

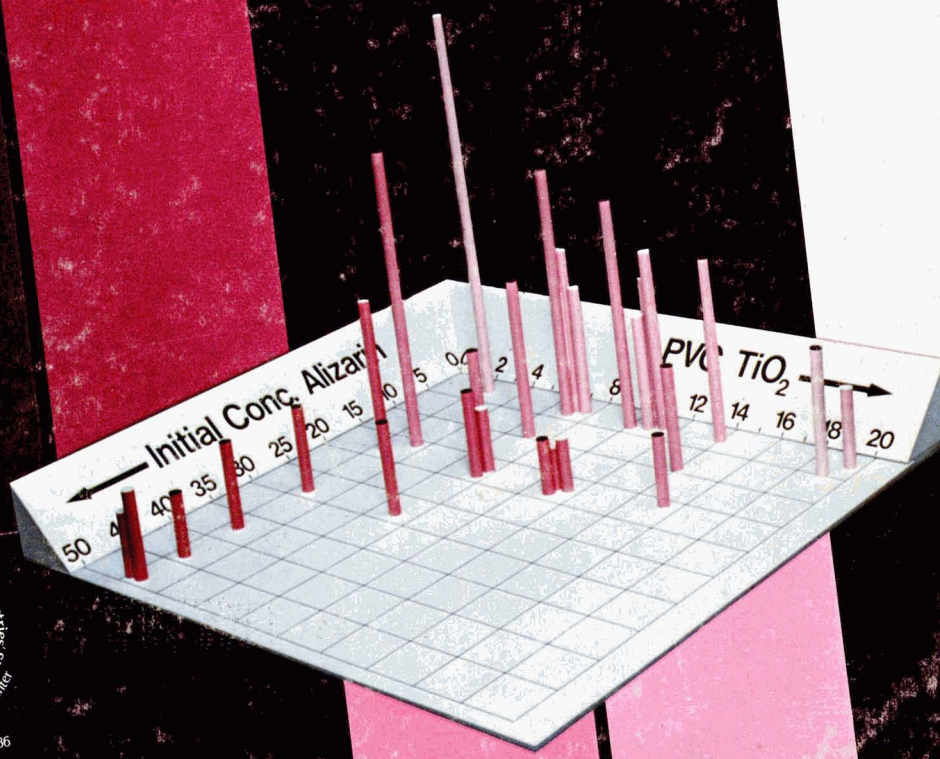


# JOURNAL OF COATINGS TECHNOLOGY

JCTAY 58 (736) 1-138 (1986)

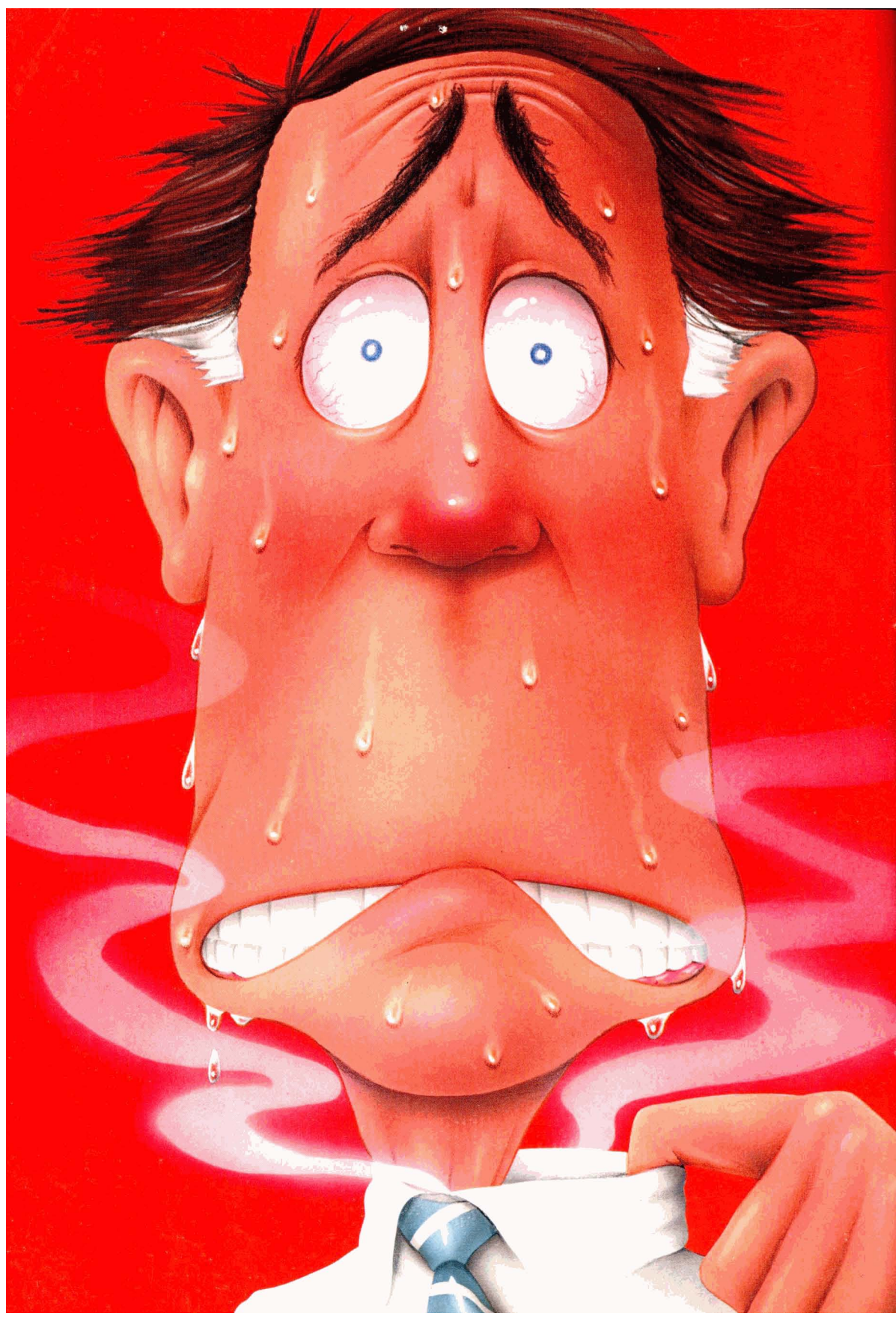
## May 1986

### Reflections on the Phenomenon of Fading 1985 Mattiello Lecture



44th Annual Meeting & 51st Paint Industry Show  
**FSCT**  
**1986**  
Atlanta, Georgia  
November 5, 6, 7, 1986  
World Congress Center







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### Technical Articles

- 32 Reflections on the Phenomenon of Fading—  
R.M. Johnston-Feller
- 53 Polyvinylacetate Emulsion Design and Rheological  
Performance in Latex Paints—B.D. Nguyen and A. Rudin
- 71 Use of A-B Block Polymers as Dispersants for Non-aqueous  
Coating Systems—H.L. Jakubauskas
- 83 Influence of Fine-Particle Size Extenders on the Optical  
Properties of Latex Paints—L. Cutrone
- 91 Structure and Properties of Amorphous Silica Gel in Coatings  
Applications—L. Kutik
- 97 Addendum to Infrared Spectroscopy Atlas—Scandinavian  
Paint and Printing Ink Institute

### Federation News

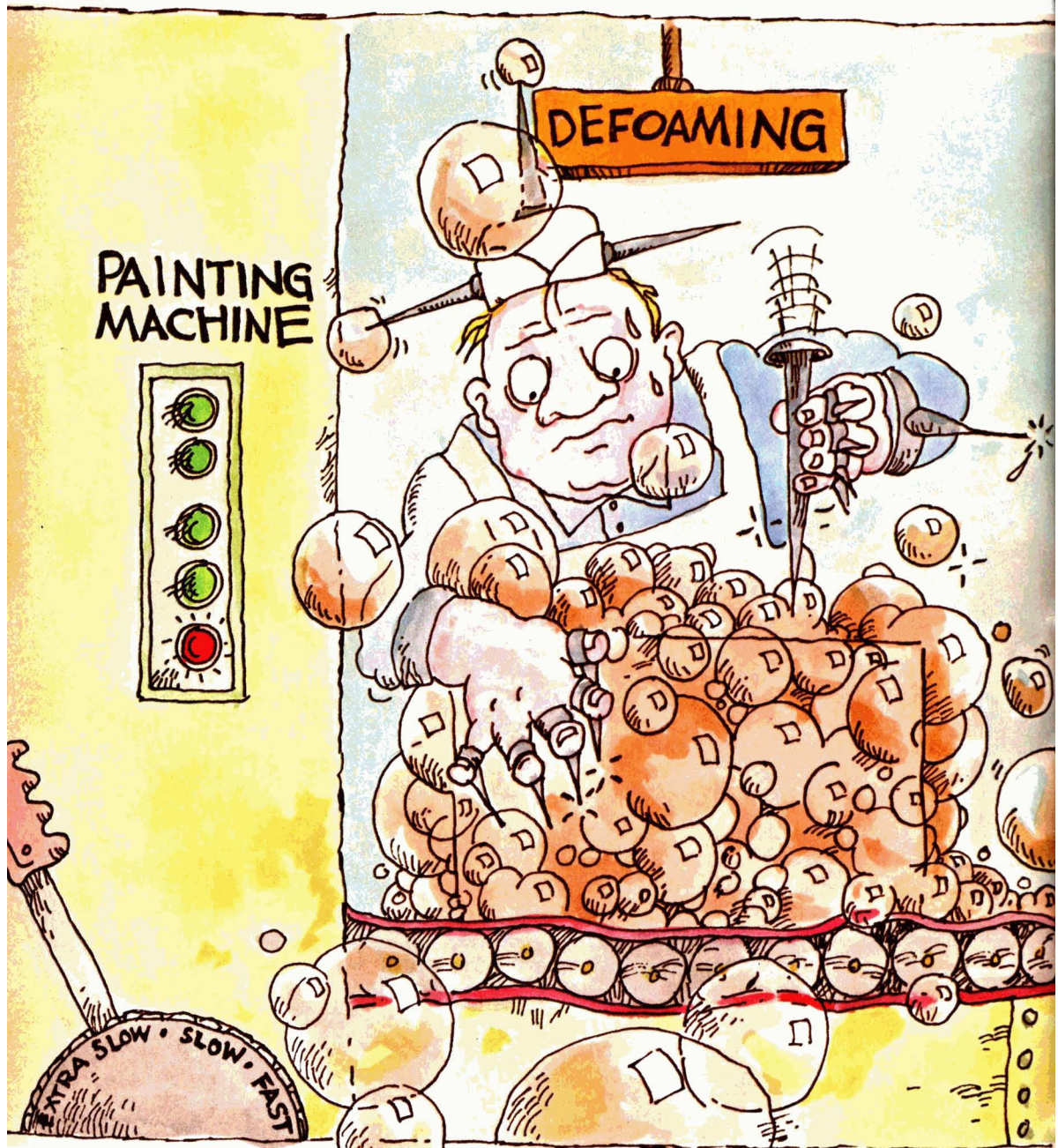
- 14 Zeno Wicks to Present 1986 Mattiello Lecture
- 16 SACC Symposium to Be Conducted in Southfield, MI, June 3-6
- 18 Southern Society's Annual Meeting Attracts 460 Individuals
- 18 FSCT Establishes Southern Society A.L. Hendry Award
- 23 1986 Annual Meeting Advance Registration and Housing Forms
- 30 List of Exhibitors for 1986 Paint Industries' Show

### Departments

- Comment 7 A Need to Know
- Abstracts 10
- Government and Industry 20
- Report of ASTM  
Committee D-1 105
- Society Meetings 119
- Elections 125
- People 127
- Obituary 128
- Meetings/Education 129 Gordon Research Conference Posts 1986 Program
- Literature 131
- Book Review 133
- CrossLinks 133 Solution to April Puzzle
- Coming Events 135
- Humbug from Hillman 138 "How You Can Tell It's Going to Be a Rotten Day" plus More!



# There are alternative



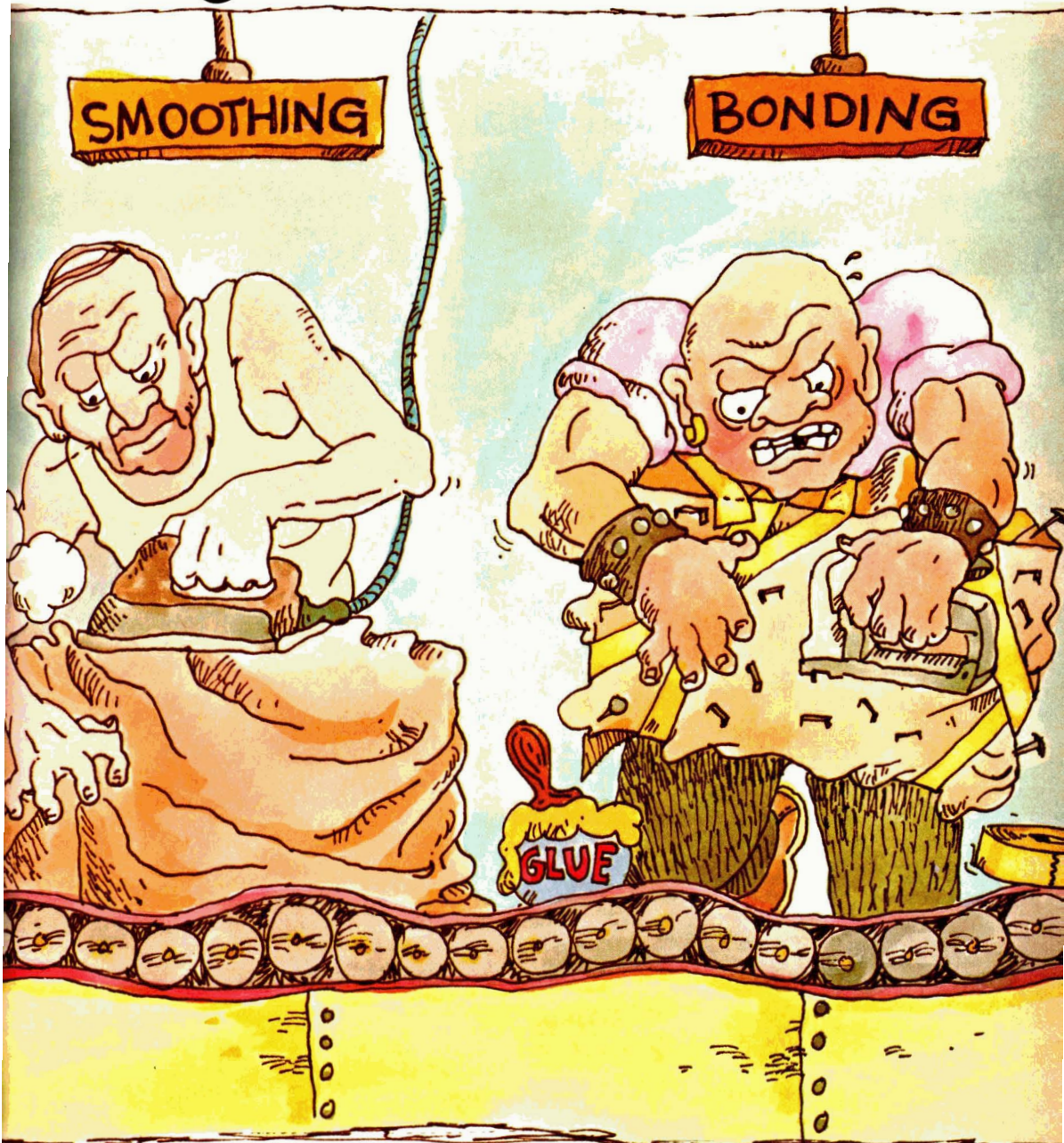
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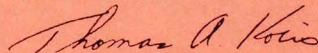
## A Need to Know

A primary objective of the newly-formed FSCT Professional Development Committee is to provide continuing education opportunities for the technical staff of our industry.

To assist in this regard, the Committee recently prepared a two-page survey form, to clarify certain demographic aspects of Federation membership and to more accurately assess member needs.

The form, which was mailed to all members, requires only a few minutes to complete, and the information it yields will be most useful in the Committee's efforts to promote professional development opportunities.

The project should be of real value, but its success is dependent on member cooperation. If you have not already done so, please complete the form and return promptly to the FSCT office. We will all benefit from this "Need to Know!"



Thomas A. Kocis,  
*Contributing Editor*



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# Abstracts of Papers in This Issue

## REFLECTIONS ON THE PHENOMENON OF FADING— R.M. Johnston-Feller

Journal of Coatings Technology, 58, No. 736, 32 (May 1986)

Evaluation of the degree of fading on exposed paints by visual means alone obscures the underlying physical behavior of the pigments. It is shown that "Mother Nature is logical" and that, when hiding is complete, and the dispersion is uniform, the rate of fading is constant and is not appreciably affected by many of the variables of paint formulation, such as pigment volume concentration. On this basis, the observed visual changes are also predictable. When hiding is incomplete, however, the rate of fading increases and is dependent on the nature of the substrate. Illustrations of these phenomena are presented.

## POLYVINYLACETATE EMULSION DESIGN AND RHEOLOGICAL PERFORMANCE IN LATEX PAINTS—B.D. Nguyen and A. Rudin

Journal of Coatings Technology, 58, No. 736, 53 (May 1986)

An instrumental procedure was used to characterize the rheological behavior and application properties of latex paints and their parent polymer emulsions. Vinyl acetate-dibutyl maleate copolymer latices were polymerized with various particle sizes. Polymerizations were carried out with or without hydroxyethyl cellulose protective colloid or a urethane block copolymer associative thickener. The latices were used to produce semi-gloss (40% PVC) paints.

Latices made without cellulosic polymer in the polymerization step were not thixotropic; those polymerized in the presence of hydroxyethyl cellulose were thixotropic. Latex viscosity increased in the order: (a) latex without cellulosic thickener in the emulsion recipe (non-thixotropic); (b) latex made with thickener in the polymerization (thixotropic); (c) latex (a) with post-added cellulosic (non-thixotropic). Addition of a water-soluble cellulosic thickener to a thixotropic latex increased the viscosity of the system but decreased the thixotropic character.

Paint behavior paralleled that of the original latex. Non-thixotropic latices yielded non-thixotropic paints even when the mill base contained a cellulosic thickener. Thixotropic latices produced thixotropic paints.

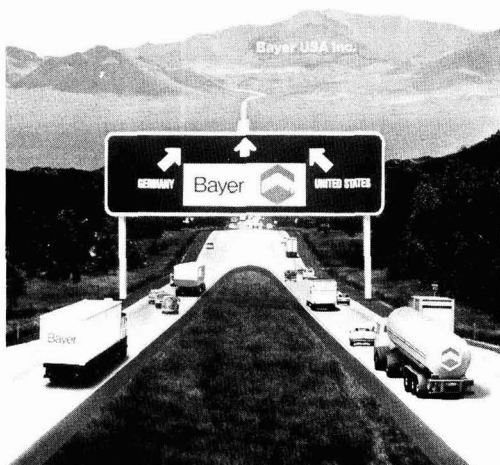
Semi-gloss paints made from non-thixotropic latices were more shear-thinning than the corresponding latices.

By contrast, paints from thixotropic latices were less shear-thinning than the parent latices. The associative thickener reduced shear-thinning character and promoted flow and levelling.

Differences in latex particle sizes were swamped by the consequences of including or eliminating the water-soluble cellulosic polymer in the emulsion polymerization recipe. Particle size had very little effect on levelling behavior of thixotropic paints. Brushability was also unaffected, except that very small particle size latices promoted brush drag.

Insights into the thickening action of water-soluble celluloses and urethane block copolymer associative thickeners are discussed.

## Who is Mobay? Who is Bayer AG? Who is Bayer USA Inc.?



For the answers turn the page.



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# Abstracts of Papers in This Issue *(continued)*

## **USE OF A-B BLOCK POLYMERS AS DISPERSANTS FOR NON-AQUEOUS COATING SYSTEMS—H.L. Jakubauskas**

Journal of Coatings Technology, 58, No. 736, 71 (May 1986)

Theoretical considerations involved in the dispersions of pigments are reviewed, including a new class of pigment dispersants for non-aqueous solvent systems that has been developed by Du Pont. These dispersants are polymer molecules containing "polar clusters" attached to one end of non-polar polymeric chain. The stability of dispersed pigment particles is achieved by adsorption of the "polar cluster" on the pigment surface with the non-polar portion of the molecule providing a barrier for pigment-to-pigment interactions. The mechanism of stabilization of particles is steric (entropic).

Pigment deflocculation is shown to be a function of dispersant structure, molecular weight polar and non-polar segments, pigment polarity, and solvent polarity used.

## **INFLUENCE OF FINE-PARTICLE SIZE EXTENDERS ON THE OPTICAL PROPERTIES OF LATEX PAINTS—L. Cutrone**

Journal of Coatings Technology, 58, No. 736, 83 (May 1986)

The "spacer" theory suggests that coarse extenders crowd titanium dioxide particles together, reducing the ideal spacing at which the pigment affects maximum light scattering. When extender particles of a size closer to that of titanium dioxide are used, they move between the titanium dioxide particles and space or separate them for greater scattering efficiency. This paper gives examples of light scattering phenomena which are better explained by a new model

involving air encapsulation. For example, fine-particle size extenders were found not to enhance the scattering ability of titanium dioxide but merely maintain the same level of opacity as binder is removed at low concentration of titanium dioxide (10% PVC), and a reduction in scattering efficiency as the titanium concentration was increased (20 and 30% PVC). A number of fine-particle size extenders were used in the study and none improved the scattering efficiency of titanium dioxide.

