioxide. For more information, request form #6905R from Liquid Carbonic Corp. Communications Dept., 800 Jorie Blvd., Oak Brook, IL 60521-2216.

Circle No. 207 on Reader Service Card

Solution to April's "CrossLinks"

Color Analysis System

The introduction of an advanced computerized color analysis system has been made through a product sheet. The system is designed to measure spectral characteristics of metallic and pearl surfaces as seen at different angles. For more information on the MULTIFLASH M 45 color analysis system, write Color and Appearance Technology, Inc., Princeton Commerce Center, 29 Emmons Dr., Bldg. G-2, Princeton, NJ 08543-3709.

Circle No. 208 on the Reader Service Card

Process Controllers

A series of universal process controllers is the topic of a new full-color brochure. The literature describes and illustrates the features of the new family of panel-mounted instruments available in three DIN sizes, 1/4, 1/8, and 1/16. For a copy of the brochure on the AT Series controllers, write Athena Controls, Inc., 5145 Campus Dr., Plymouth Meeting, PA 19462.

Circle No. 209 on the Reader Service Card

Weathering Tester

A new eight-page, full-color brochure detailing a QUV weathering tester with solar eye irradiance control has been printed. Product specifications are included, as well as figures and photos. For a copy of the brochure, contact Russell Raymond, The Q-Panel Co., 26200 First St., Cleveland, OH 44145.

Circle No. 210 on the Reader Service Card

Cellulose Fibers

A new technical bulletin discussing the use of cellulose fibers in roof coatings, crack sealers, and cementitious-base coatings has been published. Laboratory evaluations performed are detailed. Copies of the literature on Interfibe™ cellulose fibers are obtainable from Chris Sullivan, Interfibe Corp., 6001 Cochran Rd., A-202, Solon, OH 44139.

Circle No. 211 on Reader Service Card

Ultramarine Pigments

A complete line of high quality, ultramarine pigments is the subject of literature. These pigments are suitable to color a wide range of products including: styrenics, olefins, rubber compounds, paints, printing inks, and artists' colors. For additional information on the complete line of ultramarine pigments, contact Color Div., Ferro Corp., P.O. Box 6550, Cleveland, OH 44101.

Circle No. 212 on the Reader Service Card

Reactivated Carbon

A product bulletin highlighting a new powdered reactivated carbon has been issued. Applications for this product include: municipal and industrial wastewaters, cement kiln flue gas treatment, dioxin and mercury removal from incinerator flue gas, sludge stabilization, soil remediation, amine and glycol purification, and agricultural and industrial spill kits. Write Calgon Carbon Corp., P.O. Box 717, Pittsburgh, PA 15230-0717, for a copy of the product bulletin on Type WPX™.

Circle No. 213 on the Reader Service Card

Film Maker

A film maker developed to provide an easy-to-use and highly reproducible method of pressing polymers and other plastics into a thin film is the topic of a press release. The instrument produces 20 mm diameter films (an optional 3 mm platen is also available), which needs less pressure to prepare and can be produced with any hydraulic press. Write Spectra-Tech Inc., 652 Glenbrook Rd., Bldg. 8, P.O. Box 2190-G, Stamford, CT 06906 for further details on the Universal Film Maker.

Circle No. 214 on the Reader Service Card

Federation of Societies for Coatings Technology — YE OLDE PAINT STONE 1737

"Introduction to Pigments," Monograph by J. Braun, of Dupont Chemicals, Published by Federation

"Introduction to Pigments," which is authored by Juergen Braun, of Dupont Chemicals, surveys, explains, compares, and distinguishes the common elements and the important differences between pigment classes and their functions.

The new *Federation Series on Coatings Technology* is intended to serve as an educational resource for the industry.

Additional titles in the series include:

—"Film Formation," by Zeno Wicks, Jr.
—"Introduction to Polymers and Resins," by Joseph Prane
—"Radiation Cured Coatings," by J.R. Costanza, A.P. Silveri, and J.A. Vona
—"Solvents," by William Ellis
—"Coil Coatings," by Joseph Gaske
—"Corrosion Protection by Coatings," by Zeno Wicks, Jr.
—"Mechanical Properties of Coatings," by Loren W. Hill
—"Automotive Coatings," by Bruce N. McBane

—"Coating Film Defects," by Percy E. Pierce and Clifford K. Schoff
—"Application of Paints and Coatings," by Sidney B. Levinson
—"Organic Pigments," by Peter A. Lewis
—"Inorganic Primer Pigments," by Alan Smith
—"Marine Coatings," by Henry R. Bleile and Stephen Rodgers
—"Sealants and Caulks," by Joseph Prane
—"Aerospace and Aircraft Coatings," by A.K. Chattopadhyay and M.R. Zentner
—"Introduction to Coatings Technology," by Alan Brandau
—"Cationic Radiation Curing," by Joseph Koleske
—"Rheology," by Clifford Schoff
—"Powder Coatings," by Josef Jilek

The monographs are prepared in a standard 8 1/2 x 11 inch format. List price is $15 for individual copies, $10 if the existing set is purchased. The member cost is $7.50 for individual copies and $5.00 with an order of the full set. Orders may be placed by contacting Meryl Cohen, FSCT, 492 Norristown Rd., Blue Bell, PA 19422-2350.

Circle No. 215 on the Reader Service Card

Coming Events

FEDERATION MEETINGS

For information on FSCT meetings, contact Federation of Societies for Coatings Technology, 492 Norristown Rd., Blue Bell, PA 19422 (215) 940-0777, FAX: (215) 940-0292.

1993

(May 16-19)—Federation "Spring Week." Board of Directors Meeting on the 16th; Incoming Society Officers Meeting on the 17th; Spring Seminar on the 18th and 19th, "The Influence of Substrates and Application Methods/Techniques on Coatings Performance." South Shore Harbour Resort and Conference Center, League City (Houston), TX.

(Oct. 27-29)—71st Annual Meeting and 58th Paint Industries' Show. World Congress Center, Atlanta, GA.

1994

(May 12-15)—Federation "Spring Week." Spring Seminar on the 12th and 13th; Incoming Society Officers Meeting on the 14th; Board of Directors Meeting on the 15th. Marriott City Center Hotel, Minneapolis, MN.

(Oct. 12-14)—72nd Annual Meeting and 59th Paint Industries' Show. New Orleans Convention Center, New Orleans, LA.

1995

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

SPECIAL SOCIETY MEETINGS

1993

(June 4-5)—Joint Meeting of the Kansas City and St. Louis Societies. Holiday Inn, Lake of the Ozarks, MO.

(June 8-9)—New England Society. Coatings Tech Expo '93. "Compliance—Options for the 21st Century." Sheraton Tara Hotel & Resort, Danvers, MA. {Joanne Monique, Ashland Chemical, Inc., 400 Main St., Tewksbury, MA 01876; (800) 962-5388}.

(June 14)—Golden Gate Society Manufacturing Committee Seminar. "Process and Compliance." Ramada Inn, S. San Francisco, CA. {Ronald Hughes, Ashland Chemical, Inc., 8600 Enterprise Dr., Newark, CA 94560; (510) 796-9333}.

OTHER ORGANIZATIONS

1993

(May 16-21)—"Polymer Synthesis: Fundamentals and Techniques." Short course sponsored by American Chemical Society (ACS). Virginia Tech University, Blacksburg, VA. (ACS, Dept. of Continuing Education, Meeting Code VPI93, 1155 Sixteenth St., N.W., Washington, D.C. 20036).

(May 17-19)—Styrenics '93. Sponsored by Maack Business Services. Zürich Hotel International, Zürich, Switzerland. (Maack Business Services, Moosacherstr. 14, 8804 Au/Zürich, Switzerland).

(May 17-19)—"Advances in Coatings, Inks and Adhesives Technology." Third Asia-Pacific Conference. Raffles City Convention Centre, Singapore. (Mike Tarrant, Exhibition Director, FMJ Publications Ltd., Queensway House, 2 Queensway, Redhill, Surrey RH1 1QS, United Kingdom).