## **STRUCTURE AND PROPERTIES OF AMORPHOUS SILICA GEL IN COATINGS APPLICATIONS—L. Kutik**

Journal of Coatings Technology, 58, No. 736, 91 (May 1986)

Amorphous silica gel is a unique type of synthetic silica which normally exhibits a high surface area, low density, and a porous structure. Each of these properties is controlled by the manufacturing process so that highly specialized products for a variety of coating applications result. Physical properties such as pore volume, surface area, and density are important considerations during the manufacturing process. Examples are shown how these parameters are modified to optimize the adsorption properties of silica gels which are used to remove moisture from water sensitive coating materials. Similarly, the effect of particle size, density, pore volume, and surface properties on the flattening efficiency of silica gel pigments is described. Flattening efficiency curves demonstrating the use of silica gel pigments in various industrial coating systems are presented. The use of recently developed silica gel thixotropes in various nonaqueous systems is also described, together with a comparison with other types of synthetic silicas.



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Mobay Corporation is a diversified manufacturer supplying advanced materials and technology to the entire range of American industry.

Mobay ranks among the top 50 chemical and agricultural producers in the United States with sales of \$1.6 billion. Headquartered in Pittsburgh, Pennsylvania, Mobay has 6,000 employees nationwide, nine operating divisions, and six major production sites.

Mobay is the largest manufacturer of polyurethane raw materials and systems used in everything from furniture cushioning to auto bumpers and body panels, shoe soles and protective paints. The Mobay family of herbicides, fungicides, insecticides and other crop protection chemicals is labeled for use on nearly 100 different crops. Other Mobay products include engineering thermoplastics, animal health products, colorants and industrial chemicals.

Mobay is research & technology-driven, active in introducing and expanding the use of sophisticated materials in the production of high-performance products in key U.S. industrial and agricultural markets.

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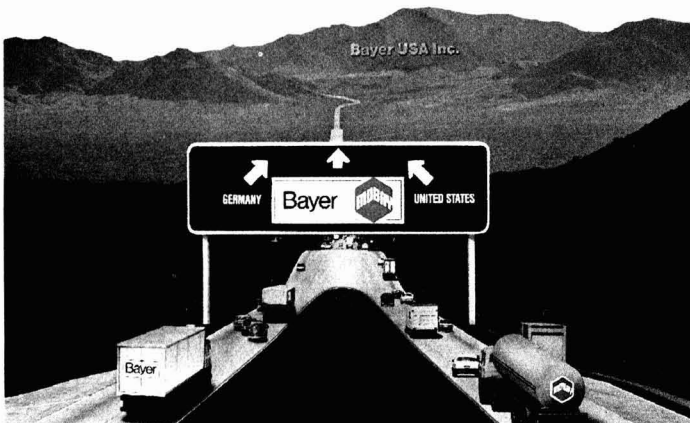
Bayer USA Inc., the newly named Management Holding Company of Bayer AG, located in Pittsburgh, Pennsylvania, will oversee the operations of Bayer's U.S. activities, total sales of which were about \$4 billion in 1985.

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## Zeno W. Wicks, Jr., Industry Educator, To Present 1986 Mattiello Lecture At Federation Annual Meeting in Atlanta

The Federation of Societies for Coatings Technology is pleased to announce that Dr. Zeno W. Wicks, Jr., former Department Chairman of Polymers and Coatings at North Dakota State University, will present the Joseph J. Mattiello Memorial Lecture at the 64th Annual Meeting of the Federation, at the Georgia World Congress Center in Atlanta, November 5-7.

Dr. Wicks will speak on "Free Volume and the Coatings Formulator" at the morning session on Friday, November 7.

The lecture commemorates the contributions of Dr. Mattiello, who did much to expand the application of the sciences in the decorative and protective coatings field. Dr. Mattiello, who served as President of the Federation in 1943-44, was Vice-President and Technical Director of Hilo Varnish Corp., Brooklyn, NY, when he died in 1984.

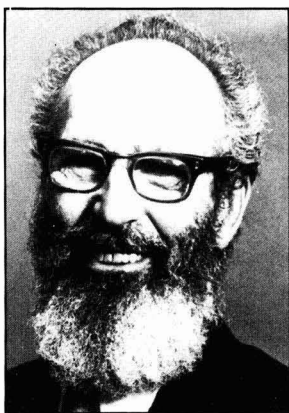
The lecturer is chosen from among those who have made outstanding contributions to science, and is selected to present a paper on a phase of chemistry, engineering, human relationships, or other science fundamental to paint, varnish, lacquer, or related protective and decorative coatings.

### Early Career

Dr. Wicks was graduated from Oberlin College in 1941 and obtained his Ph.D. in Organic Chemistry at the University of Illinois in 1944. He then joined the Central Research Laboratories of Interchemical Corp. (now BASF Inmont) in New York City. Initially, he was assigned to work on drying oil and alkyd resin research.

In his 28-year career at Inmont, Dr. Wicks served as Director of Central Research Laboratories, Vice-President of Planning and Commercial Development, Group Vice-President, and a member of the Board of Directors. His last assignment

was the responsibility for the coatings, adhesives, sealants, and coated fabrics laboratories for the automotive, building, and general industrial markets division of the corporation.



Dr. Wicks has conducted or directed industrial research projects in the areas of coatings, polymers, coated fabrics, printing inks, textile colorants, plastics, and sealants. In addition, he has served as a consultant to a number of governmental agencies with regard to environmental regulations in the coatings industry.

### Innovator and Educator

In 1972, Dr. Wicks transferred from industry to academia when

he joined the faculty of North Dakota State University as Professor and Chairman of the Polymers and Coatings Dept. With Loren Hill and Peter Pappas, he participated in the reinvigoration of the first academic coatings center in the world. In 1981, he was designated a Distinguished Professor of the university.

In research at NDSU, Dr. Wicks became involved in a broad range of work related to different aspects of the coatings field, partly due to a wide range of interests but also because of a conviction that one can teach about a topic more effectively if one has done research related to that topic. His principal areas of research included "water-soluble" acrylic resins and baking enamel systems, UV curing, chemistry of crosslinking reactions, and viscosity of oligomer solutions. With his graduate students and colleagues, Dr. Wicks published 20 research papers and 14 review and general interest papers from 1975-1985. Four of the research papers were awarded Roon prizes as the best technical papers offered for presentation at the Federation's Annual Meetings.

However, while active in research, his first interest was in teaching. He instituted a

modernization and expansion of the course work at NDSU. The primary emphasis in the courses was to achieve an understanding of the basic principles and to illustrate their application to real life problems of coatings formulation. Dr. Wicks realized that in many cases there had not yet been sufficient basic research, and hypotheses based on work in other fields of chemistry, especially polymer chemistry, were developed to provide a tentative basis to relate science to the art of coatings formulation. After some years experience with university students, courses were designed to be given as short courses for technical personnel from industry companies.

The connection between teaching and the polymers and coatings industry is seen by Dr. Wicks as being a totally interrelated system. And he is particularly interested in promoting understanding and cooperation between the two. NDSU's Polymers and Coatings Department's Industry Advisory Committee has proven to be of great value both to the Department and to the industry members. It has served as a model for other departments at NDSU and in some other universities' programs for industry-university interactions.

### Consultant and Lecturer

Although he retired from the faculty at NDSU in 1983, Dr. Wicks has remained active as a consultant to the industry, teaching workshops and short courses on subjects ranging from polymers and coatings to management practices and productivity.

By combining his fondness for travel with the love of teaching, Dr. Wicks, with his wife, Susan, has brought these courses not only to companies in the U.S., but also to China, England, and France.

In addition, he has completed writing two of the new Federation Series on Coatings Technology, "Film Formation" and "Corrosion Protection by Coatings."

Dr. Wicks is an Honorary Member of the Federation's Northwestern Society and is an Emeritus member of the American Chemical Society.

*(Continued on page 20)*



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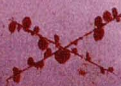
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# Automotive Color Control Symposium Will Feature General Sessions, Workshops, and Equipment Displays

A two-day symposium focusing on the new Society for Automotive Engineers Recommended Practice J1545 for instrumental color difference measurement for exterior finishes, to be held June 3-4 at the Michigan Inn, Southfield, MI, will combine general sessions and workshops with "hands-on" equipment demonstrations.

The Federation is co-sponsoring the symposium, along with the Detroit Society for Coatings Technology, the Detroit Colour Council and the Manufacturers Council on Color and Appearance.

The June 3-4 programming for coatings will be followed by similar two-day events, for Soft Trim (June 4-5) and Plastics (June 5-6).

"J1545," the culmination of work carried out by an industry-wide committee formed by the Detroit Colour Council, specifies the procedures, instruments, and documentary standards to be used for color difference measurement of colored parts and materials supplied for the manufacture of highway vehicles, and is expected to be widely adapted and useful for Statistical Process Control.

The opening session will be devoted to color difference measurement, with a complete explanation of "J1545" and its implementation. Other sessions will feature lectures on Statistics of Measuring; Effect of Metamerism and Alternative Illuminant and Standard Observer; Multi-Angle Measurement for Metallic Colors; and Reference Standards for Matching Parts.

The lectures will be presented by members of the DCC/SAE Color Measurement Committee, which developed "J1545."

Three workshops (two on instrumental color measurement, featuring equipment demonstrations; and a tutorial on sample

color difference samples from the Collaborative Reference Program will be furnished to each registrant, and a variety of automotive materials will be available for measurement. Registrants are also invited to bring samples.

Programming is designed so that registrants will have adequate opportunity to discuss topics and examine instrumentation. Time will also be available for those wishing to pursue additional inspection of equipment on display and for one-on-one discussions with representatives from the equipment manufacturers.

Attendance will be limited to assure individual participation.

Symposium General Chairman is James E. Grady, Pigments Dept., CIBA-GEIGY Corp., Birmingham, MI. General Program sessions are under the direction of William V. Longley, Design Center, Ford Motor Co., Dearborn, MI. Arrangements for the workshops and instrument displays are being handled by the Manufacturers Council on Color and Appearance.

Registration fee for each two-day segment is \$175, which includes two continental breakfasts, three coffee breaks, two lunches, reference material, and bus transportation to the airport at conclusion of symposium.

To obtain registration/housing forms, contact Federation of Societies for Coatings Technology, 1315 Walnut St., Suite 832, Philadelphia, PA 19107. Telephone (215) 545-1506.



preparation and presentation) afford an opportunity for participants to measure provided samples and determine correlation of the various instruments for metameric and non-metameric samples. Registrants will rotate from one workshop to the next until all have been attended.

Participating workshop exhibitors are: Applied Color Systems, Inc.; DIANO Color Group, Milton Roy Co.; Byk-Chemie USA, Inc.; Hunter Associates Laboratory; Macbeth Div., Kollmorgen Corp.; Minolta Co.; Pacific Scientific Co.; Gardner/Neotec Div.; and Collaborative Testing Services, Inc.

The closing session will include a panel discussion on implementation and a summary of the workshop visual-instrumental correlation study.

## Program

### Tuesday, June 3 (Morning)

GENERAL SESSION  
(9:00 am to Noon)

"Color Difference Measurement"—Ralph Stanziola, Industrial Color Technology, Bridgewater, NJ

This session will be devoted to a full and complete explanation of SAE Recommended Practice J1545 and its implementation. Special emphasis will be on its application for determining color difference for automotive materials and parts by coatings personnel.

### (Afternoon)

WORKSHOPS  
(1:30 to 3:00 pm)

Three concurrent workshop sessions:

"Sample Preparation and Presentation"—Richard Harold, Hunter Associates Laboratory, Reston, VA

"Instrumentation I"—Four participating instrument manufacturers demonstrate.

"Instrumentation II"—Three participating instrument manufacturers demonstrate, along with an explanation of the Collaborative Reference Program.

WORKSHOPS  
(3:30 to 5:00 pm)

Registrants rotate to second round of workshops, as assigned.

COLOR CLINIC  
(5:00 to 6:30 pm)

An opportunity for one-on-one discussions with representatives of exhibiting firms,

and for operation of instrumentation equipment, in an informal atmosphere.

### Wednesday, June 4 (Morning)

GENERAL SESSION  
(8:45 to 10:00 am)

"Reference Standards for Matching Parts"—Richard Pinamonti, 3M Co., Decorative Products Div., St. Paul, MN

It is important that production lots be compared to physically similar reference standards, although the official standard may be a different material.

*(Continued on page 18)*

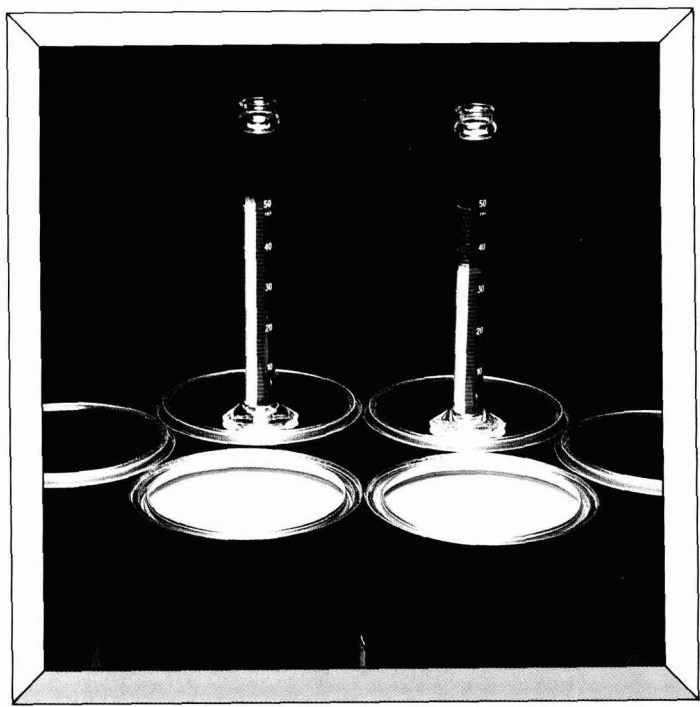


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# 'Back to School' Program Sessions Draw 460 to Southern Annual Meeting

A technical program featuring lectures by college professors attracted 330 industry registrants and 130 spouses to the Southern Society's "Back to School" Annual Meeting, April 2-4, in Savannah, GA. It was the highest attendance in recent years at the annual gathering of the Society.

The entire event was directed by Society Vice-President, Ronald B. Brown, of Unocal Chemicals Div. The program sessions, arranged by Brian Budzien, of Lilly Industrial Coatings, consisted of the following presentations:

(1) "Research and Purchasing—Partners or Adversaries for Success" (Keynote Address)—James Depew, of Glidden Coatings & Resins Div., SCM Corp.

(2) "Principles of Film Formation"—Dr. Peter Pappas, of North Dakota State University.

(3) "Chain Growth Polymerization"—Dr. Pappas.

(4) "Alkyds and Polyester Resins"—Dr. Pappas.

(5) "High Solids Coatings"—Dr. Robson F. Storey, of University of Southern Mississippi.

(6) "Fundamentals of UV Curing"—Dr. Storey.

(7) "Paint Testing and Quality Assurance"—John A. Gordon, of Eastern Michigan University.

Others who assisted Mr. Brown with meeting arrangements were: Registration—Secretary C. Lewis Davis, of Vi-Chem, Inc., and Treasurer R. Scott

McKenzie, of Southern Coatings Co.; Golf—D. Willson Blake, of Kemira, Inc.; Tennis—Melvin L. Penn, of Kerr-McGee Chemical Corp.; Spouses Activities—Robert and Ann Link, of R.T. Vanderbilt Co.; River Cruise—Philip Aultman, of Burks, Inc.; and Publicity—Terry Walsh, of Kinsman Corp. The President of the Society was Sal Sanfilippo, of Reichhold Chemicals, Inc.

Officers were elected for the coming

year and serving in these capacities now are: President—Mr. Brown; Vice-President—Mr. Davis; Secretary—Mr. McKenzie; Treasurer—Ken W. Espeut, of Carter Coatings Corp.; and Society Representative—Berger G. Justen, of Justen Associates.

The 1987 Annual Meeting will be held at the Americana Dutch Inn, Buena Vista, FL, April 1-3. In 1988, it will be in Charleston, SC, April 13-15.

## FSCT Establishes A.L. Hendry Award For Best Student-Authored Papers

The Federation of Societies for Coatings Technology is pleased to announce establishment of the Southern Society Alfred L. Hendry Memorial Award.

The Award is a \$1,000 cash prize for each of the two best student-authored papers on some aspect of coatings technology. Papers may be authored by either graduate or undergraduate students currently enrolled in a college program.

Prospective student authors are invited to submit manuscripts to the Federation headquarters office at 1315 Walnut St., Philadelphia, PA 19107. *Deadline for receipt of entries in the 1986 competition is June 20.*

Presentation to this year's winners will be made at the 1986 FSCT Annual Meeting in Atlanta, Nov. 5-7. The Award is administered by the Federation Educational Committee, which will also judge the entries.

The Award, sponsored by the Southern Society for Coatings Technology, commemorates the industry contributions of Alfred L. Hendry, President of A. L. Hendry & Co., Tampa, FL, and a Past-President and long-time member of the Society. Mr. Hendry died June 6, 1985.

Educational Committee member James E. Geiger is in charge of the 1986 Award competition. If you have any questions, or require clarification of any aspect of the competition, please contact him, c/o Sun Coatings, Inc., 12295 75th St., N., Largo, FL 33540.

## Automotive Color Control Symposium

(Continued from page 16)

"Effect of Metamerism and Alternative Illuminant and Standard Observer"—Rolf Kuehni, Mobay Chemical Corp., Rock Hill, SC

Disagreements may arise if a material, evaluated by means of J1545, is found acceptable where metameric formulations are involved.

### WORKSHOPS (10:30 am to Noon)

Registrants rotate to third round of workshops, as assigned.

### (Afternoon)

### GENERAL SESSION (1:30 to 3:30 pm)

"Multi-Angle Measurement for Metallic Colors"—Thomas Keane, Pacific Scien-

tific Co., Gardner/Neotec Div., Silver Spring, MD

Since metallic colors are sensitive to the viewing angle, it may be advantageous to compute data by using more than one instrument geometry.

"Statistics of Measuring"—Robert Marcus, Macbeth Div., Kollmorgen Corp., Newburgh, NY

Discussed are the basic statistical quantities relative to SAE J1545, along with a description of statistical criteria needed to obtain sufficient precision in the color measurements.

"Implementation"—A wrap-up session which includes a panel discussion on automotive utilization and a summary of the workshop visual-instrumental correlation study.

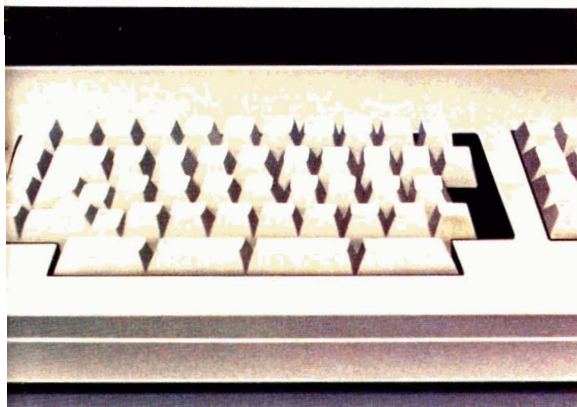
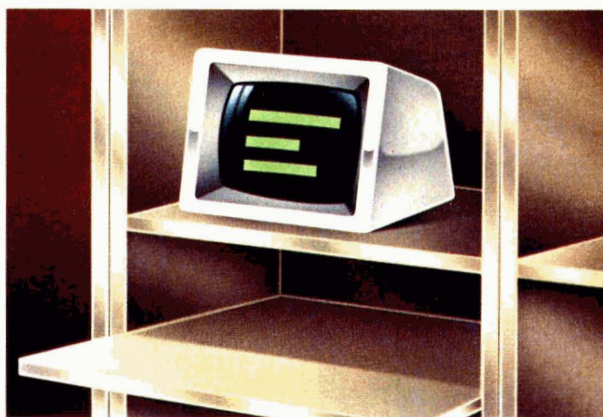
### Special Discount Fares Available from Delta To Annual Meeting

Special arrangements have been made with Delta Airlines to offer discounted fares within the U.S. to/from Atlanta, GA, for the November 5-7 Annual Meeting and Paint Industries Show, at the World Congress Center. These special fares are available only when you call the unlisted toll-free number 1-800-241-6760. Be sure to ask for the lowest fare available. You must give the FSCT Convention number, which is

**U0235**



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our most well known polymer; new RESINS QR-1003 and QR-1004—for improved chemical and solvent resistance; new RESINS QR-1033 and QR-1034—for fast lacquer dry and reduced isocyanate demand; RESIN QR-946 and REACTIVE MODIFIER QM-1007—for high solids coatings.

So get tough. Go with Rohm and Haas for acrylic polyols that come shining through. For samples and technical literature, contact your local Rohm and Haas representative today, or write our Marketing Services Dept., Independence Mall West, Philadelphia, PA 19105.

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# Zeno Wicks Presents 1986 Mattiello Lecture; And Annual Meeting Highlights *(Continued from page 14)*

The theme of the Federation's Annual Meeting, "Compliance and Quality: Recognizing the Opportunities," will focus on the emerging technologies (such as water-based, high-solids, and powder coatings) which are helping the industry meet regulatory requirements. Increased awareness and understanding of potential physiological and toxicological effects of coatings and their raw materials have spurred development of these technologies, which are also responding to increased expectations of quality products, processes, methods, and attitudes.

Program Chairman Percy Pierce, of PPG Industries, Allison Park, PA, and his committee are developing a schedule of presentations, and have announced planned sessions on:

- Powder Coatings
- Statistical Process Control
- Color
- Regulatory Compliance
- Manufacturing Seminar
- Society Papers
- Roon Awards Competition Papers

## Paint Industries' Show

The 51st Paint Industries' Show of the Federation will be held in conjunction with the Annual Meeting at the Georgia World Congress Center, and will feature the products and services of more than 200 suppliers to the coatings industry. Currently, over 96% of available exhibit space is contracted and, with 6,000 industry personnel expected to attend, exhibit hours have been expanded to: 11:00 am - 5:30 pm, on Wednesday, November 5; 9:00 am - 5:30 pm, on Thursday, November 6; and 9:00 am - 3:00 pm, on Friday, November 7.