(May 17-20)—"Coatings Science for Coatings Technicians." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Debbie Ballard or Shelby Thames, USM, Dept. of Polymer Science, Southern Station, Box 10076, Hattiesburg, MS 39406-0076).

(May 17-21)—"Introduction to Paint Formulation." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (UMR Coatings Institute, 142 Schrenk Hall, Rolla, MO 65401).

(May 17-22)—"Interpretations of IR and Raman Spectroscopy." Course sponsored by Fisk Infrared Institute. Vanderbilt University, Nashville, TN. (Fisk Infrared Institute, P.O. Box 265, French Village, MO 63036).

(May 18 and 20)—"Rheology" Seminar. Sponsored by Brookfield Engineering Laboratories, Inc. Atlanta, GA on the 18th and St. Louis, MO on the 20th. (Barbara Cunningham, Marketing Dept., Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072).

(May 24-28)—"Adhesion Principles and Practice for Coatings and Polymer Scientists." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education—Chemistry, KSU, P.O. Box 5190, Kent, OH 44242-0001).

(May 25-28)—Annual Conference in Science and Technology of Pigment Dispersion. Luzern, Switzerland. (Angelos Patsis, Institute of Materials Science, State University of New York at New Paltz, New Paltz, NY 12561).

(June 2-4)—15th Annual International Conference on Advances in the Stabilization and Degradation of Polymers. Luzern, Switzerland. (Angelos Patsis, Institute of Materials Science, State University of New York at New Paltz, New Paltz, NY 12561).

(June 3)—"Rheology" Seminar. Sponsored by Brookfield Engineering Laboratories, Inc. Boston, MA. (Barbara Cunningham, Marketing Dept., Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072).

(June 6-7)—"Industrial Lead Paint Removal and Abatement." Tutorial sponsored by Steel Structures Painting Council (SSPC). Marriott Astrodome, Houston, TX. (SSPC, 4516 Henry St., Pittsburgh, PA 15213-3728).

(June 7-9)—"Engineering Solutions to Industrial Corrosion Problems." Conference cosponsored by the National Association of Corrosion Engineers (NACE) and Norsk Korrosjonsteknisk Forening (NKF). Rica Park Hotel, Sandefjord, Norway. (NKF, Rosenkrantzgate 7, 0159 Oslo, Norway).

(June 7-10)—"Coatings Science for Coatings Formulators." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Debbie Ballard or Shelby Thames, USM, Dept. of Polymer Science, Southern Station, Box 10076, Hattiesburg, MS 39406-0076).

(June 7-11)—"Fundamentals of Chromatographic Analysis." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continiuing Education—Chemistry, KSU, P.O. Box 5190, Kent, OH 44242-0001).

(June 7-11)—"Environmentally Compliant Coatings." Short course sponsored by North Dakota State University (NDSU), Fargo, ND. (Marek W. Urban, Program Director, or Debbie Shasky, Program Coordinator, Dunbar Hall, Rm. 54, NDSU, Fargo, ND 58105).

(June 8-11)—"Getting into Compliance with Air Quality Regulations for Paints, Coatings, and Printing Facilities." Course sponsored by University of California Berkeley. San Francisco, CA. (Continuing Education in Engineering, University Extension, University of California, 2223 Fulton St., Berkeley, CA 94720).

(June 14-17)—"Coatings Science for Coatings Chemists." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Debbie Ballard or Shelby Thames, USM, Dept. of Polymer Science, Southern Station, Box 10076, Hattiesburg, MS 39406-0076).

(June 14-25)—"Coatings Science." Short course sponsored by North Dakota State University (NDSU), Fargo, ND. (Marek W. Urban,

Program Director, or Debbie Shasky, Program Coordinator, Dunbar Hall, Rm. 54, NDSU, Fargo, ND 58105).