## Registration Fees

Advance registration is available for \$50 for members and \$65 for non-members. Fee for spouses' activities is \$35 in advance.

Also, there will be a special advance registration fee of \$25 for retired members and their spouses.

On-site registration will be \$60 for full time and \$40 for one day for members. Non-member fees will be \$75 for full time and \$50 for one day. Spouses' activities will be \$45 on-site.

Registration forms were mailed in April and are included in this issue (see pages 23-30).

## Hotels and Reservations

The Federation headquarters hotel will be the Atlanta Marriott Marquis. Other

hotels which have reserved blocks of rooms are: Atlanta Hilton, Atlanta Marriott Downtown, Holiday Inn-Downtown, Hyatt Regency, Omni International, Ritz Carlton, and Westin Peachtree Plaza. All requests for housing must be furnished on the official housing form which has been mailed and is included in this issue (see page 29).

## Special Air Fares

Delta Airlines, in cooperation with the Federation, is offering a special discount fare which affords passengers a 40% minimum savings off Delta's round trip, undiscounted day coach fares for those who travel to the Annual Meeting on Delta's domestic system. (For travel from Canada the discount minimum is 30%).

To take advantage of this discount, you must:

(1) Leave for Atlanta between November 1-6, 1986.

(2) Stay no longer than 15 days.

(3) Purchase tickets at least seven days prior to departure.

(4) Phone 1-800-241-6760 for reservations. Immediately reference the FSCT file number: **U0235**. (The special discount fares are available only through this number).

Delta also has a variety of other promotion fares, some of which may represent an even greater savings. When phoning for reservations, please ask for the best discount available to your itinerary.

If a travel agent is used, they should place the reservation through the toll-free number to obtain the same fare advantages.

## Government and Industry

### Du Pont Improves Safety Training Program

Du Pont Co., headquartered in Wilmington, DE, has reportedly improved its Safety Training Observation Program (STOP). The text has been updated to provide current safety technology and positive behavior modification techniques. Additionally, videotapes are being added to the program to supplement the self-study workbooks, group discussions, and on-the-job applications.

STOP was designed to teach supervisors observation techniques to identify and prevent unsafe acts which cause more than

90% of industrial injuries. Improved STOP is comprised of seven training units.

### Rohm and Haas Constructs Resins Manufacturing Plant

Plans for a state-of-the-art plant for the manufacturing of acrylic thermoplastic and thermosetting solvent-borne resins have been announced by Rohm and Haas Co., headquartered in Philadelphia, PA.

The plant, located at Bristol, PA, is slated for completion in 1987 and will have a rated capacity in excess of ten million pounds annually.

### Anspec Co. Buys HPLC Equipment from Du Pont

The Anspec Co., Inc., Ann Arbor, MI, has purchased the HPLC equipment line from Du Pont Co., Wilmington, DE. Included in the purchase were the pump, detector, and column heater instruments produced by Du Pont, as well as a new solvent delivery system developed by Du Pont and never introduced on the market. In addition, two patents were also part of the purchase. One is a method for solvent blending, and the other is a method for solvent optimization.

### HunterLab Breaks Ground For Headquarters

Construction of a 61,000 sq ft corporate headquarters facility for Hunter Associates Laboratory, Inc. (HunterLab), Reston, VA, is underway. The new building will be situated next to the current headquarters building which was established in 1978.

HunterLab, a manufacturer of instruments for measuring color, gloss, haze, and opacity, was started in 1952 by Richard S. Hunter, currently Chairman of the firm.



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siding refinish.

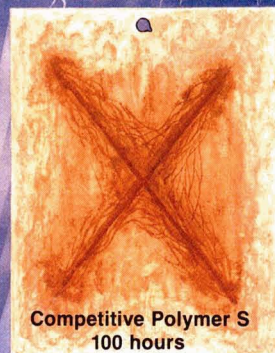
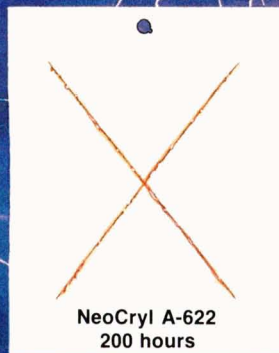
NeoCryl A-622 is precoalesced so it's ready to go to work. Formulators tell us, "We love it. It's so easy to use in our plant!"

For test information, call (800) 225-0947 (in MA (617) 658-6600), or write Dept. GR2, 730 Main Street, Wilmington, MA 01887. Telex RCA 200240.



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FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY



## **Housing and Advance Registration Forms**

Georgia World Congress Center • Atlanta, Georgia  
November 5, 6, 7, 1986

1986 Annual Meeting  
Paint Industries' Show  
November 5, 6, 7  
Georgia World Congress Center  
Atlanta, Georgia



TO OUR MEMBERS AND FRIENDS:

It is a pleasure for me to invite all those associated with the coatings manufacturing industry to attend the Federation's 64th Annual Meeting and 51st Paint Industries' Show at the magnificent Georgia World Congress Center, in Atlanta, November 5-7.

It has been six years since the AM & PS have been in the Peachtree City. The twin events have grown appreciably since, especially the Paint Show which will be 50% larger than in 1980. Attendance has also risen about 20%—to 6,000.

Complementing and running concurrently with the exhibits will be the Program Sessions under the theme—"Compliance and Quality: Recognizing the Opportunities." Presentations will highlight the new technologies which

are helping the industry meet regulatory requirements.

Please come to Atlanta to see the largest and finest exhibit of materials, equipment, and services for paint and coating manufacturers—and—to attend program sessions which will help you to better understand and prepare for the future.

William Mirick

President, FSCT

**DR. PERCY PIERCE, OF PITTSBURGH,  
IS CHAIRMAN OF PROGRAM COMMITTEE**

The Chairman of this year's Annual Meeting Program Committee is Dr. Percy E. Pierce, of PPG Industries, Inc., Allison Park, PA. Seven other members of the Federation working with him are: William A. Wentworth, Vice-Chairman, of Jones-Blair Co., Dallas; Granville D. Edwards, of Shell Chemical Co., Houston; Loren W. Hill, of Monsanto Polymer Products Co., Springfield, MA; Thomas Hill, of Pratt & Lambert, Inc., Buffalo; George R. Pilcher, of Hanna Chemical Coatings Co., Columbus, OH; Ralph Stanziola, Color Consultant, from Bridgewater, NJ; and Robert Thomas, of PPG Industries, Inc., Barberton, OH.

**JAMES E. GEIGER, OF SOUTHERN,  
IS CHAIRMAN OF HOST COMMITTEE**

The Southern Society of the Federation will serve as official host for the 1986 Annual Meeting and Paint Show. Heading the Host Committee is James E. Geiger, of Sun Coatings, Inc., Largo, FL. The subcommittee chairmen are: Spouses—Mrs. James E. (Lynne) Geiger; Program Operations—Thad T. Broome, of Precision Paint Corp., Atlanta; Registration Area—Ronald R. Brown, of Unocal Chemicals Div., Charlotte, NC; Federation Exhibit—Dan M. Dixon, of Engelhard Kaolin Co., Gordon, GA; and Information Services—Berger C. Justen, of Justen & Associates, Tampa, FL.



**FEDERATION OF SOCIETIES FOR COATINGS TECHNOLOGY  
1986 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW  
GEORGIA WORLD CONGRESS CENTER, ATLANTA, GEORGIA  
WEDNESDAY, THURSDAY, AND FRIDAY, NOVEMBER 5, 6, 7**

The Combined Annual Meeting and Paint Industries' Show Is a Major Educational Activity of the Federation. This Significant Coatings Manufacturing Industry Event Consists of Three Days of Technical Program Sessions and Exhibits, Running Concurrently.

The outstanding papers being readied for presentation at the Annual Meeting will be centered about this year's theme—"Compliance and Quality: Recognizing the Opportunities."

The theme focuses upon emerging technologies (such as water-based, high-solids, and powder coatings) which are helping the industry meet regulatory requirements. Increased awareness and understanding of potential physiological and toxicological effects of coatings and their raw materials have spurred development of these technologies, which are also responding to increased expectations of quality products, processes, methods, and attitudes.

Speakers will come from throughout the world of coatings science.

The Paint Industries' Show—biggest and best of its kind in the world—is an international show featuring attractive exhibitor displays devoted to a wide variety of raw materials, production equipment, containers, laboratory apparatus and testing devices, and services furnished to the paint and coatings manufacturing industry.

Key personnel from coatings manufacturers in the U.S., Canada, and several other countries attend annually. The Paint Show gives them the opportunity to learn of the latest developments in the industry and to discuss them with the top technical/sales staffs of the exhibitors. . . of which there will be more than 225 this year.

Show hours will be: Wednesday—11:30-5:30; Thursday—9:00-5:30; and Friday—9:00-3:00.

Eight hotels in downtown Atlanta have reserved blocks of rooms for the Federation. A map showing the location of the hotels and a schedule of rates are included in this brochure. All room rates are subject to an 8% City and Occupancy Tax.

All reservations will be processed by the FSCT Housing Bureau, 233 Peachtree St. NE, Suite 2000, Atlanta, GA 30043.

Confirmations of reservations will come to you directly from the hotel to which you have been assigned. Changes and cancellations—prior to October 5—must be submitted in writing to the Housing Bureau at the above address.

After October 5, please direct all inquiries regarding reservations directly to the hotel, the phone numbers of which are (Area Code 404):

Marriott Marquis	521-0000	Atlanta Marriott	659-6500
Atlanta Hilton	659-2000	Holiday Inn	659-2727
Hyatt Regency	577-1234	Omni International	659-0000
Westin Peachtree	659-1400	Ritz-Carlton	659-0400

Requests for accommodations at the Marriott Marquis will be limited to six rooms per company. A parlor counts as one room.

Reservations for the Atlanta Hilton will be accepted for arrival beginning Wednesday, November 5, only. Any reservations requesting the Hilton prior to Wednesday will be assigned to another hotel.

Most hotels require deposits. Please read your confirmation carefully. If a deposit is required, mail it directly to the hotel.

The phone number of the FSCT Housing Bureau in Atlanta is: 404-521-6630. Telex 804357.

The Board of Directors of the Federation will meet on Tuesday, November 4, at 9:00 a.m. in the Marriott Marquis Hotel. Luncheon is included.

# HOTEL ROOM AND SUITE RATES <sup>1</sup>

No. on Map	Hotel	Singles	Doubles	Suites
1.	<b>Marriott Marquis<sup>2</sup></b> (FSCT Headquarters)	88/98/108 150 Concierge	108/118/128 170 Concierge	\$275 & up
2.	<b>Hilton &amp; Towers<sup>3</sup></b>	82/92/102 135 Towers	102/112/122 155 Towers	250 & up
3.	<b>Marriott Downtown</b>	79	90	225 & up
4.	<b>Holiday Inn</b>	68	68	—
5.	<b>Hyatt Regency</b>	82/92/102	104/108/116	225 & up
6.	<b>Omni International</b>	88/98/108	108/118/128	250 & up
7.	<b>Ritz Carlton</b>	99/109	119/129	350 & up
8.	<b>Westin Peachtree Plaza</b>	80/90/100	90/100/110	250 & up

(1) All room rates are subject to an 8% City and Occupancy Tax.

(2) Requests for accommodations at the Marriott Marquis will be limited to six rooms per company. A parlor counts as one room.

(3) Reservations for the Atlanta Hilton will be accepted for arrival beginning Wednesday, November 5, only.

Note: Most hotels require deposits. Please read your confirmation carefully. If a deposit is required, please mail directly to the hotel.





**FSCT 1986 ANNUAL MEETING AND PAINT INDUSTRIES' SHOW**  
**GEORGIA WORLD CONGRESS CENTER, ATLANTA, GEORGIA**  
**WEDNESDAY, THURSDAY, AND FRIDAY, NOVEMBER 5, 6, 7**

APPLICATION FOR HOTEL ACCOMMODATIONS

**MAIL FSCT Housing Bureau**  
**TO: 233 Peachtree St. NE #2000**  
**Atlanta, GA 30043**

Please indicate below the type of accommodations requested and choice of hotels. All reservations will be processed by the Housing Bureau of the Atlanta Convention & Visitors Bureau. Hotel assignments will be made in accordance with prevailing availability. The confirmation of your reservation will come to you directly from the hotel to which you have been assigned. Changes and cancellations—prior to October 5—must be submitted in writing to the Housing Bureau at the above address. After October 5, please direct all inquiries to the hotel (phone numbers in this brochure).

All reservations will be held until 6:00 p.m. and none can be guaranteed after October 5.

Single (1 person)		
Double (2 persons)		
Twin (2 persons)		
Suite (parlor and 1 bedroom)		
Suite (parlor and 2 bedrooms)		

1st
2nd
3rd
4th

PERSONAL INFORMATION AND DATES OF ARRIVAL OR DEPARTURE

Type of Room	Name	Dates	
		Arrive	Depart

Please Type Additional Reservations on a Separate Sheet and Attach to This Form

PERSONAL INFORMATION FOR ALL RESERVATIONS TO:

Name \_\_\_\_\_ Telephone \_\_\_\_\_

Company \_\_\_\_\_

Address \_\_\_\_\_

City \_\_\_\_\_ State or Province \_\_\_\_\_

Country \_\_\_\_\_ Mailing Code \_\_\_\_\_

Name of Credit Card and # \_\_\_\_\_ Exp. Date \_\_\_\_\_ Signature \_\_\_\_\_

**Note:** Requests for accommodations at the Marriott Marquis will be limited to six rooms per company. A parlor counts as one room. Reservations for the Atlanta Hilton will be accepted for arrival beginning Wednesday, November 5, only.



The 51st Annual Paint Industries' Show will be the largest ever sponsored by the Federation

#### FLY DELTA AIRLINES TO ATLANTA AT A 40% SAVINGS IN FARES

Delta Airlines, in cooperation with the Federation of Societies for Coatings Technology, is offering a special discount fare which affords passengers a 40% minimum savings off Delta's round trip, undiscounted day coach fares for those who travel to the AM&PS on Delta's domestic system. (For travel from Canada the discount minimum is 30%).

To take advantage of this discount, you must:

1. Leave for Atlanta between November 1-6, 1986.
2. Stay no longer than 15 days.
3. Purchase tickets at least seven days prior to departure.
4. Phone 1-800-241-6760 for reservations. Immediately reference the FSCT File Number: **U0235**. (The special discount fares are available only through this number).

*Note:* Delta also has a variety of other promotion fares, some of which may represent an even greater savings. When you phone for reservations, please ask for the best discount applicable to your itinerary.

If you use a travel agent, have them place your reservation through the toll-free number to obtain the same fare advantages.

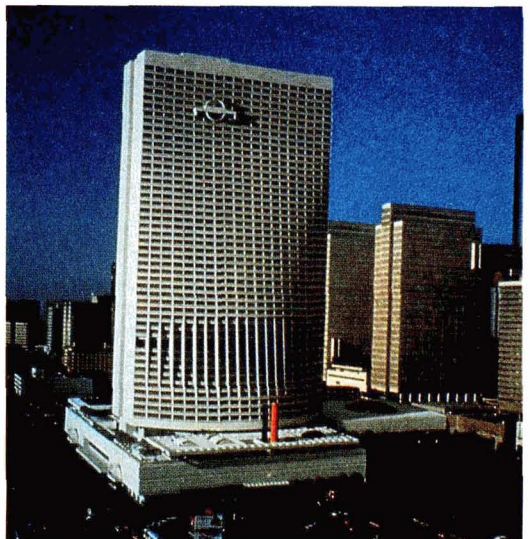
Delta's fully-trained meeting specialists will assist you or your travel agent in making reservations. They will handle every call regardless of the airline needed or desired. Delta will maintain a transportation desk in the World Congress Center during the Federation's AM&PS.

#### FEDERATION AWARDS LUNCHEON TO BE HELD FRIDAY AT GWCC

The annual Federation Awards Luncheon will be held on Friday at the World Congress Center.

#### NPCA MEETS SAME WEEK AT ATLANTA HILTON

The National Paint and Coatings Association will hold its annual meeting on November 3-5, 1986, at the Atlanta Hilton Hotel. Persons wearing NPCA badges (who sign up at a special registration desk) will be admitted to the Paint Show on Wednesday only, with the compliments of the Federation.



FSCT Headquarters hotel, Marriott Marquis, is the newest in Atlanta





# EXHIBITORS SIGNED UP FOR 1986 PAINT INDUSTRIES' SHOW

(As of April 7, 1986)

Aceto Chemical Co., Inc.  
Advanced Coating Technologies  
Air Products & Chemicals, Inc.  
Alcan Powders & Pigments  
C. M. Ambrose Co.  
American Cyanamid Co.  
American Hoechst Corp.  
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American Chemical Society  
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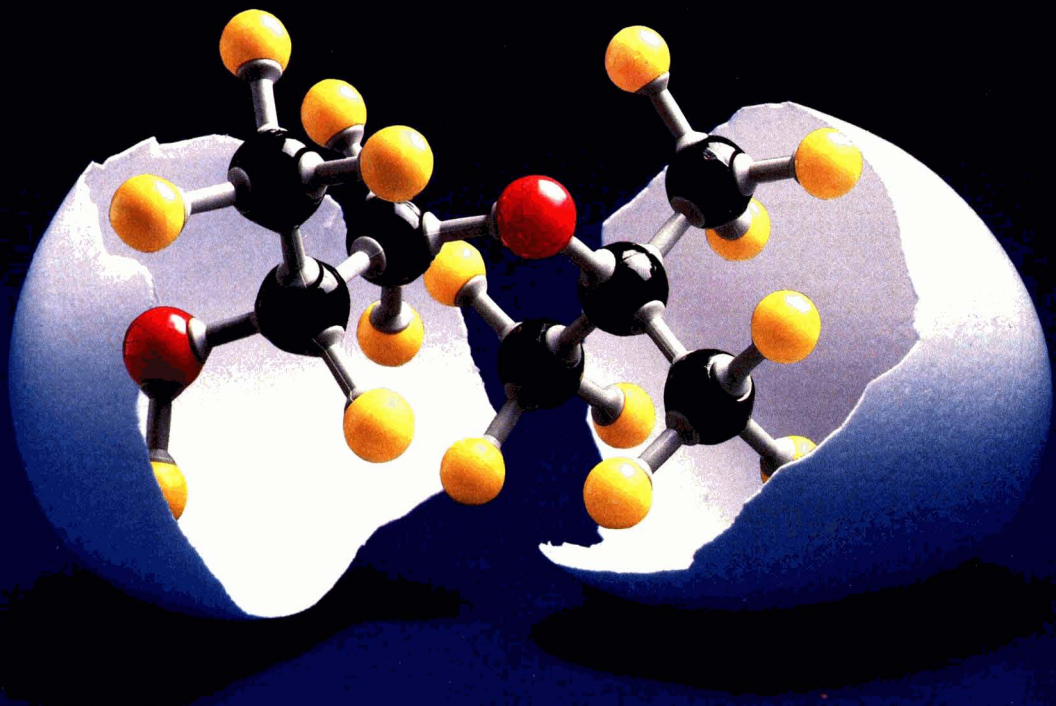
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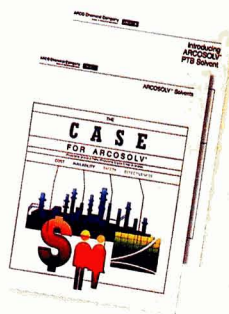
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## Ruth M. Johnston-Feller

Ruth Johnston-Feller has more than 30 years' experience in industrial color technology. Before joining CIBA-GEIGY in September 1973, she was Director of Application Services of the Color Systems Division of the Kollmorgen Corporation. Prior to joining Kollmorgen in 1967, she was Project Leader for Color Research at the Coatings and Resins Division of PPG Industries, Inc. She was one of the first in America to introduce instrumental color control in the manufacture of paint.

Author of more than 45 technical articles, Mrs. Johnston-Feller contributed the major chapter on "Color Theory" in the three-volume *Pigment Handbook*, edited by Temple Patton and published by J. Wiley & Sons. She has taught numerous courses on color and the behavior of colorants and has lectured on color science at many universities.

Ruth Johnston-Feller attended Carthage College and is a graduate in Chemistry from the University of Illinois where she worked for the Chemistry Department in the development of spectrophotometric techniques. During World War II, she was associated with the Penicillin Research Project at the U. S. Department of Agriculture's Northern Regional Research Laboratory.

Mrs. Johnston-Feller, the former Ruth M. Johnston, is presently consultant in color science to the Research Center on the Materials of the Artist and Conservator at Mellon Institute, Carnegie-Mellon University, Pittsburgh, where she directs the

color-science laboratory and the research program on fading of paints and textiles. The Research Center, which receives its basic support from the Andrew W. Mellon Foundation, is directed by her husband, Dr. Robert L. Feller, who, for 35 years, has been engaged in research on new methods and materials for the preservation of artistic and historic works.

Before joining her husband in Pittsburgh in 1975, Mrs. Johnston-Feller was Manager of the Coatings and Colorimetry Laboratories of the Pigment Department of CIBA-GEIGY Corp. in Ardsley, NY, where she was responsible for the testing and evaluation of pigments for the coatings industry and for supervision of the Colorimetry Laboratory's work on the application of color technology in the plastics, paint, textile, and paper industries.

In 1970 Ruth Johnston-Feller received the Federation's Armin J. Bruning Award for her outstanding contributions to the science of color. In 1977, and again in 1978, Mrs. Johnston-Feller and her associate at CIBA-GEIGY, Dennis Osmer, were awarded the Dry Color Manufacturer's Award for the year's outstanding publications in the *JOURNAL OF COATINGS TECHNOLOGY* on the subject of color science as applied to the paint and coatings field. Their two award-winning publications concerned the quantitative evaluation of various aspects of appearance changes of paints following exterior exposure. In 1984, Mrs. Johnston-Feller received the Inter-Society Color Council's Macbeth Award in recognition of her outstanding recent contributions to the subject of color.