(June 15 and 17)—"Rheology" Seminar. Sponsored by Brookfield Engineering Laboratories, Inc. Chicago, IL on the 15th and Lansing, MI on the 17th. (Barbara Cunningham, Marketing Dept., Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072).

(June 21-22)—"Thin Film Coatings: Topics in Coating and Drying Technology." Short course sponsored by the University of Minnesota, Minneapolis, MN. (Jackie O'Brien, Dept. of Chemical Engineering & Materials Science, University of Minnesota, 421 Washington Ave. SE, Minneapolis, MN 55455).

(June 21-23)—International Colouristic Symposium. Sponsored by the Colouristic Section of the Hungarian Chemical Society, Balatonszéplak (by Lake Balaton), Hungary. (Hungarian Chemical Society, Colouristic Symposium Organizing Committee, H-1027 Budapest Fo u. 68, Hungary).

(June 22-24)—"Predictive Technology" Symposium and Exhibition. Sponsored by American Defense Preparedness Association. Twin Towers Hotel and Convention Center, Orlando, FL. (Tracy Stuckrath, American Defense Preparedness Association, Two Colonial Place, 2101 Wilson Blvd., Ste. 400, Arlington, VA 22201-3061).

(June 23-25)—Tecnopinturas '93. First Argentine Congress on the Technology of Coatings, Resins, Varnishes, Printing Inks, and Related Products. Sponsored by the Argentine Chemical Association. Buenos Aires, Argentina. (TAL Organizaciones y Servicios, A.J. de Sucre 1552, 8vo. Piso "A" (1428) Buenos Aires, Argentina).

(June 23-25)—"Coating Process Fundamentals." Short course sponsored by University of Minnesota, Minneapolis, MN. (Jackie O'Brien, Dept. of Chemical Engineering & Materials Science, University of Minnesota, 421 Washington Ave. SE, Minneapolis, MN 55455).

(June 27-July 2)—ORGABROM '93. 2nd International Symposium. Sponsored by the Dead Sea Bromine Group in cooperation with the Casali Institute of Applied Chemistry, The Hebrew University of Jerusalem. (Secretariat, P.O. Box 50006, Tel Aviv 61500, Israel).

(July 11-14)—Sixth International Symposium on "Polymer Analysis and Characterization." Crete, Greece. (Judith A. Sjoberg, Professional Association Management, 815 Don Gaspar, Santa Fe, NM 87501).

(July 12-16)—19th International Conference in Organic Coatings Science and Technology. Ledra Marriott Hotel, Athens, Greece. (Angelos Patsis, Institute of Materials Science, State University of New York at New Paltz, New Paltz, NY 12561).

(July 25-29)—Conference on "Lead in Paint, Soil, and Dust." Cosponsored by ASTM Committees D-22, E-6, D-1, and D-18. University of Colorado, Boulder, CO. (ASTM, 1916 Race St., Philadelphia, PA 19103).

(July 28-30)—"Basic Coatings for Sales and Marketing Personnel." Short course sponsored by University of Missouri-Rolla (UMR), Rolla, MO. (UMR Coatings Institute, 142 Schrenk Hall, Rolla, MO 65401).

(Aug. 2-6)—Gordon Research Conference on "Chemistry and Physics of Coatings and Films." Colby-Sawyer College, New London, NH. (A.K. St. Clair, Head, Advanced Aircraft Program Office, Materials Div., NASA, Langley Research Center, Hampton, VA 23681-0001).

(Aug. 3-5)—"Fundamentals of Corrosion and Its Control." Course sponsored by LaQue Center for Corrosion Technology. Blockade Runner Hotel, Wrightsville Beach, NC. (LaQue Center for Corrosion Technology, Inc., P.O. Box 656, Wrightsville Beach, NC 28480).

(Aug. 9-12)—"Coatings Science of Powder Coatings." Short course sponsored by University of Southern Mississippi (USM), Hattiesburg, MS. (Debbie Ballard or Shelby Thames, USM, Dept. of Polymer Science, Southern Station, Box 10076, Hattiesburg, MS 39406-0076).