Affiliation with the work of the Center at Mellon Institute has given Mrs. Johnston-Feller the opportunity to expand her research interests in the evaluation and quantification of colorant fading. In a series of papers with Robert Feller and Catherine Bailie, she has documented the way that both transparent and opaque paints fade in a predictable manner. It is this latter work which is the subject of her Mattiello Lecture.

Mrs. Johnston-Feller has given many years of dedicated service to the Federation. A former member of the Board of Directors, she has served as a member of the Publications Committee, the Professional Development Committee, and of the Editorial Review Board of the *JOURNAL OF COATINGS TECHNOLOGY*, and continues to

work on the Definitions Committee. She served for several years as a member of the Board of Trustees of the Paint Research Institute.

Much of her service to the Federation was associated with color science. For many years she was Chairman of the Federation's Inter-Society Color Council Committee and Chairman of the FSCT delegation to the Inter-Society Color Council, as well as Chairman of the Bruning Award Committee. She assisted the FSCT Annual Meeting Program Committees, arranging many sessions of particular interest to the ladies as well as to the men of the Federation. She coordinated the work of the ISCC Committee of the Federation in contributing definitions of more than 400 color terms in the *Paint/Coatings Dictionary*, 1978, a section that was published separately in 1979 as the *Glossary of Color Terms*. She is past Chairman of the Problems Committee of the Inter-Society Color Council.

In addition to the Federation and the Inter-Society Color Council, Mrs. Johnston-Feller is a member of numerous other professional societies: American Chemical Society, American Society of Testing and Materials, American Association of Museums, Optical Society of America, the American Institute for Conservation of Historic and Artistic Works, and the International Institute for Conservation of Historic and Artistic Works. She is listed in *American Men and Women of Science*.





# Reflections on the Phenomenon of Fading

Ruth M. Johnston-Feller  
Mellon Institute, Carnegie-Mellon University\*

Evaluation of the degree of fading on exposed paints by visual means alone obscures the underlying physical behavior of the pigments. It is shown that "Mother Nature is logical" and that, when hiding is complete, and the dispersion is uniform, the rate of fading is constant and is not appreciably affected by

many of the variables of paint formulation, such as pigment volume concentration. On this basis, the observed visual changes are also predictable. When hiding is incomplete, however, the rate of fading increases and is dependent on the nature of the substrate. Illustrations of these phenomena are presented.

*Before addressing the subject of fading I would like to pay tribute to the many friends and to the suppliers to the coatings industry who, each year, put on the wonderful Paint Industries' Show. The event is always informative and is often entertaining as well. I have a special appreciation for the effort required, having worked for suppliers—I can remember the rush to get the booth set up, to get everything working, and then the long, tiring hours of standing. But I also remember the fun of it and the enlightenment of learning about problems that need answers. It's a wonderful forum for learning for supplier and user alike. Happy 50th birthday, Paint Industries' Show.—R.M. J-F.*

## INTRODUCTION

Many of the beautiful colors which provide us with enjoyment in our surroundings will ultimately fade and deteriorate. The purpose of these remarks is to describe a technique for quantifying the degree and rate of fading of colored material during exposure to light or other deleterious conditions. Visually we observe the change and particularly so when we can compare the color to the original, unexposed material. However, because our visual system is integrative rather than analytical, we cannot quantify the degree of observed change in physical units (see *Figure 1*). By making spectrophotometric measurements and by using color-matching equations, we can determine the amount of a pigment that remains after

exposure by computing the pigment concentrations necessary to match it in its faded state.

In previous publications,<sup>1,2</sup> a method was described for quantifying the changes in different appearance attributes, such as the amount of specular reflectance (gloss), degree of chalking, pigment fading, and vehicle yellowing, following exposure. The techniques to be described here apply only to the pigment fading. Corrections for the other changes in appearance must first be made for reasonable results to be achieved.

## HISTORY

Back in the late 50's and early 60's, the first color-matching computer, COMIC, an analog computer developed by Davidson and Hemmendinger,<sup>3</sup> came into use. It occurred to Bob Feller and to me (Ruth Johnston at that time), that if the Kubelka-Munk equation could be used to compute a pigment formulation for a measured color which we desired to match, it might also be used to determine the pigment composition necessary to "match" a measured faded paint color. So we tried it and got a clue as to what appeared to be happening in the course of fading.

In the intervening years, we worked somewhat sporadically on the problem and studied a few of the variables. After we were married in 1975, and I was back in Pittsburgh, we set about to study the problem more concerted-ly and systematically. Most of the results reported here have been obtained during these past ten years.

\*4400 Fifth Ave., Pittsburgh, PA 15213

Presented at the 63rd Annual Meeting of the Federation of Societies for Coatings Technology, in St. Louis, MO, October 9, 1985.

The photograph in Figure 1 in the article, "Reflections on the Phenomenon of Fading," by Ruth M. Johnston-Feller, (JCT May, p.34) was reversed. Original alizarin concentration should have been displayed in the left column of chips, evidenced by darker shades of color, with the right column of chips reflecting the faded state.

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cause of the presence of some purpurin—1,2,4-trihydroxy-anthraquinone—which is less stable to light. The purpurin fluoresces a bright yellow-red, providing a means for distinguishing the natural madder from the synthetic variety. Natural madder lakes were also made from other *Rubia* plants. The lake made from *Rubia Peregrina*, which contains a high proportion of pseudopurpurin, 1,2,4-trihydroxyanthraquinone-3-carboxylic acid, is more stable than the alizarin lake.<sup>4</sup> The natural madder lake sold by art suppliers today may be of this type.

Feller identified the presence of madder lake in the beautiful Fayum portrait, shown in *Figure 2*, painted sometime in the third century, A.D. Fayum, a region in Egypt southwest of Cairo, was under Roman domination

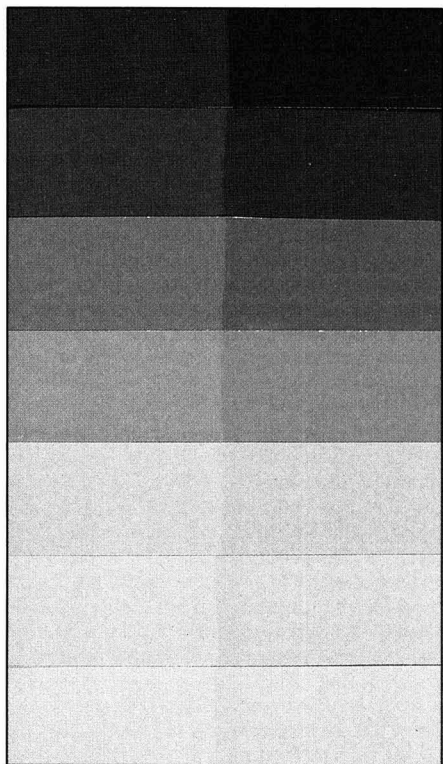
(The original color of the portrait, a number of other Fayum portraits of women were similarly altered.) This sophisticated "personality portrait" was painted more than 1200 years before the Mona Lisa yet she appears just as modern. It is in the collection of the lovely Toledo (Ohio) Museum of Art.

Returning to our choice of alizarin lake as a major pigment in our studies, a second reason for its selection is that it is still used by artists today who love its high chroma and transparency and, most of all, its unique red hue unmatched by any modern red pigments. Alizarin was the first natural dyestuff to be synthesized successfully, by a pair of German chemists in 1868.<sup>5</sup> Much of the alizarin used since that time is the synthetic variety, although natural madder lake is still available today.

A further reason for choosing alizarin lake for our fading studies is that it is only marginal in lightfastness relative to modern organic pigments. Hence, fading results could be achieved in a reasonable period of time. The specific pigment we used is X-686 from Imperial, then Hercules, and now CIBA-GEIGY.

Our early work with COMIC indicated that, regardless of the ratio of alizarin lake to titanium dioxide—that is, regardless of whether the mixture was light or dark in color—the same percentage of the colorant originally present faded during a specific period of exposure in a xenon-arc Fade-ometer<sup>®</sup>, using Pyrex<sup>®</sup> filters. Thus, if a paint with 5.0% alizarin lake and 95% TiO<sub>2</sub> lost 10% of the lake in 100 hours of exposure, leaving 4.5% unchanged lake in the faded paint, it was found that a paint panel containing 50% alizarin lake-50% TiO<sub>2</sub> would also lose 10% of the colorant originally present during the same period of exposure, leaving an alizarin-lake concentration of 45% in the faded film. Moreover, we also found that we had to add some yellow—which was not there originally—in order to match the faded samples. We suspected that the alizarin red lake was going through a yellow intermediate form before becoming colorless. To a physical chemist, this behavior suggests a first-order behavior, with an intermediate, or a consecutive reaction involving the formation of the yellow,  $A \rightarrow Y \rightarrow C$ , where A denotes the original alizarin, Y denotes the yellow intermediate, and C represents the final colorless form.

A search of the paint literature at that time revealed only one article attempting to treat the fading of paints analytically. Haug, in 1952, proposed that pigments in paints faded according to a first-order rate, but his data were so poor as to cast considerable doubt.<sup>6</sup> In contrast, the fact that molecularly-dispersed dyes in gelatin, methyl ethyl cellulose, or collodion, frequently faded according to first-order behavior had been demonstrated by Baxter, Giles, and coworkers in the late 1950's.<sup>7,8</sup> They



\* Figure 1—Can you tell by visual evaluation the fraction of the original alizarin concentration in the left column of color chips that remains in the corresponding color chips on the right?



used the Beer-Bouguer Law in determining the concentration of the dyestuffs remaining. A few years later Kortum used the Kubelka-Munk K/S function to follow the thermally-induced fading of photo-darkened dyes absorbed on various substrates.<sup>9</sup> Subsequently, the Kubelka-Munk K/S function was used to demonstrate that light-induced fading of dyes in pharmaceutical tablets followed the first-order behavior.<sup>10</sup> Incidentally, in the late 50's and 60's, Rushton and his coworkers showed that rhodopsin, the light-sensitive pigment in the rods of the eye, and erythrolabe and chlorolabe, light-sensitive pigments in the cones of the eye, bleach and also regenerate according to first-order kinetic rates.<sup>11</sup>

Some of the data and ideas to be presented here have been published previously, but in journals which industrial paint chemists are not likely to see, publications associated with the conservation of art objects.<sup>12-14</sup> These articles included details, Kubelka-Munk equations, for example, which need not be reviewed for the technical readership of the JOURNAL OF COATINGS TECHNOLOGY.

## EXPERIMENTAL

We applied the paints over glass lantern slides so that we could measure them over white and over black substrates to determine their degree of opacity and also so that we could expose them over white and over black backgrounds. The white background used for the exposure was a stack of Millipore® filters which makes a nearly perfect reflector over the near ultraviolet, visible, and near infrared range. The black background used was a matte optical black paint, which absorbed most of the radiation in this range. The backgrounds were not in optical contact with the glass, but were touching the glass.

The vehicle used for these studies was a solvent-dry system based on a poly (vinyl acetate) of moderate molecular weight, Vinac® B-7, a polymer of excellent photochemical stability similar to those sometimes used by art conservators as a vehicle for retouching paintings. The major pigments used were alizarin lake (CIBA-GEIGY X-686) and a rutile titanium dioxide which exhibits low photochemical activity (Ti-Pure® R-960).<sup>15</sup>

Separate dispersions of the TiO<sub>2</sub> and of the alizarin lake were made by ball-milling in a solution of the vehicle. The TiO<sub>2</sub> dispersion was made by weight with 29.24% of the R-960 pigment, 31.13% of Vinac B-7, and 39.6% xylene. The clear base was a mixture of 44.0% Vinac B-7 and 56.0% xylene. The best dispersion of the alizarin lake X-686 was made with 6.95% pigment, 28.56% Vinac B-7, 36.44% xylene, and 28.06% toluene. The alizarin-lake dispersion which we label as poorest was made with 7.25% of alizarin X-686 (different lot), 37.8% of Vinac B-7, 48.1% of xylene, and 6.8% of cellosolve acetate. Blends of these single pigment dispersions were then made to achieve the desired concentrations of each.

Spectrophotometric measurements of the glass panels were made using the "Trilac" recording spectrophotometer (Leres, Alfortville, France) equipped with a small digital computer for 0% and 100% line corrections at 10 nanometer intervals from 400 to 700 nm. Tristimulus calculations and

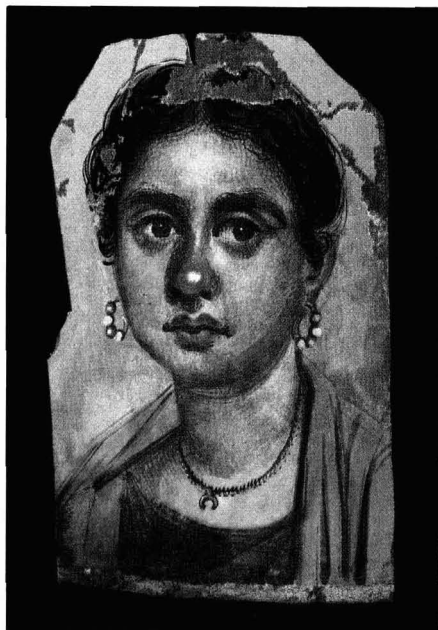


Figure 2—Portrait of a woman, Fayum region of Egypt, painted in the Third Century, A.D. on linden wood. The red pigment in the gown was identified as alizarin-based. (Portrait of a Woman, 71.130, the Toledo Museum of Art, Toledo, Ohio. Gift of Edward Drummond Libbey)

color-difference calculations were based on these 31 wavelength-corrected measurements. Measurements were made with the specular reflectance included (total reflectance) and excluded (diffuse reflectance) to monitor any changes which might occur in the surface reflectance. For some of the samples, the instrument was operated manually to obtain data in the near ultraviolet region, to 360 nm. Spectrophotometric measurements on a few very small samples were made using the Color-Eye® Model 1500.

Depth of fading measurements were made on cross-sections of the paints imbedded in beeswax using a Leitz microscope at a magnification of 275 diameters with a calibrated grid in the eyepiece. Since these measurements of the depth of the faded zone were relatively subjective judgments, several observations were made by several observers and the results averaged.

The only equations presented here are those describing first-order kinetics and those for consecutive first-order reactions.

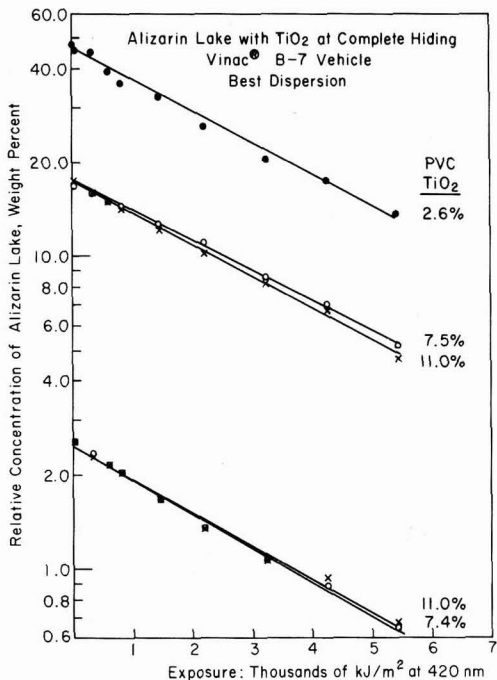
The kinetic first-order rate equation is

$$\frac{dC}{dt} = k_1 C \quad (1)$$

where  $dC/dt$  is the rate of change in concentration,  $C$ , and  $k_1$  is the proportionality constant, i.e., the specific rate constant. Expressed in the integrated form

$$\ln C = \ln C_0 + k_1 t \quad (2)$$

where  $C$  is the concentration at time  $t$ ,  $C_0$  is the initial



**Figure 3**—Alizarin-lake concentration remaining plotted on the log-based y-axis vs exposure on the linear x-axis. The slope of the lines is indicative of the rate of fading according to first-order kinetics. Note that after about 5400 kJ/m<sup>2</sup> exposure, about 365 hours, less than 30% of the initial alizarin lake remains. Thus the rate of fading remains constant over a wide range of concentration

concentration, and ln refers to the logarithm to the base, e.<sup>16</sup> For decay reactions, k<sub>1</sub> is negative; for first-order growth, k<sub>1</sub> is positive.

In the case of consecutive reactions, each of which exhibits first-order behavior, there is a second rate constant, k<sub>2</sub>, describing the rate of change of Y to C. This may be represented by



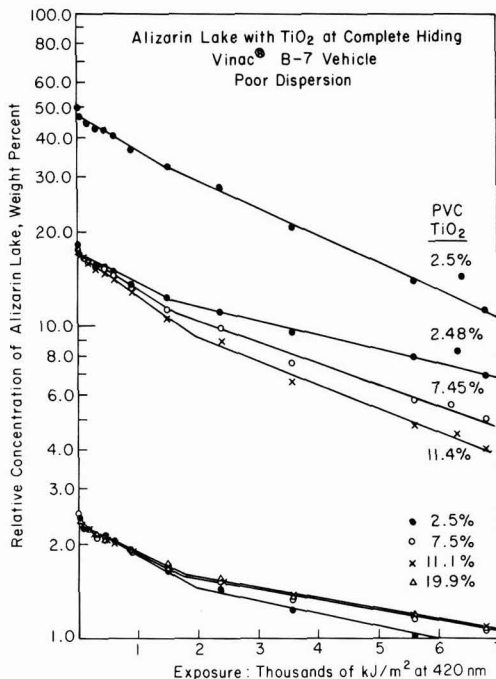
The method for determining k<sub>2</sub> when the absolute concentration of Y is not known is complex and will not be described here.<sup>17</sup>

Unless noted otherwise, the color-matching calculations using the Kubelka-Munk two-constant equations

were used to determine the concentrations of pigments.<sup>18</sup> The Saunderson correction for surface reflectance was used with the first surface reflectance, k<sub>1</sub>, as 0.04 (4%) and the internal surface reflectance, k<sub>2</sub>, as 0.60 (60%).

The panels were exposed in a Model 65-WRC xenon-arc Fade-ometer equipped with Pyrex glass filters which effectively eliminate radiation below 310 nm. The air temperature in the chamber averaged 30.5° to 35°C; the black panel temperature ranged between 59° and 60°C. The ambient air in the chamber remained at approximately 20% relative humidity. Each time the panels were removed for measurement, their position was randomized for the next exposure period. Attempts to raise the humidity to determine the effect of higher humidity on the rate of fading resulted in severe water spotting on the panels and was abandoned.

Because the intensity of the polychromatic irradiance in the Fade-ometer declines with the age of the xenon-arc lamps, the fading rate calculations are expressed in terms of "exposure" rather than time, the exposure expressed in kilojoules per square meter (kJ/m<sup>2</sup>), as determined with a light-monitoring device that measures the intensity of the radiation at 420 nm. During these experiments an average of 14.8 kJ/m<sup>2</sup> was equivalent to one hour of exposure.



**Figure 4**—Rate curves similar to Figure 3 but based on data obtained on a poorer dispersion of alizarin lake. In this case, the rate remained constant only until about 1500 kJ/m<sup>2</sup> exposure, at which point the rate slowed increasingly as the alizarin concentration in the original panels decreased. At the break in the curves, about 30% of the alizarin lake initially present had been lost

**Table 1**—Experimental Design of Alizarin Lake-TiO<sub>2</sub> Mixtures

PVC of TiO <sub>2</sub>	Relative Percent Alizarin Lake		
	2.5	17.5	50
2.5	X	X	X
7.5	X	X	
11	X	X	
20	X		



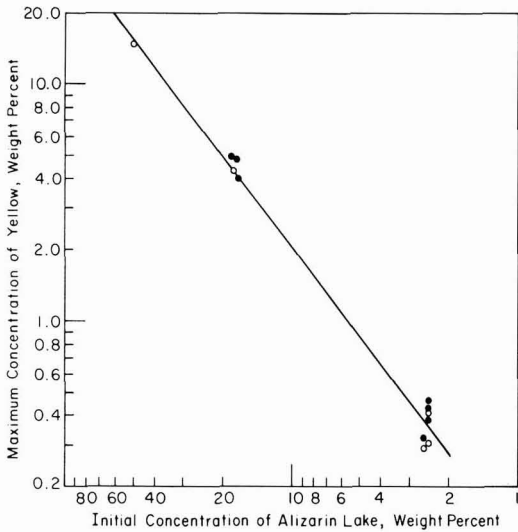


Figure 5—Log-log graph of the maximum amount of the transitory yellow formed vs the initial concentration of alizarin lake, indicating that they are related although not proportional

RESULTS

Throughout this section describing the results on the many samples studied, data measured on representative samples will be illustrated and only summary data included for many of the other panels studied.

Alizarin Lake-TiO<sub>2</sub> Mixtures At Complete Hiding

The variables to be considered in this series of experiments include the relative ratio of alizarin lake to TiO<sub>2</sub>, the pigment volume concentration (PVC) in the dried film, the quality of the alizarin-lake dispersion, the variability between lots of alizarin lake X-686, and measured differences in fading rate between alizarin-lake pigments from other suppliers.

In all of our work in the poly(vinyl acetate) vehicle Vinac B-7, the mixtures at complete hiding were made using a very simple experimental design shown in Table 1. Other samples were made but we tried to include a sufficient number of these eight samples to provide a ready correlation between batches of panels each time.

Equation (2) is the integrated form of the rate equation and tells us that if we plot the concentration-of-alizarin-remaining on the log axis (y axis) of semi-log paper, against the time-of-exposure on the linear axis (x axis), we should get a straight line when behavior is according to first-order kinetics. The slope of the line is equal to k<sub>1</sub>, the specific rate constant of change (fading) in the concentration of alizarin lake. In Figure 3, we see such plots for five mixtures of alizarin mixed with TiO<sub>2</sub> in the Vinac B-7. The relative alizarin-lake concentration is given on the log scale on the left. The exposure, that is "intensity times time," is presented on the x axis, expressed as thousands of kJ/m<sup>2</sup> at 420 nm.

To me this graph is as beautiful as the lovely Fayum portrait because it is telling me that Mother Nature is wonderfully logical and does things in an orderly fashion. Because the lines are parallel, the graph tells us that the rate of fading is a constant whether the amount of alizarin lake is 50% with 50% TiO<sub>2</sub>, as in the case of the top curve of Figure 3, or whether the amount of alizarin lake is 2.5% with 97.5% TiO<sub>2</sub>, as in the case of the two lowest curves. The two lower sets of curves represent paints with the same alizarin/white ratio, but different pigment volume concentrations in the film. At the time we stopped the exposure, after about 5400 kJ/m<sup>2</sup>, a little over 15 days, less than one-third of the initial alizarin remained. The average fading rate constant, k<sub>1</sub> for these samples was  $-2.4 \times 10^{-4}$ . The slope of each curve was calculated by the least-squares method.

(We use a convention in our kinetic studies for our own convenience: the specific rate constant is given a negative sign when it applies to the rate of loss or degradation of the characteristic we are measuring. We use a positive sign for k<sub>1</sub> when it applies to the rate at which a characteristic we are determining increases. For example, we calculated the rate at which the colorless upper layer was forming, and in this case the k<sub>1</sub> for that determination is positive and also first order. We will not describe that work here.)

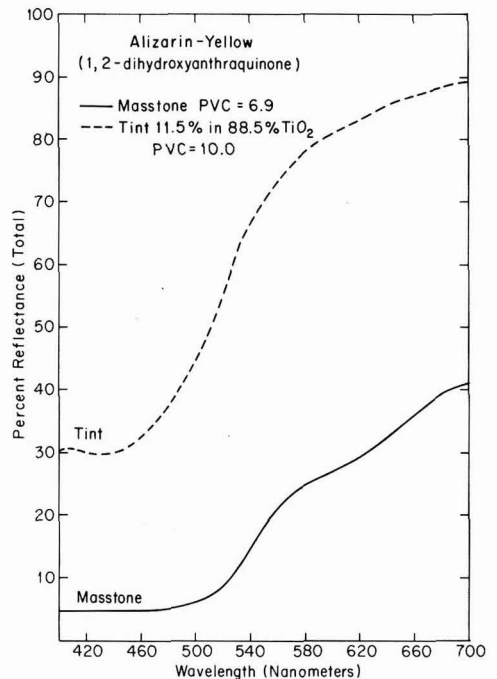
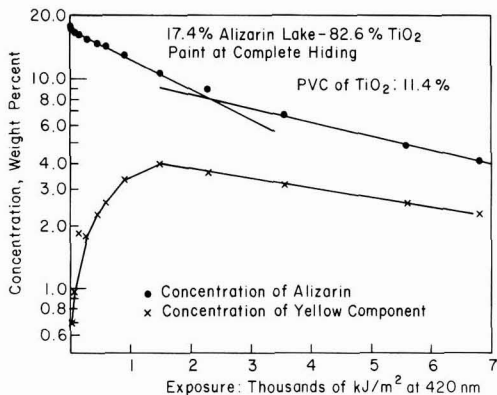


Figure 6—The optical constants used for determining the amount of the transitory yellow were determined from the spectral curves of the pure "unlaked" alizarin. The tint shown here was later exposed in like fashion to the other mixtures and found to fade according to first-order kinetics but much more slowly than the transitory yellow component



**Figure 7—Curves showing loss of alizarin-lake concentration and change in concentration of the transitory yellow vs exposure. The increase to a maximum concentration and subsequent decrease is characteristic of a sequence of reactions, each obeying first-order kinetics involving an intermediate, A → Y → C. This example involves the poorer dispersion of alizarin lake**

The results are not always as neat as those shown in Figure 3. Consider Figure 4. These samples were made from a different dispersion and a different lot of alizarin-lake pigment mixed with the same TiO<sub>2</sub> dispersion. The exposure of this set was continued for almost 7000 kJ/m<sup>2</sup>. The curves appear to have two first-order stages, the first up to 1500 kJ/m<sup>2</sup> followed by a slower second stage. Such a break in the rate curve has been reported by others.<sup>7,9,19,20</sup>

During the initial stage, up to 1500 kJ/m<sup>2</sup>, the reaction constants agree well with those obtained in the series illustrated in Figure 3, i.e., an average value of the slopes in this series was  $-2.6 \times 10^{-4}$ . We suspected that the dispersion was not as uniform in this series because of a difference in handling properties. If this were the case,



**Figure 8—Photomicrograph of a cross-sectioned cut made through a ridge of faded pink paint found in a modern oil painting. The white faded portion at the top and the unfaded pink below illustrate clearly that fading occurs in layers. Magnification is about 10 diameters**

we considered the slower rate in the second stage may have been the result of the presence of some larger particles, which would fade at a slower rate than the smaller particles, a behavior well-known to the paint industry.<sup>21</sup> Fractionation of the X-686 alizarin-lake pigment by sedimentation confirmed this—paints made with the very fine particles faded at the same rate as the initial stage observed here, and the same as observed in the series illustrated in Figure 3, much faster than the paints made with the larger-sized particles that had rapidly settled out of the suspension. The fading rate curves for these larger particles were not linear for very long. The linearity held up to a loss of 10%, however, and the rate constant was  $-0.36 \times 10^{-4}$ , almost an order of magnitude slower than exhibited by the smaller particles.

Even with the series illustrated in Figure 4, however, the first-order behavior was obeyed until 30% of the pigment present originally had faded, extending over a very large visual change. Just how large this color difference is after 30% of the original alizarin lake has faded is illustrated in Figure 1. All of the points on the right side of the pairs contain 30% less alizarin lake than their opposites on the left side. For most applications in the paint industry these differences are very large; so, even though this first-order kinetic behavior interpretation begins to fail after 30% of the colorant has faded, application of kinetic techniques is still practical. And any prediction based on this initial rate will hold over major loss of original color.

One of the effects we have found in all of our panels made with alizarin lake and TiO<sub>2</sub>, when the paints are at complete hiding, is that our color-matching calculations invariably indicated that the exposed panels contained a little yellow not present in the unexposed panels, a hint to us that the red-colored alizarin lake probably passed through a yellow form before becoming colorless. This effect is not surprising because any red colorant, in a sense, has more chromophoric groups in its molecule than a yellow material which in turn has more than a colorless material. That the yellow which developed is associated with the alizarin lake is illustrated in Figure 5, which shows the calculated maximum concentration of yellow component plotted against the initial concentration of alizarin. We have observed this same behavior on other red colorants. For example, carmine red (carminic acid lake) and Fastusol Red (C.I. Direct Red 81, a diazo compound) behave similarly.

We do not know what the yellow substance is and, hence, did not have its optical constants to put into our color-matching computations. We used for the calculation, instead, the optical characteristics of any yellow pigment of moderately high chroma. For the purpose of determining the rate of this transitory yellow's behavior, it doesn't matter, as long as the same spectral character of the yellow is used throughout the calculations of the rate constants. We need to know only relative concentrations. In the case illustrated in Figure 5, we used the calibration curve of pure 1,2-dihydroxyanthraquinone, a yellow compound.

We had used the K and S constants of various other yellows, such as synthetic yellow oxide, but were curious to know whether there was a possibility that the bonds



between the alizarin and the calcium-alumina complex were being attacked first; therefore, we made a dispersion of the pure alizarin chemical and made paint mixtures with  $\text{TiO}_2$ , the same as we had done with the laked pigment. The unlaked alizarin compound was found to be far more lightfast than our transitory yellow by an order of magnitude. It is interesting to note that the pure 1,2-dihydroxyanthraquinone also faded according to first-order behavior with  $k_1 = -7.1 \times 10^{-5}$ . Figure 6 illustrates the spectrophotometric curves of paints made as the masstone and as a tint of the pure chemical.

In two consecutive first-order reactions, the second, intermediate component, starting at zero concentration, first reaches a maximum and then begins to decrease. A typical change in the relative concentration of our transitory yellow is illustrated in Figure 7 for one of the samples displaying the break in the curve. The value of  $k_2$ , the fading of the yellow, was always faster than the value of  $k_1$ . For the sample illustrated in Figure 7,  $k_2$  was  $-8.4 \times 10^{-4}$ .<sup>17</sup> The amount of the transitory yellow component varied depending on the particular alizarin dispersion being used. Thus, much smaller amounts were detected in the samples illustrated in Figure 3, the better dispersion, than in those illustrated in Figure 4, the poorer dispersion.

### Depth of Fading

In paints at complete hiding, fading tends to occur in a layer or zone, as can be seen in Figure 8, which illustrates the change in color with depth when a small chip has been removed from a thick portion of a faded painting. (Please don't think conservators and conservation scientists take great chunks out of the masterpieces they are studying. This is a photomicrograph at a magnification of about  $10\times$ . The tiny "section" was made by the conservator to demonstrate beyond any doubt that this area of the painting had suffered severe fading.)

We measured the depth of fading in our paint samples by microscopically examining a cross-section imbedded in beeswax using a calibrated grid in the eyepiece of the microscope. Because this is a subjective technique, several observers estimated the apparent depth of the faded zone several times and the results were averaged. Figure 9 shows these estimated average depths of fading in terms of the height of the dowels placed on a plane grid with the relative alizarin lake- $\text{TiO}_2$  concentration on one axis and the pigment volume concentration on the other axis. The color of the dowels represents the color of the unexposed samples. As is to be expected, the depth of fading into the film is a function of both the relative concentration of the alizarin and the pigment volume concentration. In other words, it depends on the number of alizarin-lake particles present. Figure 10 is a graph of the depth of fading plotted against the relative number of alizarin-lake particles, calculated as the product of the relative volume of the lake to the  $\text{TiO}_2$  particles multiplied by the total PVC in the film. Depending upon these factors, the depth of the faded zone in this series of test paints varied from about 4 to 40 micrometers.

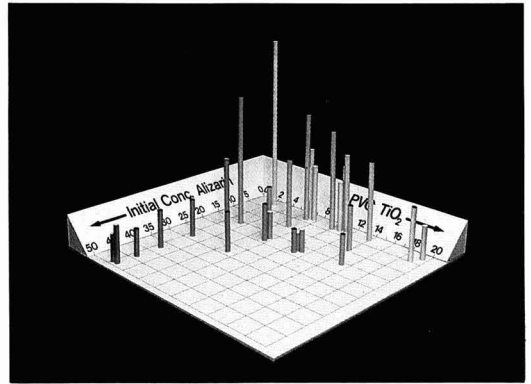


Figure 9—Depth of fading illustrated in terms of the height of the painted dowels placed on a plane whose axes are the relative alizarin- $\text{TiO}_2$  concentrations vs the pigment volume concentration. The color of the dowels represents the color of the unexposed samples

### Alizarin Lake from Different Suppliers

Samples of alizarin-lake pigment, C.I. Pigment Red 83, were obtained from three other suppliers. Two of them faded at about the same rate as the three lots of X-686 (CIBA-GEIGY) (one dating back to 1968). The third faded more quickly initially but the rate leveled off as exposure was continued and then paralleled the others.

### Effect of the Substrate Color

In the case of samples at complete hiding, there was no difference between the rates of fading over the black and over the white substrates. Thus, we see in Figure 3 and in the initial stage of Figure 4 that, as long as hiding is

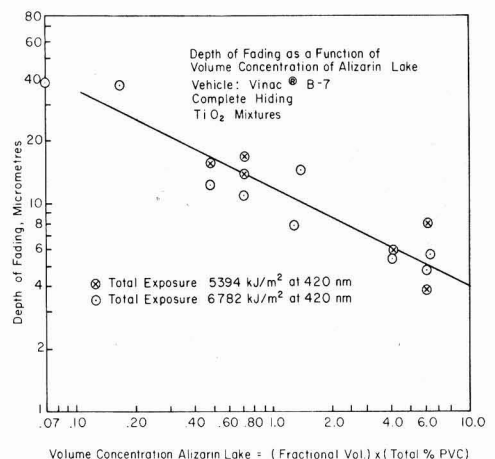


Figure 10—The three-dimensional presentation of the factors affecting the depth of fading into the film (Figure 9) are reduced here to two dimensions by multiplying the volume percentage of alizarin by the total pigment volume concentration of the dried film. Though the precision is poor, the general trend is apparent

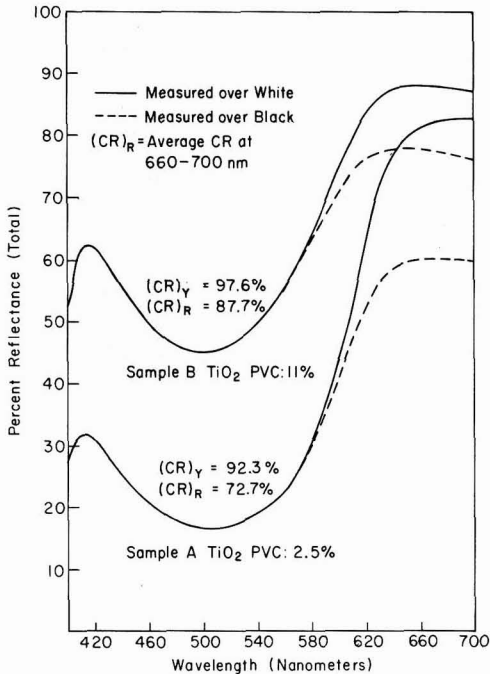


Figure 11—Spectrophotometric curves of two alizarin lake-TiO<sub>2</sub> mixtures that are at incomplete hiding in red wavelength region but at complete hiding throughout the rest of the visible region. CR is the contrast ratio expressed as percent

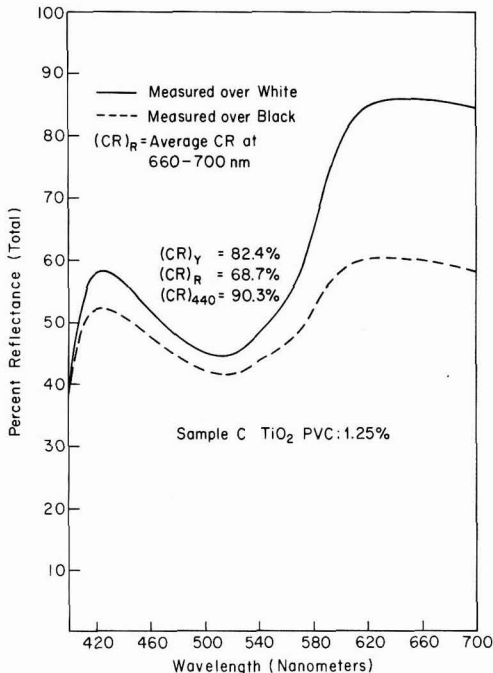


Figure 12—Spectrophotometric curves of an alizarin lake-TiO<sub>2</sub> mixture that is at incomplete hiding at all wavelengths, labeled Sample C

complete and stays that way during the exposure—that is, the paint remains opaque at all wavelengths—the PVC does not affect the rate of fading. But what happens if the paint film does not completely hide the substrate at all wavelengths?

### Incomplete Hiding Samples Of Alizarin Lake-TiO<sub>2</sub> Mixtures

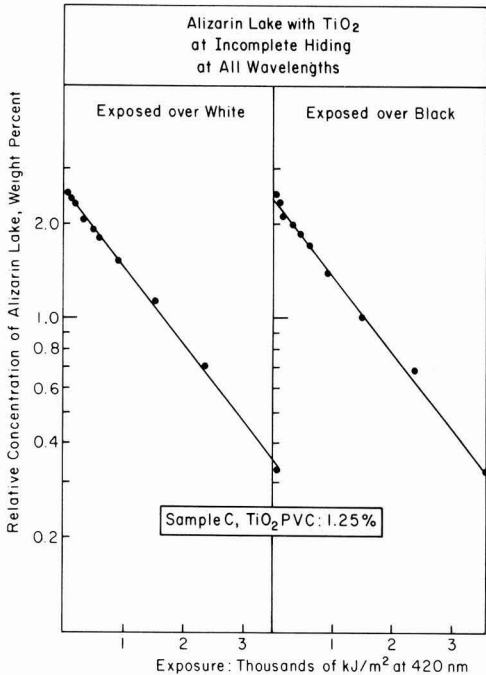
Figure 11 illustrates the spectral curves of two alizarin lake-TiO<sub>2</sub> mixtures which are at incomplete hiding in the red (long wavelength) region of the spectrum, but are at complete hiding in the other regions of the spectrum. The dotted curves were measured over black, the solid curves were measured over white. The CR figures noted on the graphs are the contrast ratios (CR).<sup>22,23</sup> (CR)<sub>Y</sub> is calculated from the Y tristimulus value as measured over black of 0% reflectance divided by the Y tristimulus value as measured over white of 100% reflectance. CR<sub>(R)</sub> is the average contrast ratio calculated from the reflectances measured over black of 0% reflectance and over white of 100% reflectance at 660-700 nm, indicating the degree of incomplete spectrophotometric hiding in the red region of the spectrum.

The rate of fading for these samples exposed over white was 30% faster than the rate at complete spectrophotometric hiding. When exposed over black the rate was 50% faster. Examination of the paint cross-sections in this case showed that fading had not proceeded all the way through the film and that the depth of fading varied with the relative alizarin lake-TiO<sub>2</sub> concentration and the pigment volume concentration in accord with the results on the complete hiding films. The transitory yellow intermediate was also present.

An additional point I might make here is in regard to the concept of "complete hiding": note that sample labeled B, the top pair of curves, which had a contrast ratio of 97.8%, based on the Y tristimulus value, was very close to the figure of 98.0% that is frequently considered to be "complete hiding." It should be evident that visually observed hiding, as well as the degree of hiding calculated from the Y tristimulus value, may be an unreliable indicator of spectrophotometric hiding.

What happens to the fading rate if the paint is at incomplete hiding at all wavelengths across the visible spectrum? The spectrophotometric curves of such a sample, measured over a white and over a black substrate, are shown in Figure 12. The fading rate curves for this sample, exposed over white and over black, are shown in Figure 13. The steepness of the fading rate lines for both the sample exposed over black and over white indicate the higher fading rate,  $k_1 = -5.6 \times 10^{-4}$ . The specific rate constant is more than twice the average rate for samples at complete spectrophotometric hiding. This sample was made using the poorer dispersion which exhibited a break in the fading rate curve for the complete hiding samples (Figure 4). Note in Figure 13 that there is no break in the curve so that no change in rate occurs in this case where the sample is at incomplete hiding at all wavelengths. Microscopic examination of the cross-sections indicated that fading had taken place uniformly through the sam-





**Figure 13—Fading rate curves for Sample C. Note that the curves are steep and that there is no break in the curve. The rates are the same whether the sample was exposed over white or over black. Fading occurred uniformly throughout the film and no transitory yellow was present**

ples. The transitory yellow was missing in this case as is to be expected because its specific fading rate,  $k_2$ , is always faster than  $k_1$ .

Two conclusions concerning the rate of fading of incompletely hiding samples of alizarin lake- $\text{TiO}_2$  mixtures are apparent:

(1) When spectrophotometric hiding is incomplete at the long wavelength region but complete at the short wavelength visible and near-ultraviolet region, a slight increase in the rate of fading occurs, larger when exposed over the black absorbing substrate than over the white reflective substrate.

(2) When spectrophotometric hiding is incomplete at all wavelengths, the rate of fading increases considerably more. (In the case cited, it was double the average rate for the complete hiding samples.)

#### Glazes: Alizarin Lake with No $\text{TiO}_2$ , at Incomplete Hiding

The next type of sample I would like to describe is made without titanium dioxide or other scattering pigment, generally a dispersion of a single pigment applied at incomplete hiding over a reflective substrate. The artist refers to this type of paint layer as a "glaze." Applied over a white reflective substrate, colors of very high chroma can be made in this manner which cannot be

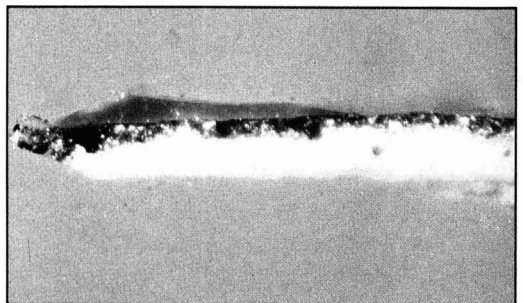
matched by *mixing* pigments in an opaque layer. The early 19th century English landscape painter, J.M.W. Turner, used this technique extensively to achieve his ethereal and unique effects of light and atmosphere, frequently using one thin glaze over another.<sup>24</sup>

Though Turner exploited it so beautifully, the use of transparent colored layers at incomplete hiding was not new. Figure 14 shows a microscopic cross-section of a painting by the incredible Dutch painter of the seventeenth-century, Jan Vermeer. It shows the presence of a copper resinate (green) glaze across the top surface of the chip. This sample came from the *Mistress and the Maid Servant* in the Frick Museum in New York.

What connection does this type of coating have to the paint industry? The answer is important. Paints made with transparent chromatic pigments combined with reflecting flake pigments, loosely called metallic paints, are an analogous system. The reflective flake pigment acts as the reflective substrate and the transparent chromatic colorants are the glaze. Just because the reflective substrate changes its reflectance with the angle of illumination does not negate the analogy.

The glazes that I will describe were simple mixtures of the alizarin-lake dispersion mixed with clear vehicle, applied on glass slides as described and exposed over white and over black substrates as with the other samples. The behavior of three types of samples were selected as illustrative of the variables affecting the rates of fading:

- A. A sample which absorbs all of the violet, blue, green, and yellow region and transmits only red light.
- B. A sample which absorbs all of the light at the absorption maximum (520 nm, green) region but transmits a little short wavelength violet light in addition to long wavelength orange and red light.
- C. A sample which transmits slightly at the absorption maximum, and more violet than sample B in addition to the orange and red light.