(Sept. 5-10)—3rd International Congress on Polymer Photochemistry. Sponsored by Manchester Metropolitan University and University of Milan. Genova, Italy. (Emmezeta SRL, Via C. Farini, 70, I-20159, Milano, Italy).

(Sept. 8-10)—3rd International Paint Congress and Exhibition on Paint Industry Suppliers. Sponsored by the Brazilian Association of Paint Manufacturers. Palácio de Convenções do Anhembi, São Paulo, Brazil. (Especifica S/C Ltda, Rua Augusta, 2516-2nd, Cj 22, 01412-100 São Paulo SP, Brazil).

(Sept. 8-11)—Conference on "Innovative Responses from an Industry under Siege." Sponsored by the Skandinaviska Lackteknikers Forbund (SLF) Congress. Copenhagen, Denmark. (Michael Symes, President, SLF, Strandboulevarden 38, DK-2100, Copenhagen, Denmark).

(Sept. 12-14)—"Back to Basics." 81st Annual Convention of the Canadian Paint and Coatings Association (CPCA). Queen's Landing Inn, Niagara-on-the-Lake, Ontario, Canada. (CPCA, 9900 Cavendish Blvd., Ste. 103, St.-Laurent, Quebec H4M 2V2, Canada).

(Sept. 14-17)—Eurocoat '93 and XXth Congress of the French Association of Technicians of Paints, Varnishes, Printing Inks, and Adhesives (AFTPV). Acropolis Palace, Nice, France. (Secretariat UATCM, c/o AFTPV, 5 rue Etex, F. 75018 Paris, France).

(Sept. 23)—Detroit Colour Council Meeting. Michigan State Management Education Center, Troy, MI. (James Hall, General Motors Corp., 30009 Van Dyke, Warren, MI 48090).

(Sept. 29-Oct. 1)—"Accelerated and Natural Weathering Techniques for Coatings and Polymers." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education—Chemistry, KSU, P.O. Box 5190, Kent, OH 44242-0001).

(Oct. 3-5)—"Crosslinked Polymers: Chemistry, Properties and Applications"; "Fundamentals of Adhesion: Theory, Practice and Applications"; and "Polymer Blends and Alloys: Phase Behavior, Characterization, Morphology, Alloying Technology." Conferences sponsored by State University of New York (SUNY) at New Paltz. New Orleans, LA. (Angelos Patsis, Institute of Materials Science, SUNY, New Paltz, NY 12561).

(Oct. 4-6)—"Polyethylene '93." Conference sponsored by Maack Business Services, Swissôtel Zürich, Switzerland. (Maack Business Services, Moosacherstrasse 14, CH-8804, AU/ZH, Switzerland).

(Oct. 5-8)—"Introduction to Coatings Technology." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education—Chemistry, KSU, P.O. Box 5190, Kent, OH 44242-0001).

(Oct. 10-12)—"Advances in Polymer Colloids/Emulsion Polymers"; and "Principles of Polymer Degradation and Stabilization." Confer-
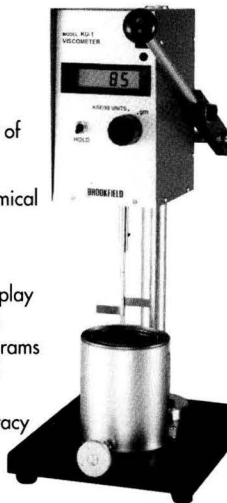
ences sponsored by State University of New York (SUNY) at New Paltz. Orlando, FL. (Angelos Patsis, Institute of Materials Science, SUNY, New Paltz, NY 12561).

(Oct. 12-14)—"Industrial Painting: Application Methods." Short course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education—Chemistry, KSU, P.O. Box 5190, Kent, OH 44242-0001).

(Oct. 19-20)—"Aspects of Photoinitiation." Conference sponsored by Paint Research Association (PRA). Anugraha Conference Centre, Egham, England. (Richard Kennedy, Radcure Services Dept., PRA, 8 Waldegrave Rd., Teddington, Middlesex, TW11 8LD, England).