**Figure 14—Photomicrograph of a cross-section of a paint chip taken from the "Mistress and the Maid Servant" by Jan Vermeer (17th century), illustrating the use of a transparent color glaze, copper resinate green, over a reflective substrate. Immediately over the white ground-layer there is a very thin brownish-black layer, then the glaze. Magnification 112.5 diameters. (Photomicrograph by H. Kühn, reproduced courtesy of the Frick Museum of Art, New York)**

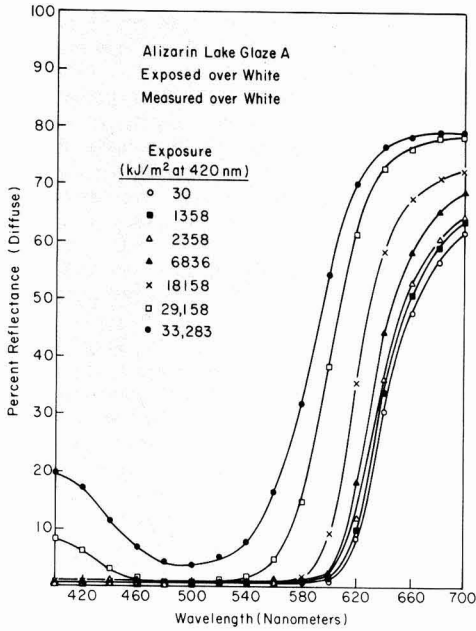


Figure 15—Spectrophotometric reflectance curves of alizarin-lake glaze of high concentration exposed and measured over white. It took about three months of exposure in the Fadeometer® before light of all wavelengths was transmitted. This concentrated glaze which initially transmits only red light is labeled type A

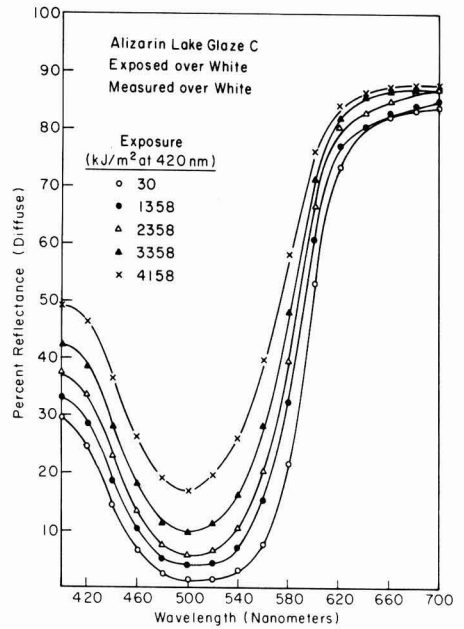


Figure 17—Spectrophotometric reflectance curves of alizarin-lake glaze type C that transmits light at all wavelengths

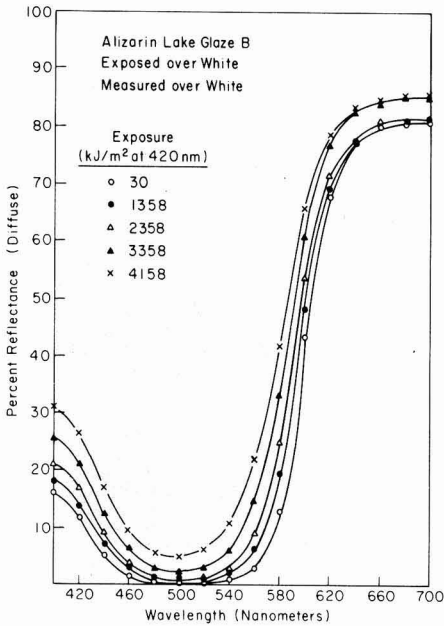


Figure 16—Spectrophotometric reflectance curves of alizarin-lake glaze type B that transmits violet light but initially absorbs completely in the blue-green region where the absorption maximum occurs

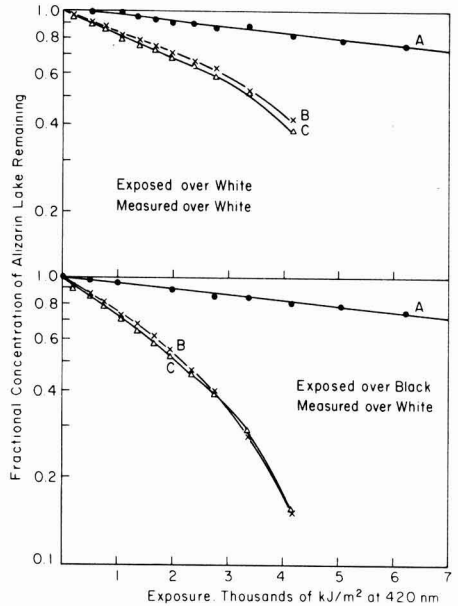
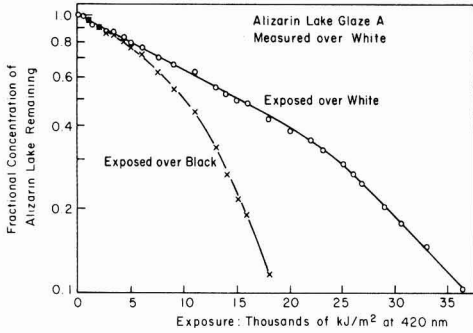


Figure 18—Fading rate curves for glaze types A, B, and C. Note that glaze A faded very slowly but that glazes B and C faded at a much faster rate. Glazes B and C faded faster when exposed over the black substrate than when exposed over the white reflective substrate



**Figure 19—Fading rate curve of glaze A extended to about 36,000 kJ/m<sup>2</sup> (approximately three and a half months in the Fade-ometer). At this point about 10% of the original pigment remained in the panel exposed over white. When exposed over black, it took about half as long to reach this same concentration (approximately one and three-fourths months)**

Spectrophotometric curves of these three samples exposed and measured over white are presented in Figures 15, 16, and 17.

The concentration analysis on the glazes could not be carried out readily as was done on the samples mixed with white. Instead, we resorted to the use of only those wavelengths where there was measurable transmittance and used single-constant Kubelka-Munk theory. Since these samples measured near-zero reflectance over black, the scattering coefficient could be ignored. This method is valid because there is no wavelength where the absorption coefficient is equal to zero. In this situation, the concentration is described in terms of the negative log of the internal transmittance,  $\theta$ . For ease in presentation graphically, the fractional amount of the initial concentration remaining is used to plot the rate curves shown in Figure 18 and to calculate the rate constants. In Figure 18 the upper section shows the data for the three samples exposed over white and the lower section shows the data for them when exposed over black.

Several observations can be made from these graphs:

(1) Sample glaze A, opaque at all wavelengths except the red region, faded at an extremely slow rate over both substrates for the length of exposure shown.

(2) Samples B and C, both transmitting some violet light, faded much faster, with fading rate constants over the initial linear portion, to 2758 kJ/m<sup>2</sup>, approaching the rate of fading of the alizarin lake-TiO<sub>2</sub> mixtures at complete hiding,  $-2.4 \times 10^{-4}$ .

(3) After this point the fading rate increasingly speeded up.

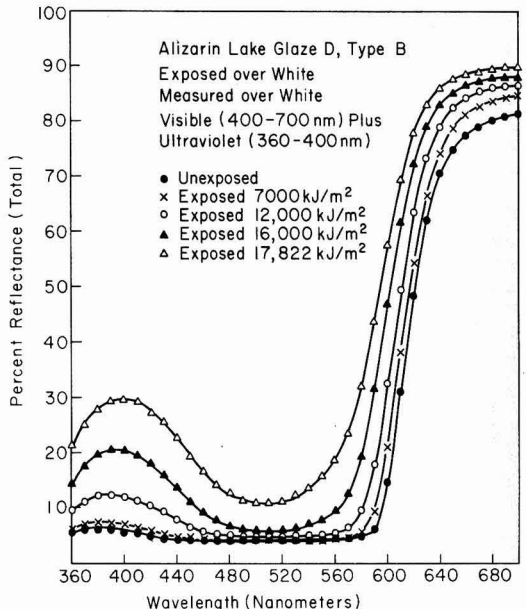
(4) Both samples B and C faded at a faster rate when exposed over black than when exposed over white. Exposure on sample A, the very deep red glaze, was continued well beyond the 4160 kJ/m<sup>2</sup> limit on samples B and C. As illustrated in Figure 19, when exposed long enough, the deeply-colored glaze A also showed an increase in rate of fading, the sample exposed over black doing so much sooner than the sample exposed over white.

To summarize the study of the fading rates of these

glazes: A concentrated glaze of alizarin lake, exposed over a reflective white substrate, fades at an extremely slow rate until enough has faded that short wavelength violet light begins to be transmitted through the sample. Then, the fading rate begins to increase and continues to speed up. The initial rate is very slow but is accounted for in terms of first-order kinetics. The concentrated glaze exposed over black, however, has only a short period when it follows first-order kinetics. Thereafter, it speeds up continuously.

A review of the rates of fading found for samples at incomplete hiding, those with TiO<sub>2</sub>, as well as those without, i.e., the glazes, indicates that transmission of light in the short wavelength region was most damaging to this paint system but that transmission of light in the red long wavelength end is also damaging if the substrate is absorbing, such as the black background used here. When the paint is transmitting short wavelength visible light, it is also transmitting some light in the near ultraviolet, the increasingly actinic wavelength region. Figure 20 illustrates the spectral curve of a glaze of type B measured into the ultraviolet region. The increase in transmittance in the ultraviolet as the fading progressed is readily apparent.

It is logical, therefore, to ask if the rate of fading could be slowed by covering the glaze with a clear layer containing an ultraviolet absorber. Also, if such a layer would protect samples mixed with TiO<sub>2</sub>.



**Figure 20—Spectrophotometric reflectance curves of type B glaze, which initially transmitted only a few percent at 400 nm in the violet, measured in the ultraviolet down to 360 nm at intervals during exposure. Note the increase in ultraviolet transmittance as the exposure continued (as transmittance increases, the reflectance over the white reflective substrate increases)**



### Effect of Protective UV-Filters

We applied a 1.4-mil film of Vinac B-7 containing 0.5% Tinuvin® 328 UV-absorber and 0.5% Tinuvin 770 as a protective filter over three types of samples—(a) a mixture with TiO<sub>2</sub> at complete hiding; (b) a mixture with TiO<sub>2</sub> at incomplete hiding at the red region only; and (c) a glaze of our type A. We exposed these along with uncoated controls. The fading rate results are summarized in Table 2.

In the case of the samples containing titanium dioxide, a reduction in the fading rate of 30 to 40% is shown when exposed under the filter. The rate of fading for the glaze exposed over black under the protective filter was also significantly decreased. However, the rate of fading of the glaze exposed over white under the protective filter was not changed within the experimental error of ±0.03 (at 95% confidence).

In Figure 21, we show the fading rate curves for the glazes; those at the top have no protective layer, and those at the bottom have the protective layer. Note that the glaze exposed over black was protected by the clear UV-absorbing layer such that its rate of fading equaled that of the glaze exposed over white. Note also that the protective layer had no effect on the rate of fading of the glaze sample exposed over white.

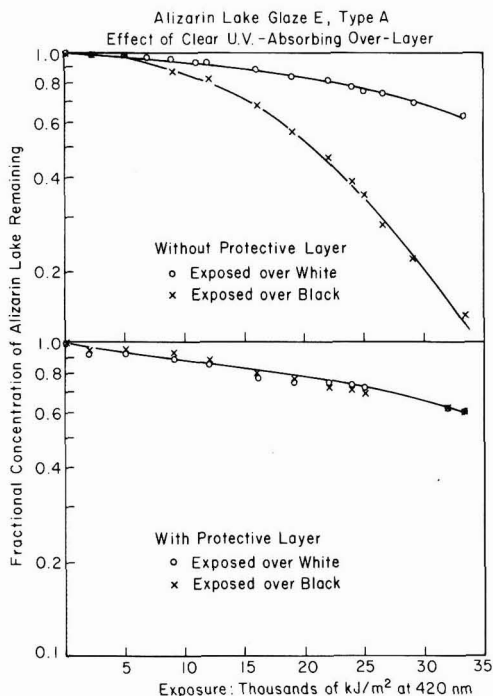


Figure 21—Fading rate curves for a type A glaze with and without a clear 1.4-mil top coat containing 0.5% each of Tinuvin® 328 and 770. Note that the protective layer has no effect on the fading rate of the sample exposed over white—after 33,500 kJ/m<sup>2</sup> about 60% of the original pigment remains in both cases—but that it does provide significant protection for the sample exposed over black

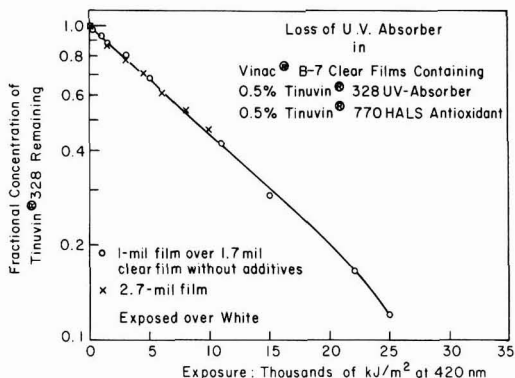


Figure 22—"Fading" rate curve of Tinuvin 328 in clear films of Vinac B-7 containing also Tinuvin 770, each at 0.5%. After 25,000 kJ/m<sup>2</sup> (about two and a half months in the Fade-ometer) only 12% of the Tinuvin 328 remains.

The explanation for this phenomenon is that the Tinuvin 328 UV absorber was also fading and losing its effectiveness. Remember that the white reflective background is a good reflector in the ultraviolet: the Millipore filters we used owe their whiteness to air bubbles. Figure 22 shows the "fading" rate curves of two different layers of the Tinuvin in the clear film exposed over white. Note that the Tinuvin 328 is lost according to a first-order kinetic rate. The calculated rate constants were  $-0.78 \times 10^{-4}$  for the thinner film and  $-0.74 \times 10^{-4}$  for the thicker, much faster than the rate of fading of the glaze exposed over white, about  $-0.1 \times 10^{-4}$ .

The ultraviolet filters of most interest to museums, however, are used to filter the illumination—skylights, fluorescent lamps, etc. Rohm & Haas' Plexiglas® UF-3, which passes no light below 400 nm, i.e., "cuts off" at 400 nm, is typical of the type used. Figures 23 and 24 illustrate the potential effectiveness of installing such filters in reducing the rate of fading of alizarin lake.

### Effect of TiO<sub>2</sub> Ultraviolet Absorption

One might gather that making a tint with TiO<sub>2</sub> increases the rate of fading—after all, the dark glazes didn't fade very rapidly. Some of our early studies made on the fading of alizarin lake compared mixtures of the lake mixed with white lead to those made with TiO<sub>2</sub>, in this case Du Pont's R-610. A photograph of some old panels which had been exposed under "daylight" fluorescent lamps presents the results most vividly (Figure 25). Sample 4a was made with "flake" white (basic lead carbonate); sample 6a was made with TiO<sub>2</sub>. The right half of the series had been exposed under Plexiglas UF-1 UV filter which cuts off at about 400 nm. The UV filter did not have much effect on sample 6b made with titanium dioxide, but provided good protection for samples such as 4b made with the white lead. We can, therefore, conclude that the ultraviolet absorption of the TiO<sub>2</sub> actually does provide protection that helps slow the rate of fading of the alizarin lake.

**Observations Concerning the Fading of Glazes**

When most of us think of fading, we picture in our mind's eye a color that is becoming both lighter and less saturated. Neither of those tendencies is without exception. We are all familiar with the *darkening* that occurs with some chrome yellow pigments following exterior exposure. Less familiar, however, is the phenomenon of increasing chroma during exposure. The alizarin-lake glazes did become lighter and eventually faded to a colorless (white) form. However, along the way the initially dark glazes first increased in chroma until a maximum was reached, after which the chroma steadily decreased towards the neutral point. At about the same time as this maximum chroma was reached, there was an abrupt shift in hue towards the yellow.

This phenomenon is perfectly logical and predictable. The shift towards the yellow in the case of the glazes is not due to the presence of our transitory yellow, but is caused by the change in the shape of the spectrophotometric curve as the concentration of alizarin decreases. The chroma increase is also accounted for in the same way, by a change in spectrophotometric curve shape.

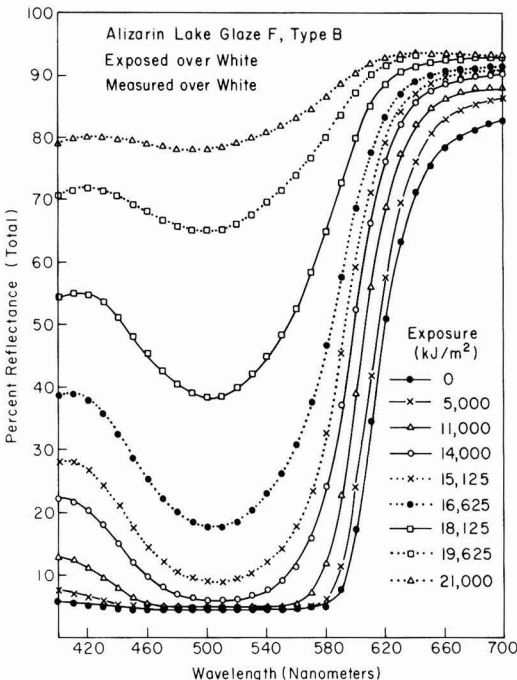
To make this a little more understandable, the spectrophotometric curve of the highest achievable chroma for a red is illustrated by the heavy line in *Figure 26*. Here, all of the light below 620 nm is absorbed (0% reflectance) and all of the light above 620 nm is reflected (100% absorption). If only 90% of the light beyond 620 nm is reflected,

**Table 2—Effect of Ultraviolet Absorbing Over-Layer On the Rate of Fading of Alizarin Glazes Compared with the Rate of Fading Of an Alizarin Lake-TiO<sub>2</sub> Paint at Complete Hiding**

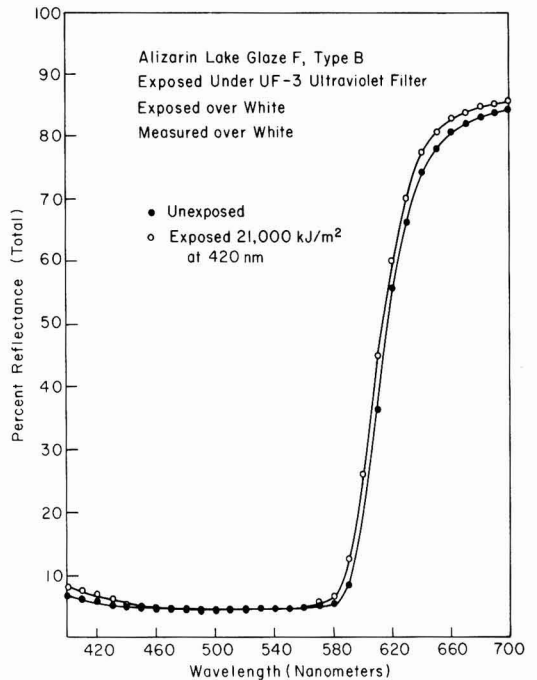
Type <sup>a</sup>	Substrate	Exposure (kJ/m <sup>2</sup> ) at 420 nm	k <sub>f</sub> (× 10 <sup>-4</sup> )	
			Without Protective Filter	With Protective Filter
CH, A + TiO <sub>2</sub>	(White or Black)	8,500	-2.7	-1.6
IH, A + TiO <sub>2</sub>	White	8,500	-3.5	-2.6
IH, A + TiO <sub>2</sub>	Black	8,500	-3.4	-2.2
Glaze Type A	White	22,000	-0.09	-0.12
Glaze Type A	Black	16,000	-0.24	-0.12

(a) (A = Alizarin Lake; CH = Complete Hiding; IH = Incomplete Hiding).

and all of the light below 620 is still absorbed, the chroma, or the saturation, or the purity—all terms of similar meaning—decreases. If the reflectance from 400-620 nm increases to 10%, for example, but the light beyond 620 nm is all reflected (100%), the chroma also goes down. If the light from 400-620 nm is increased to 10% reflectance and the long wavelength light is decreased to 90%, the chroma goes down further. If the long wavelength reflectance is decreased to 50% and the shorter wavelength 400-620 nm reflectance is increased to 50%, there is no chroma—a neutral gray of 50% reflectance results. In other words, the more difference



**Figure 23—Spectrophotometric reflectance curves for Sample F, type B glaze, exposed in the Fade-ometer without protective ultraviolet filter**



**Figure 24—Spectrophotometric reflectance curves for the same glaze as in *Figure 23*, Sample F, exposed under a Plexiglas UF-3 ultraviolet filter**

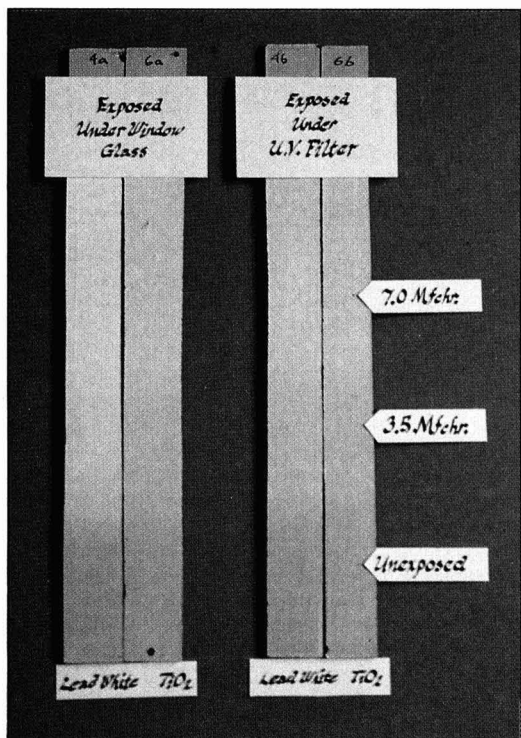


Figure 25—Alizarin lake and white panels exposed to “day-light” fluorescent lamps with and without Plexiglas UF-1 filters. Sample 4 was made with white lead (basic carbonate). Sample 6 with Ti-Pure R-610 titanium white. The samples labeled “a” were not protected by the UV-filter; those labeled “b” were protected. The protection provided by the UV filter for the paint prepared with white lead is roughly equivalent to the protective effect of the UV-absorption of the titanium white pigment

that occurs between the region of maximum absorption and the region of maximum reflectance, the higher the chroma will be.