(Oct. 25-27)—"Polypropylene '93." Conference sponsored by Maack Business Services, Swissôtel Zürich, Switzerland. (Maack Business Services, Moosacherstrasse 14, CH-8804, AU/ZH, Switzerland).

(Oct. 25-29)—"Image Analysis and Measurement in Scanning Electron Microscopy"; "Scanning Electron Microscopy and X-Ray Microanalysis for Materials Science. An Introductory Course"; and "Scanning Electron Microscopy for Polymeric Science. An Introductory Course." Conferences sponsored by State University of New York (SUNY) at New Paltz. Nevele Resort Hotel, Ellenville, NY. (Angelos Patsis, Institute of Materials Science, SUNY, New Paltz, NY 12561).

(Nov. 1-4)—12th Biennial Symposium on Managing Corrosion with Plastics. Sponsored by National Association of Corrosion Engineers (NACE), American Society for Testing Materials, Materials Technology Institute of the Chemical Process Industries, Inc., SPI Composites Institute, and Technical Association of the Pulp and Paper Industry. Baltimore, MD. (NACE, P.O. Box 218340, Houston, TX 77218-8340).

(Nov. 2-4)—"Fundamentals of Corrosion and Its Control." Training course sponsored by LaQue Center for Corrosion Technology. Holiday Inn, Wrightsville Beach, NC. (LaQue Center for Corrosion Technology, Inc., P.O. Box 656, Wrightsville Beach, NC 28480).

(Nov. 10-12)—Electroless Nickel '93. Conference sponsored by *Products Finishing*. Orlando Airport Marriott, Orlando, FL. (Cindy Goodridge, Gardner Management Services, 6600 Clough Pike, Cincinnati, OH 45244).

(Nov. 15-16)—"Fundamentals of HPLC." Short course sponsored by Kent State University (KSU), Kent, OH. (Carl J. Knauss, Director, Cooperative & Continuing Education—Chemistry, KSU, P.O. Box 5190, Kent, OH 44242-0001).

(Nov. 15-17)—"Powder Coatings." Paint Research Association's (PRA) 13th International Conference. Brussels. (Conference Secretary, PRA, 8 Waldegrave Rd., Teddington, Middlesex TW11 8LD, United Kingdom).

(Nov. 19-21)—46th Annual National Decorating Products Show. Sponsored by National Decorating Products Association (NDPA). Indiana Convention Center, Indianapolis, IN. (Ruth Wilms, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Nov. 23-25)—Conchem '93. International Exhibition and Conference sponsored by Reed Exhibition Companies. KKA Congress and Exhibition Centre, Karlsruhe, Germany. (Diane R. Tiberio, Reed Exhibition Companies, 999 Summer St., P.O. Box 3833, Stamford, CT 06905-0833).

(Dec. 6-8)—"Styrenics '93." Conference sponsored by Maack Business Services, Swissôtel Zürich, Switzerland. (Maack Business Services, Moosacherstrasse 14, CH-8804 AU/ZH, Switzerland).

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(Feb. 11-13)—37th Annual Southern Decorating Products Show. Sponsored by National Decorating Products Association (NDPA). Georgia International Convention & Trade Center, College Park (Atlanta), GA. (Ruth Wilms, NPDA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Mar. 5-6)—38th Annual Canadian Decorating Products Show. Sponsored by National Decorating Products Assoication (NDPA). International Centre, Toronto, Ontario, Canada. (Ruth Wilms, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Mar. 13-18)—"High Solids Coatings" Symposium. Sponored by American Chemical Society Division of Polymeric Materials: Science and Engineering. San Diego, CA. (Frank N. Jones, Coatings Research Institute, Eastern Michigan University, 430 W. Forest St., Ypsilanti, MI 48197 or George R. Pilcher, Akzo Coatings, Inc., P.O. Box 147, Columbus, OH 43216-0147).

(Mar. 14-18)—"Recycle '94." Forum and Exposition. Sponsored by Maack Business Services. Congress Centre, Davos, Switzerland. (Maack Business Services, Moosacherstrasse 14, CH-8804 AU/ZH, Switzerland).