In Figure 27 we show some spectral curves of our Type B glaze, measured over white and exposed over white, of such thickness and concentration that only a very small portion of short wavelength light is transmitted originally. For reference, we include a different sample from another series which transmits no light at the short wavelength region, sample “a”. (Sample “a” is the 6836 kJ/m<sup>2</sup> exposure from Figure 15.)

Sample “a” reflected almost no light below 600 nm and about 68% at 700 nm; sample 1 also reflected little light below 600 nm, but about 78% at 700 nm; its chroma is higher. Sample 2 reflects little light below 570 nm and about 81% at 700 nm. It reflects more than samples “a” and 1 at 575 nm, which is yellow light; hence, this glaze is both higher in chroma and yellower than the first two. Sample 3 reflects still more yellow and a little more red but only a little violet so its chroma is high and it is yellower than sample 2. Sample 4 reflects only a little more red than 3 but a very much greater amount of blue and violet and even some green; its chroma will be

lower. These results can best be described in Munsell notation; Table 3 presents the data.

In Munsell notation, 10R is the same as zero yellow-red (0YR). The above hue designations thus show the samples moving in the yellow direction as the concentration of the alizarin in the glazes is decreased. Because alizarin lake has its maximum absorption in the blue-green region, where its concentration is indicated, the increase in the Y tristimulus value is a relative indicator of the concentration of the lake. Hence, Munsell Value, determined from the Y tristimulus value, is also an indicator of the alizarin-lake concentration. Figure 28 shows two graphs of the data from Table 3, the change in Hue with changes in Chroma, and the change in Chroma with changes in Value.

One further comment should be made about these alizarin-lake glazes. Note the very high chromas achieved over the white reflective substrate (Table 3 and Figure 28). The highest chroma, 17.3, is a very brilliant color and, as can be seen from the spectral curves in Figure 27, all of the curves have very steep rises to their reflection maxima in the red region. For this reason, information from an abridged spectrophotometer which measures reflectances at 20 nanometer intervals is completely inadequate to characterize these colors. We used 10 nm intervals for determining the Munsell Notation and color-difference calculations and wished we had more data

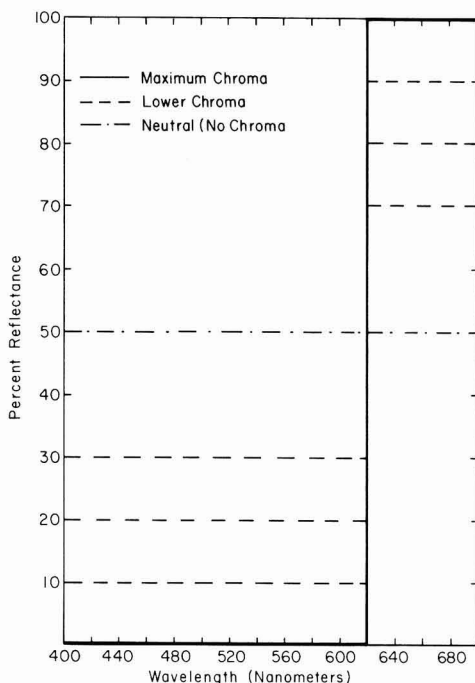


Figure 26—Idealized illustration of the effect of the spectrophotometric curve shape on the perceived saturation or chroma of a red color. The solid line, which shows zero reflectance from 400 to 620 nm and 100% reflectance from 620 to 700 nm, represents the highest possible saturation



points. Further, any spectrophotometer used to measure such high chroma colors must be operated very carefully with good wavelength calibration procedures and 0% and 100% line corrections used. If the reader wonders why we do not present more Munsell information on the many glazes we've studied, it's because the notations had to be determined graphically by hand. Our computer program doesn't calculate Munsell Chromas of 16 and 17. Most of the color-difference equations are also worthless in this region in comparison to visual evaluations, so we have not presented results in these terms either.

That there is a critical concentration (pigment concentration, C, times the thickness, X) at which maximum chroma will occur for any chromatic layer applied at incomplete hiding over any reflective substrate, is a fact for most chromatic pigments. So far as is known to this author, the concept that there is a critical concentration where maximum chroma occurs has not been discussed in the literature. It is accounted for simply by the laws of physics and is the result of the calculable changes which take place in the spectrophotometric reflectance curve shape with changes in concentration (CX) for any defined substrate.

There is a corollary for pigments *in mixture* with white (or other scattering) pigments that applies most vividly to transparent pigments: For paints applied at complete hiding there is a critical concentration in mixture with any

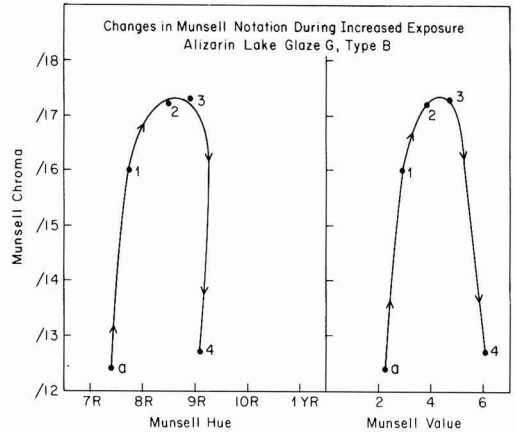


Figure 28—Munsell data plotted for the series of curves of glaze G shown in Figure 27. It can be seen that as the Munsell Value increases with fading, the Chroma first increases to its critical value at Munsell Value of about 4.5 and then decreases. Also note the change in hue towards the yellow at the same time

particular scattering pigment at which maximum chroma will occur.<sup>25</sup> If paints are made with absorbing pigments at concentrations above this critical value, chroma will increase as fading occurs also until this critical value is reached and then decrease as further fading takes place.

These principles are important to those who study the history and care of paintings because they result in some strange distortions in the color balance of old paintings. To illustrate this, my husband painted a portrait of me wearing a blouse with red satin sleeves. He first painted a portrait rather freely and then assigned areas of the blouse to be painted with one of five different concentrations of alizarin glaze. The portrait was then painted "by the numbers" in these glazes on Mylar so it could be exposed over white on the drum of the Fade-ometer. The bodice was painted with an ultramarine blue plus a little black, much more stable pigmentation than alizarin lake. For exposure, we backed the painting with an excellent quality white water color paper. The portrait was removed periodically to be photographed and to be measured in areas representing the five different glaze concentrations. The unexposed portrait is designated a in Figure 29. The photograph after the first period of exposure of 106 hours is designated b, c was taken after 219 hours, d after 286 hours, and e after 393 hours. The last picture, f, was taken after 530 hours (7617 kJ/m<sup>2</sup>). Note that the palest

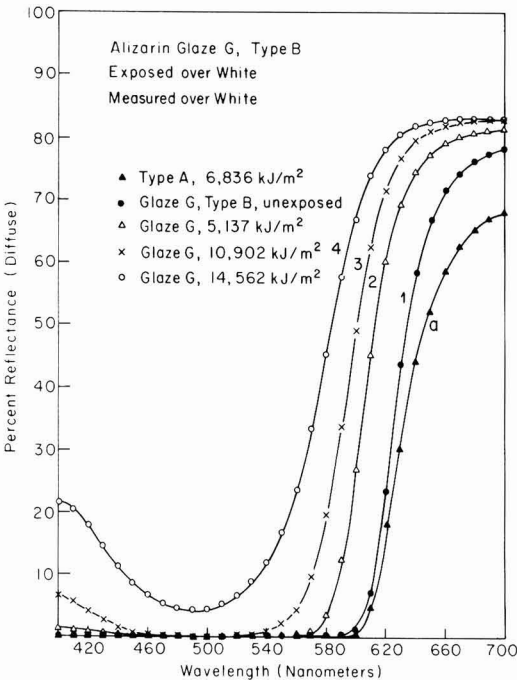


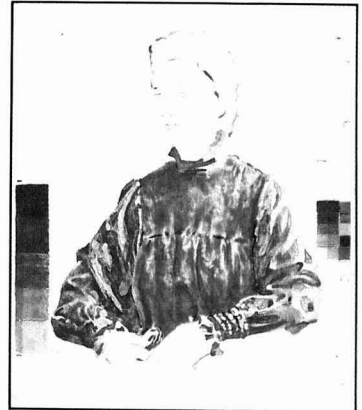
Figure 27—Using the criteria shown in Figure 26, it can be predicted that this glaze, type B, Sample G, will exhibit highest chroma after exposure of 5000 to 10,000 kJ/m<sup>2</sup>. The increased reflectance in the yellow region of these samples also suggests that the hue would be more yellow

Table 3—Munsell Notation for Curves Shown in Figure 27 For a Series of Alizarin Red Glazes (Measured Over White)

Sample	Hue	Value/Chroma
a	7.4R	2.26/12.4
1	7.75R	2.97/16.0
2	8.5R	3.84/17.2
3	8.9R	4.75/17.3
4	9.1R	6.10/12.7



A



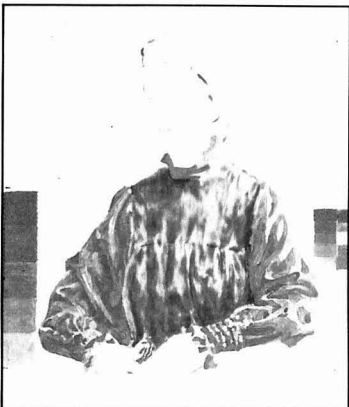
D



B



E



C



F

Figure 29—Portrait painted on Mylar using five concentrations of alizarin-lake glazes for the modeling of the sleeves. Painting was backed with white and mounted around the drum of the Fade-ometer. The unexposed condition is labeled a, condition b shows the painting after 106 hours' exposure, c after 219 hours, d after 286 hours, e after 393 hours, and f after 530 hours (7617 kJ/m<sup>2</sup>). The total exposure is roughly equivalent to 100 years on a museum wall well illuminated by diffuse daylight

tints were almost gone after 393 hours. At the end, the increased chroma of the deepest colors is readily seen in the shadows of the folds when compared to the original. Figure 30 shows a graph of the Munsell chroma versus the Munsell value for the measurements made on the portrait. The behavior is exactly as predicted in Figure 28.

When one looks at old paintings in a museum, one can't help but wonder what they must have looked like when painted originally. The painting shown in Figure 31 is the *Madonna and Child* painted by Zoppo (1433-1478), in the mid-15th century, presently in the collection of the National Gallery of Art in Washington. One may question whether the passage of time has resulted in an apparent distortion of the modeling in the Madonna's red-glazed sleeves in much the same manner as the demonstration portrait.

## DISCUSSION AND CONCLUSIONS

I am not going to reflect at length on the significance of the work I have reported to you here. I would like to emphasize that, while I described our work on the red pigment, alizarin lake, we have found that many other colorants fade in a similar fashion. My major reflection concerns the use of some "back to basics" scientific tools, in this case the use of classical chemical kinetics, combined with classical computer color-matching techniques, to study the phenomenon of colorant fading in an analytical and physically meaningful way.

In our effort to develop more meaningful accelerated weathering procedures, these techniques should be ex-



Figure 31—*Madonna and Child* painted by Zoppo in the mid-15th century. The modeling presently seen in the sleeves may reflect changes similar to those shown in the portrait of Figure 29. (Courtesy of the National Museum of Art in Washington, D.C.)

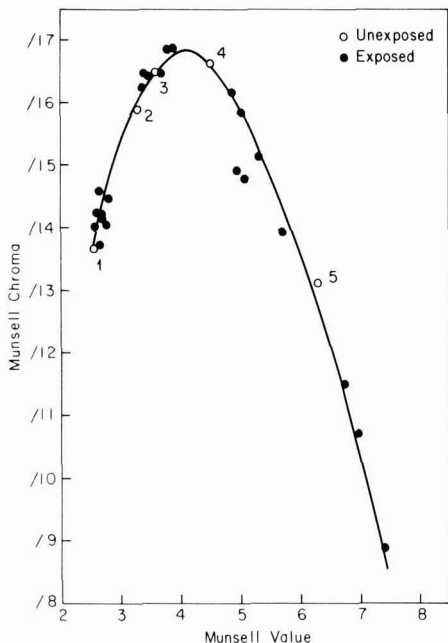


Figure 30—Changes in Munsell Chroma plotted against Munsell Value for the five glazes employed in the portrait of Figure 29

tremely helpful in evaluating and correlating the effect of variables. Applications have been described in the study of several variables that influence the rate of fading. The importance of the degree of spectrophotometric hiding and the nature of the substrate have been particularly emphasized. The techniques described should be extremely helpful in establishing realistic warranties on appearance aspects of coatings in physical terms. They could, in the future, decrease both the time and numbers of panels submitted for exposure evaluations and, one may hope, eliminate a lot of the subjectiveness concerning the stability of pigmented systems.

## ACKNOWLEDGMENTS

I wish to thank the Toledo Museum of Art, Toledo, Ohio, for permission to reproduce the Fayum Portrait (Figure 2), the Frick Museum in New York for use of the cross-section shown in Figure 14, and the National Gallery of Art for allowing me to reproduce the Zoppo *Madonna and Child* (Figure 31).

I cannot express enough thanks to my husband, Robert L. Feller, without whom this work would probably never have been done, for all his help and support. To Mrs. Catherine Baillie, who did most of the laboratory work



described here, I want to express my most heart-felt gratitude. The Research Center on the Materials of the Artist and Conservator at Mellon Institute, which receives its major support through the generosity of the Andrew W. Mellon Foundation, made this work possible by allowing me the use of its equipment and facilities.

Most important, I want to thank the Federation members for their friendship and helpfulness through the years. It has been an honor for me to have been asked to participate in the program for this special Paint Industries' Fiftieth Anniversary Show and I thank you all.

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Color-Eye . . . . . Macbeth Div. of Kollmorgen Corp.  
 Fade-ometer . . . . . Atlas Electric Devices Co.  
 Millipore . . . . . Millipore Corp.  
 Plexiglas . . . . . Rohm and Haas Co.  
 Pyrex . . . . . Corning Glass Works  
 Ti-Pure . . . . . DuPont Co.  
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# Polyvinylacetate Emulsion Design And Rheological Performance In Latex Paints

Binh Dinh Nguyen and Alfred Rudin  
University of Waterloo\*

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An instrumental procedure was used to characterize the rheological behavior and application properties of latex paints and their parent polymer emulsions. Vinyl acetate-dibutyl maleate copolymer latices were polymerized with various particle sizes. Polymerizations were carried out with or without hydroxyethyl cellulose protective colloid or a urethane block copolymer associative thickener. The latices were used to produce semi-gloss (40% PVC) paints.

Latices made without cellulosic polymer in the polymerization step were not thixotropic; those polymerized in the presence of hydroxyethyl cellulose were thixotropic. Latex viscosity increased in the order: (a) latex without cellulosic thickener in the emulsion recipe (non-thixotropic); (b) latex made with thickener in the polymerization (thixotropic); (c) latex (a) with post-added cellulosic (non-thixotropic). Addition of a water-soluble cellulosic thickener to a thixotropic latex increased the viscosity of the system but decreased the thixotropic character.

Paint behavior paralleled that of the original latex. Non-thixotropic latices yielded non-thixotropic paints even when the mill base contained a cellulosic thickener. Thixotropic latices produced thixotropic paints.

Semi-gloss paints made from non-thixotropic latices were more shear-thinning than the corresponding latices. By contrast, paints from thixotropic latices were less shear-thinning than the parent latices. The associative thickener reduced shear-thinning character and promoted flow and levelling.

Differences in latex particle sizes were swamped by the consequences of including or eliminating the water-soluble cellulosic polymer in the emulsion polymerization recipe. Particle size had very little effect on levelling behavior of thixotropic paints. Brushability was also unaffected, except that very small particle size latices promoted brush drag.

Insights into the thickening action of water-soluble cellulose and urethane block copolymer associative thickeners are discussed.

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## INTRODUCTION

The flow properties of a paint are crucially important to its quality. Most latices and latex-based paints are shear-thinning and thixotropic. The term shear-thinning means that the viscosity of the material decreases at faster shearing (or application) rates. Thixotropy refers to behavior in which the viscosity is reduced by shearing and recovers when the liquid is allowed to rest. Thus, the viscosity of a thixotropic material may depend both on the shearing rate and the duration of the particular shearing process.

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\*Department of Chemical Engineering, Waterloo, Ontario, Canada N2L 3G1. Address correspondence to A. Rudin.

Properties such as those mentioned are thought to be influenced by the distribution of latex particle sizes, type and amount of emulsifier, and the nature of other ingredients of the formulation, such as thickeners.

Various thickeners may be used to increase the viscosity of a paint and produce thixotropy and a yield behavior. The yield value or yield stress is the minimum shear stress (shearing force per unit area of material surface) that must be exerted to cause flow. A system exhibiting a yield stress will not flow until the applied shear stress exceeds this minimum value. Anti-sag quality is obviously related to yield behavior.

Here we report the results of a rheological study of the effects of particle size in vinyl acetate-dibutyl maleate

Table 1—Semi-Gloss Paint

Mill Base	Gallons	Pounds
Water	24.00	240.34
Propylene glycol	7.86	83.79
Bactericide	0.10	0.97
Defoamer	0.20	1.96
Surfactants	0.20	2.16
Amine	0.20	1.90
Dispersant	1.60	17.64
TiO <sub>2</sub>	4.67	191.5
CaCO <sub>3</sub>	4.67	126.1
Calcined clay	4.67	121.4
Methocel J5MS (thickener)	0.34	4.7
<b>Let Down</b>		
Coalescent	1.26	11.97
Polymer <sup>a</sup>	39.86	436.5
Water <sup>a</sup>	9.97	99.83
Defoamer	0.20	1.82
Nonionic surfactant	0.20	2.05
	100.00	1344.61
Pigment Volume Conc. (PVC) = 40%		
Film Forming Solids = 35% by volume		

(a) These calculations are for a polyvinyl acetate latex with 56.7% solids and wet density 10.95 lb/gal.

copolymer emulsions and paints. While it has been recognized that all the ingredients of the coating may be important in determining rheological performance, we report here that the composition of the polymerization system may also be of great significance.

Recently, there has been very active interest in the development of new thickeners for water-based systems<sup>1,2</sup> as well as in the mechanism of thickener action in general.<sup>3,4</sup> The rheological assessment of thickener action of this work differs from those of others in that stress is laid here on thixotropic behavior. Thixotropy is considered to be a desirable feature of architectural coatings but it seems not to have been considered very much in studies of the mechanisms of the action of thickeners. The present results provide some new information on the effects of

Table 2—Latex Recipes

	Parts by Weight		
	45.90	45.90	45.90
Vinyl acetate	45.90	45.90	45.90
Dibutyl maleate	8.10	8.10	8.10
Ammonium persulfate	0.13	0.13	0.13
NaHCO <sub>3</sub>	0.10	0.10	0.10
Mixed emulsifier <sup>a</sup>	2.50	2.50	2.50
Natrosol 250 MR <sup>b</sup>	—	0.25	—
QR708 <sup>c</sup>	—	—	0.25
Propylene glycol	—	—	1.00
Water	43.27	43.02	42.02
	100.00	100.00	100.00

(a) Nonionic—Alkasurf NP40 (nonylphenol ethoxylate = 40 moles ethylene oxide (Alkaryl Chemicals Ltd.); anionic = Saponate DS-10 (sodium dodecylbenzene sulfonate; Alcolac, Inc.).  
 (b) Natrosol 250 MR = hydroxyethyl cellulose, which typically has 2.5 moles ethylene oxide per anhydroglucose unit and Brookfield viscosity of 4500-6500 mPas in 2% aqueous solution (Hercules, Inc.).  
 (c) QR708 = a urethane-poly(ethylene oxide) block copolymer associative thickener (Rohm and Haas).<sup>11</sup>

latex composition on the action of several rheology modifier types.

## EXPERIMENTAL

### Emulsion Polymerization

Polymerizations were carried out in a 3L reaction kettle equipped with reflux condenser, two-blade stainless steel stirrer, and two graduated dropping funnels. Deionized water was charged to the kettle and heated to 70°C. Emulsifiers, buffer, and thickeners (if used) were added. Thickeners were predissolved in water (in the case of Hercules Natrosol<sup>®</sup> 250 MR) or in a propylene glycol-water mixture (for Rohm and Haas QR<sup>®</sup> 708) and added gradually, with stirring. The mixture was sparged with N<sub>2</sub> for 30 minutes and then maintained with 200 rpm agitation under a nitrogen atmosphere. Seventy-five percent of the initiator was added 10 minutes before monomer addition was started. The batch temperature was raised to 75°C and the mixed monomers were added to the reactor at a constant rate over 5.5 hours. Monomer addition was at a sufficient rate to keep the reaction mixture at reflux (66°C vapor temperature). Finally, the remainder of the initiator solution was added and the emulsion was cooked for one hour at 85°C. The batch was cooled to room temperature and passed through a 200 mesh filter to remove any coagulum.

### Particle Size Measurements

The particle size distributions of the latices were measured by hydrodynamic chromatography (HDC).<sup>5</sup> Latex samples were diluted to 0.005 to 0.010 percent solids in an aqueous eluant containing 1.0 g/L Aerosol<sup>®</sup> MA (dihexyl sodium sulfosuccinate), 0.526 mM Na<sub>3</sub>PO<sub>4</sub>, and 370 ppm formaldehyde.

HDC columns were packed with Dow 50W cation exchange resin in the sodium form. Calibration was with narrow particle size distribution polystyrene latices. The eluant was monitored with an ultraviolet detector operating at 250 nm wavelength. Elution volumes were reported relative to those of sodium dichromate, which was used as a marker.