(Apr. 14-16)—Paint Show '94. Sponsored by Japan Paint Manufacturers Association, Japan Paint Commerce Association, Japan Painting Contractors Association, and Nippon Toryo (Paint) Club. Nippon Convention Center, Tokyo, Japan. (Paint Show '94 Executive Committee, Overseas Operation,c/o Space Media Japan Co., Ltd., Asahi Bldg. 4F, 2-31-3 Taito, Taito-ku, Tokyo 110 Japan).

(Apr. 17-19)—"Volatile Organic Compounds (VOCs) in the Environment" Symposium. Sponsored by ASTM Committee E-47. Montreal, Que., Canada. (Wuncheng Wang, Symposium Chairman, U.S. Geological Survey, WRD, P.O. Box 1230, Iowa City, IA 52244).

(Apr. 23-24)—Eastern Decorating Products Show. Sponsored by National Decorating Products Association (NDPA). Garden State Convention & Exhibit Center, Somerset, NJ. (Ruth Wilms, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(May 1-5)—RadTech '94 North America. Conference sponsored by RadTech International North America. Walt Disney World Dolphin Hotel, Orlando, FL. (Chris Dionne, RadTech International North America, 60 Revere Dr., Suite 500, Northbrook, IL 60062).

(June 5-9)—XXIInd FATIPEC Congress and Exhibition. Budapest Convention Centre, Budapest, Hungary.

(July 11-15)—MACROAKRON '94 International Symposium. Sponsored by the International Union of Pure and Applied Chemistry. University of Akron, Akron, OH. (Dr. Joseph P. Kennedy, Chairman of the Organizing Committee, or Cathy Manus-Gray, Symposium Coordinator, College of Polymer Science and Polymer Engineering, The University of Akron, Akron, OH 44325-0604).

(Nov. 6-10)—International Adhesion Symposium. Sponsored by The Adhesion Society of Japan. Tokyo, Japan. (Hiroshi Mizumachi, Professor, Chemistry of Polymeric Materials, The University of Tokyo, Yayoi 1-1-1, Bunkyo-ko, Tokyo 113, Japan).

'Humbug' from Hillman

The merry month of May brings us the Platt family—Doris and Dave, to add to the spring merriment. I chose a quick little gem from Doris' file:

A Fish Story

As soon as we met the spell was cast.
"Reel" sweethearts we became very fast.
I was hooked that first night,
I knew I was caught and put up no fight.

—Doris Platt

And after more than 50 years, she's still hooked! (If Doris is the hookee, does that make Dave the ?????—Can't be!)

While you ponder that, consider these, from David:

• Using cows to predict the weather is an old custom. "When the cows are standing," the old farmer assured me, "it means no rain for 24 hours and when they're lying down, it means it's going to rain." "What if half are lying down and half are standing?" I asked.

"That means half of 'em are wrong," he answered gravely.

• Children go through some fascinating stages. First they call you Da Da. Then they call you Daddy. Then they call you Dad. Then they call you collect.

—*Winnebago Catalogue*

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A secret friend sent a bunch of items a few months back. Included was a column written by one Andrew Heller entitled, "20's Survival Guide." My recollection is that nothing helped. Here are some of Andrew's words of wise(?) advice:

• You do not have to shampoo, rinse, then "repeat," no matter what the shampoo bottle says.

• Complaining is useless. Unfortunately no one gives a rat's hiney.

• Fake until you know it. Everyone does but no one admits it.

• Don't make any major life decisions in your 20s. You are too stupid. Wait until you are in your 30s, at which point you will be wise enough to realize that you are far too stupid to make any decisions until you are in your 40s.

• Adulthood stinks. Who decided that adults no longer get the summer off?

• Socks are optional. Flossing isn't.

• Marriage is tougher and more rewarding than you could ever imagine. Approach it as you would an unfamiliar dog: slowly and with caution.

• Some things in life defy explanation: ice fishing, Geraldo, Brussels sprouts, heavy metal, Spam, and bosses.

• Most of all—forgive and forget. It's the only way.

My anonymous friend must be a fellow Lion since he sent me a "Laugh" page from an old issue of *Lions Magazine*—from which I regularly steal stuff, like this:

• The noblest of all animals is the dog, and the noblest of all dogs is the hot dog. It feeds the hand that bites it.

• Did you hear about the cat that ate some cheese and then waited for the mouse with baited breath?

• Tricycle: A tot rod.

• Vacations would be a lot more pleasant if you could stop the lawn along with the paper and mail.

• The trouble with self-made men is that they worship their creator.

• Optimist: Someone who tells you to cheer up when things are going his way.

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Let us turn to Dr. Bob Brady, our "technical" Technical Editor for some inside stuff on how government inspection works (as if we didn't know):

• There is the story of three government inspectors running a viscosity test for a federal specification. The first inspector had never seen a Krebs-Stormer Viscometer, so when he ran the test he got a value that was 15 KU too high. The second inspector didn't know how to adjust the weights. When he ran the test and got a value that was 15 KU too low, the third inspector cried out, "We've got it! We'll take all 10,000 gallons!"

Bob is sure you heard this one, but nonetheless:

• Two sodium atoms were sitting in a bar and one said to the other, "Good grief! I've lost an electron!" "Are you sure?" asked his friend. "Yes," replied the first. "I'm positive!"

If I think you can stand it there'll be more Bradyisms to come.

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Earl Hill doesn't quite outdo Dr. Bob, but this is bad enough:

• A boy's pet bird fell into a can of varnish and drowned. "It was a sad way to die," the boy said, "but he sure had a beautiful finish!" . . . Well, it's appropriate.

And Nancy Lindquist had a Russian immigrant professor who responded "to a tangential (is that bad?) question with 'If you're interesting, we'll talk about it later.'"

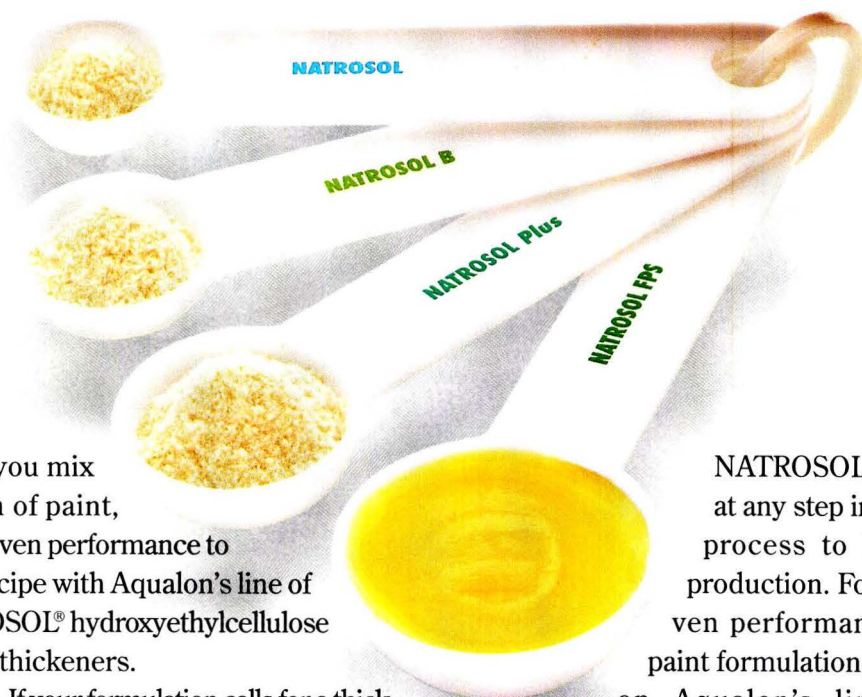. . . As Cliff Claven would say, "So what's bad about that?"

—*Herb Hillman*
Humbug's Nest
P.O. Box 135
Whitingham, VT 05361