Axial dispersion is very prominent in HDC. A correction for this perturbation was applied with a computer program based on the size exclusion chromatography correction method of Ishige, Lee, and Hamielec.<sup>6</sup>

A brief comparison<sup>7</sup> of styrene-butadiene and polyvinyl acetate latices in the 100-200 nm diameter range showed good agreement between HDC and transmission electron microscopy or sedimentation field flow fractionation values.<sup>8</sup>

### Rheological Measurements

Rheological measurements in this study were made with a Contraves Rheomat-30 cup-and-bob viscometer over a shear rate range of 0.09 s<sup>-1</sup> to 662 s<sup>-1</sup>. All samples were characterized at 25.0 ± 0.2°C.

The experimental procedure followed was essentially that of Rudin and Baas,<sup>9</sup> with a few modifications. Materials examined include vinyl acetate-dibutyl maleate la-

Table 3—Latices Polymerized Without Use of Protective Colloid

No.	Nonionic Anionic Ratio	Solids (wt. percent)	Particle Size Distribution (nm)				
			$\bar{D}_N$	$\bar{D}_V$	$\bar{D}_W$	Standard Deviation	Skewness
1	3	56.5	80	95	170	28	5.3
2	7	56.6	80	105	205	38	3.9
3	19	56.5	110	140	230	50	2.2
4	39	56.7	120	160	260	63	1.9
5	99	56.3	190	210	280	59	1.9
6	199	56.8	290	300	340	80	2.2
7	349	56.7	220	240	320	58	3.4

tices polymerized with and without thickeners in the emulsion reaction and semi-gloss paints prepared according to the formulation described below (Table 1).

Before addition of any thickeners, the pH of the latex or paint was adjusted to  $9.0 \pm 0.3$  with concentrated ammonium hydroxide. Two cellulosic thickeners were used. These are Natrosol 250 MR hydroxyethyl cellulose, described later in connection with Table 2, and Methocel® J5MS (Dow Chemical Company). The latter is a hydroxypropyl methylcellulose with 16.5-20% methoxyl and 23-32% hydroxypropyl content. The intrinsic viscosity in water is reported to be 1.2 dL/g at 20°C.<sup>10</sup> Both cellulose thickeners were added as slurries in water. The associative thickener QR 708 (Rohm and Haas Co.<sup>11</sup>) was dissolved in a minimum volume of 10% (w/w) propylene glycol/water. When cellulosic thickeners were added to latices that had been made without protective colloid in the polymerizations, 10 minutes were required to ensure complete hydration.

Approximately 120 mL of sample were poured into the rheometer cup, using system A of the Contraves unit. The bob was then immersed in the cup and attached to the calibrated viscometer. The viscometer was set at the maximum speed setting ( $662 \text{ s}^{-1}$  shear rate in the cup and bob used in this work) and sheared for five minutes to release air bubbles entrapped in the liquid. After shearing, the cup was covered with stretchable polyethylene film to prevent skinning on the surface. The test material

was left to stand for 30 minutes for temperature equilibration and recovery of sheared-out thixotropic structures.

After the recovery period, the polyethylene film was removed and the viscometer shear rate was varied automatically from  $0.9 \text{ s}^{-1}$  to  $6.6 \text{ s}^{-1}$  using the programmable shear rate options of the instrument. The shear rate was then increased from  $9 \text{ s}^{-1}$  to  $662 \text{ s}^{-1}$ , held at the highest value for five minutes, and then decreased progressively to  $9 \text{ s}^{-1}$ .

This procedure provides the characteristic thixotropic loop of the material and the yield value of the unsheared liquid. The unsheared material is defined operationally as the material which has rested for 30 minutes in the rheometer cup after initial shearing at maximum speed.<sup>9</sup> The shearing at  $662 \text{ s}^{-1}$  for five minutes was sufficient to bring the thixotropic shear stress to its equilibrium "sheared" value with all materials tested in this study.

The viscometer was then set to manual control and the liquid was sheared again for five minutes at  $662 \text{ s}^{-1}$ . Twenty seconds after cessation of this shearing, the viscometer was returned to automatic control and the shear rate was increased from  $0.09 \text{ s}^{-1}$  to  $6.6 \text{ s}^{-1}$ . This procedure provides the yield value and low shear rate flow curve of the "sheared" material. The twenty second interval was chosen because levelling of typical coating films is believed to be completed in 10-30 s.<sup>12</sup>

Yield values of the sheared and unsheared materials were estimated by extrapolating the shear rate-shear

Table 4—Latices Polymerized in Presence of Thickeners

No.	Thickener	Nonionic Anionic Ratio	Solids (wt. percent)	Particle Size Distribution (nm)				
				$\bar{D}_N$	$\bar{D}_V$	$\bar{D}_W$	Standard Deviation	Skewness
1	Natrosol 250 MR	3	56.5	90	120	270	42	4.8
2	Natrosol 250 MR	7	57.0	100	130	340	49	5.4
3	Natrosol 250 MR	19	55.3	130	210	540	98	3.4
4	Natrosol 250 MR	39	57.0	140	230	650	106	4.2
5	Natrosol 250 MR	99	56.9	180	290	720	126	4.2
6	Natrosol 250 MR	199	56.4	160	270	650	131	3.2
7	Natrosol 250 MR	349	56.9	150	270	780	127	4.3
8	QR 708	99	56.9	160	190	320	62	3.4



Table 5—Effect of Natrosol 250 MR on Particle Size

Nonionic Anionic Ratio	Emulsion Made without Protective Colloid				Emulsion Made with Natrosol 250 MR			
	$\bar{D}_N$	$\bar{D}_W$	Standard Deviation	Skewness	$\bar{D}_N$	$\bar{D}_W$	Standard Deviation	Skewness
3	80	170	28	5.3	90	270	42	4.8
7	80	205	38	3.9	100	340	49	5.4
19	110	230	50	2.2	130	540	98	3.4
39	120	260	63	1.9	140	650	106	4.2
99	190	280	59	1.9	180	720	126	4.2
199	290	340	80	2.2	160	650	131	3.2
349	220	320	58	3.4	150	780	127	4.3
					Emulsion Made with QR 708			
99	190	280	59	1.9	160	320	62	3.4

stress curve to the shear stress axis. When stress overshoot occurred, only the linear portion of the apparent flow curve was used for extrapolation. The yield values obtained in this fashion are approximate and are intended only for comparison with each other. Unfortunately, we are not aware of any better method for measuring yield stress of materials like those used in this study.

The variation of viscosity at shear rates characteristic of levelling and the low shear rate equilibrium viscosity were also investigated. The sample was sheared out at  $662 \text{ s}^{-1}$  for five minutes and the viscometer was set to rotate at  $0.6 \text{ s}^{-1}$  shear rate. The sample was sheared at this shear rate until an equilibrium shear stress was reached. The  $0.6 \text{ s}^{-1}$  shear rate was chosen because this lies within the range of shear rates estimated to prevail during levelling<sup>13</sup> and because the Contraves rheometer was not sensitive enough to provide good readings at lower shear rates with the materials studied here. For convenience and reproducibility, the time between high speed shearing and low speed data accumulation was kept constant at 15 s.

## Paint

The various polyvinyl acetate latices were used to make a standard semi-gloss paint, with the formulation listed in Table 1.

## RESULTS

### Latex Preparation

Polymerization experiments were carried out according to the three recipes listed in Table 2. Seven latices were made without the use of protective colloid and seven corresponding emulsions were produced with hydroxyethyl cellulose in the polymerization recipe. One latex was made in which the urethane block copolymer associative thickener was used in the polymerization instead of the cellulosic derivative. All latices were stable. No agglomeration was observed during the polymerization reaction. The pH of the final emulsions was about 4 and solids were close to the target 56.5%.

Tables 3 and 4 summarize the particle size distributions and experimentally determined solids contents of latices

made with the recipe in Table 2. In general, the mean particle size increases with increase in the nonionic/anionic surfactant ratio in this recipe. There seems to be an upper limit to particle size, however, at a nonionic/anionic weight ratio of about 200. This is hardly surprising, since the anionic surfactant concentration is already very low under this condition.

Incorporation of hydroxyethyl cellulose in the polymerization reaction increased the breadth of the particle size distribution, and particularly promoted the production of large particles. The number average particle size was not increased to any great extent. The polymerization mixture was also noticeably more viscous at later stages of the reaction in the presence of hydroxyethyl cellulose. The associative thickener QR 708 did not have a significant effect on the sizes of polymer particles that were produced (compare reaction 8 of Table 4 with reaction 5 of Table 3).

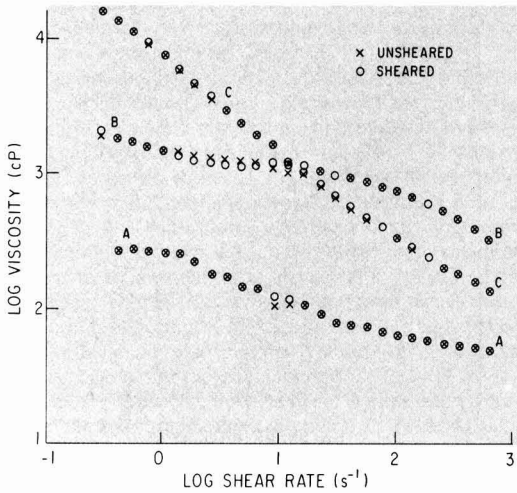
Table 5 summarizes the effects of hydroxyethyl cellulose and the associative thickener on particle sizes, with different anionic/nonionic surfactant ratios.

### Rheological Characterization

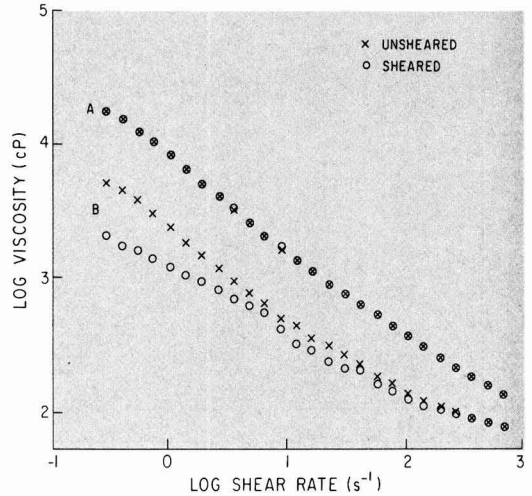
Most of the rheological data plots given here are for the shear rate range  $0.6 \text{ s}^{-1}$  to  $662 \text{ s}^{-1}$ . Extensions of the shear rate range to  $10,000 \text{ s}^{-1}$  showed that the relative viscosities of different formulations at  $660 \text{ s}^{-1}$  were in the same order at  $10,000 \text{ s}^{-1}$ . Essentially all significant thixotropic structures were sheared out at shear rates  $<660 \text{ s}^{-1}$ . Similar observations have been reported by earlier workers.<sup>14</sup>

Table 6 records viscosities at  $662 \text{ s}^{-1}$  and  $9760 \text{ s}^{-1}$  for semi-gloss paints made with latices that were polymerized with thickeners in the reaction mixture (as in Table 4). The sheared and unsheared paints have identical viscosities at these shear rates. It is clear from Table 6 that the ratio of  $9760 \text{ s}^{-1}$  viscosity to the  $662 \text{ s}^{-1}$  viscosity is almost the same for all semi-gloss paints, at least in this series.

Figure 1 shows the log viscosity-log shear rate trace for a typical latex polymerized without the use of hydroxyethyl cellulose protective colloid. The upper curve is the flow curve for this latex to which had been added 0.25 (w/w) % Methocel J5MS hydroxypropyl methyl cellu-



**Figure 1**—Viscosity-shear rate plots for latex made without protective colloid and with nonionic/anionic ratio of 99 (reaction 5 of Table 3). Curves are shown for sheared and unsheared systems consisting of the latex as-polymerized (A); latex plus 1% rheology modifier QR 708 (B); and latex plus 0.25% Methocel J5MS (C). All concentrations are on a (w/w) basis and are based on the wet latex



**Figure 2**—Viscosity-shear rate plot for latex of Figure 1 plus 0.25% Natrosol 250 MR hydroxyethyl cellulose (curve A) and for latex made with nonionic/anionic ratio of 99 and 0.25% Natrosol 250 MR in the polymerization (latex 5 of Table 4) (curve B). Water, 6.7% (w/w), was added to the latex of curve B, to equal the amount of water added with the thickener in the case of curve A

lose. Curves for the sheared and unsheared states superimpose in both cases, showing that this latex has negligible thixotropy. The post-addition of hydroxypropyl methyl cellulose increases the viscosity and makes the material more shear-thinning, but does not confer significant thixotropy on the system. Similar changes were observed when QR 708 associative thickener was used instead of Methocel J5MS. The latex formulation with QR 708 has a higher high shear rate viscosity and lower low shear rate viscosity, for equivalent viscosity at about  $10 \text{ s}^{-1}$ .

Figure 2 shows the flow curves of the corresponding latex polymerized in the presence of 0.25% Natrosol 250 MR. The flow curves for the as-made latex are shown here after addition of 6.7 (w/w) % water. This is the same amount of water as was added after polymerization with Methocel J5MS or Natrosol 250 MR thickeners. The latex made with Natrosol 250 MR in the polymerization is more viscous than its cellulosic-free counterpart (shown in Figure 1 as curve A). The difference is particularly

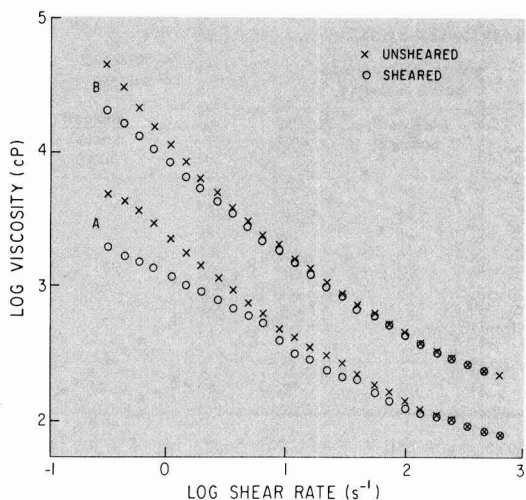
noticeable at low shear rates. Also, the emulsion made with the protective colloid is thixotropic, whereas that polymerized without cellulosic is not.

The viscosity of the latex of Figure 2 (made with cellulosic in the polymerization recipe) is, however, considerably lower than that of a system consisting of the emulsion of Figure 1 (with no cellulosic in the polymerization) to which 0.25% Natrosol 250 MR was added after polymerization. The latter mixture is shown as the upper curve in Figure 2. It will be noticed, also, that post-addition of the hydroxyethyl cellulose to a non-thixotropic latex again did not produce a thixotropic system. Thixotropy is exhibited with these ingredients only if the cellulosic polymer is present in the emulsion polymerization reaction.

To summarize, latex viscosity increases in the order (a) latex made without cellulosic thickener in the emulsion recipe (non-thixotropic); (b) latex made with cellulosic thickener in the polymerization (thixotropic); (c) latex (a) with post-added cellulosic (non-thixotropic).

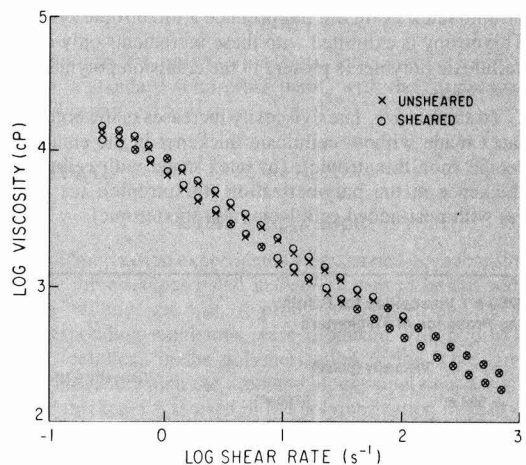
**Table 6**—Correlation of  $662 \text{ s}^{-1}$  and  $9760 \text{ s}^{-1}$  Viscosities for Paints Made with Latices Polymerized in the Presence of Thickeners

Thickener Used in Polymerization	Nonionic Anionic Ratio	Latex Particle Size $\bar{D}_w$ (nm)	Viscosity (poise)		Viscosity Ratio
			$662 \text{ s}^{-1}$	$9760 \text{ s}^{-1}$	
Natrosol 250 MR	7	340	1.9	0.9	0.48
Natrosol 250 MR	19	540	2.0	1.0	0.50
Natrosol 250 MR	39	650	2.6	1.4	0.54
Natrosol 250 MR	99	720	2.2	1.1	0.50
Natrosol 250 MR	199	650	2.4	1.2	0.50
Natrosol 250 MR	349	780	2.1	1.0	0.48
QR 708	99	320	2.1	0.9	0.42

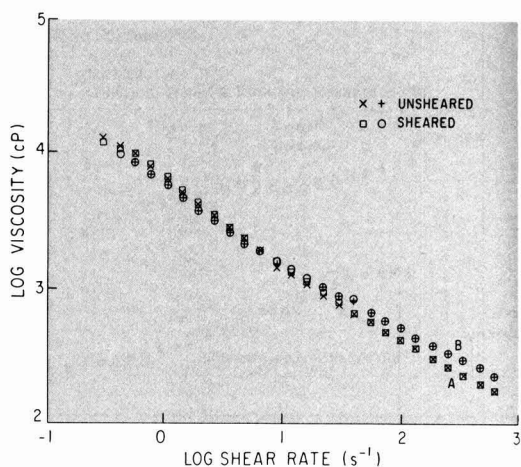


**Figure 3**—Viscosity-shear rate plots for sheared and un-sheared versions of: **A**—latex polymerized with nonionic/anionic ratio of 99 and 0.25% Natrosol 250 MR; **B**—latex **A** plus 0.25% Methocel J5MS. Curve **A** is for the latex plus 6.7% water to be equivalent to the dilution in adding the Methocel in system **B**. All percentages are (w/w) and are based on wet latex

Although the latices made with and without cellulosic in the polymerization differ in particle size, it is believed that the major reason for the greater thickening effect of post-added cellulosic is the higher molecular weight of the latter thickener. It is likely that hydroxyethyl cellulose in the polymerization recipe is degraded by attack of radicals from the initiator and from macroradicals.<sup>15</sup> Furthermore, a substantial fraction of the thickener in the emulsion polymerization recipe may have been consumed in the reaction, with the formation of graft polymers.



**Figure 4**—Viscosity-shear plot for semi-gloss paint made from latex of *Figure 1*. The lower curve is for the paint made according to the formulation of *Table 1*, while the upper curve is for this paint plus 0.165% (w/w) Methocel J5MS, based on wet paint



**Figure 5**—Viscosity-shear rate plot for semi-gloss paint made from latex of *Figure 1* (**A**); plot for the same paint plus 0.33% (w/w) rheology modifier QR 708 (**B**)

Evidence for grafting onto hydroxyethyl cellulose in vinyl acetate polymerizations has been shown by Donescu and coworkers.<sup>15</sup>

A comparison of *Figure 1* and *2* shows also that Natrosol 250 MR and Methocel J5MS produce almost the same flow behavior when added to the non-thixotropic latex of *Figure 1*. The conclusions from this work are therefore probably not strongly affected by our usual use of Natrosol 250 MR in the polymerization and Methocel J5MS in the paint mill base.

Addition of hydroxypropyl methylcellulose (Methocel J5MS) to thixotropic latices generally increased the viscosity but decreased the degree of thixotropy. An example is given in *Figure 3*.

*Figure 4* shows the rheological behavior of a semi-gloss paint made from a latex that was produced without cellulosic in the polymerization step. The paint formulation is given in *Table 1*. Basically, the paint behavior parallels that of the latex in being shear-thinning and non-thixotropic. Addition of Methocel J5MS to this paint (over that used in the mill base) increased the viscosity through the whole shear rate range but did not induce any thixotropic character. The mill base itself was somewhat thixotropic, but this characteristic did not carry over into the paint.

The effects of Methocel J5MS and the associative thickener QR 708 on the viscosity of a non-thixotropic latex were compared in *Figure 1*. Neither additive produced thixotropic behavior but the QR 708 caused much less increase in low shear rate viscosity for a given higher shear rate viscosity. The flow curve for paint made from this non-thixotropic latex and 0.33 (w/w) % QR 708 (based on wet paint) is shown in *Figure 5*. The paint is like the latex in being non-thixotropic. A comparison of *Figures 4* and *5* shows that the addition of Methocel J5MS and QR 708 to the paint has the same effect on the viscosity as the post-addition of these thickeners to the original latex. In both the latex and the paint, more QR 708 than Methocel J5MS is required to achieve a particu-



lar high shear viscosity, but the QR 708 system is less shear-thinning (i.e., has lower low shear rate viscosity) at the same high shear rate viscosity).

Figure 6 compares the effects of Methocel J5MS cellulosic and QR 708 associative thickener on the rheological properties of a latex that was polymerized with hydroxyethyl cellulose in the emulsion recipe. The latex is thixotropic, as shown earlier in Figure 3, and the cellulosic thickener or the nonionic associative thickener both increase the viscosity, with some reduction in thixotropic character at very low shear rates. Thixotropic structures appear to be sheared-out at about  $100 \text{ s}^{-1}$  shear rate in this latex. The QR 708 gives less low shear rate viscosity increase than Methocel J5MS with the same higher shear rate behavior. It also permits retention of more thixotropic character of the original latex.

Figure 7 shows the flow curves of a paint made from a different thixotropic latex (reaction 4 of Table 4) along with the flow curve of the paint to which Methocel J5MS had been added. This latex was polymerized with a different soap ratio from that used for the thixotropic latex of Figure 2, but the particle sizes are quite similar. The cellulosic thickener has increased the viscosity and reduced the thixotropic nature of this paint. This behavior parallels the effects of this cellulosic additive on the rheology of the base latex. The addition of an equivalent concentration of QR 708 (Figure 8) produces a paint with less shear-thinning character and less viscosity increase than that from an equal concentration of Methocel J5MS.

A latex polymerized in the presence of the associative thickener QR 708 (reaction 8 of Table 4) exhibited a small degree of thixotropy. The semi-gloss paint made from this latex was not thixotropic.

Yield values for different systems are listed in the following tables. Table 7 records the values for latices

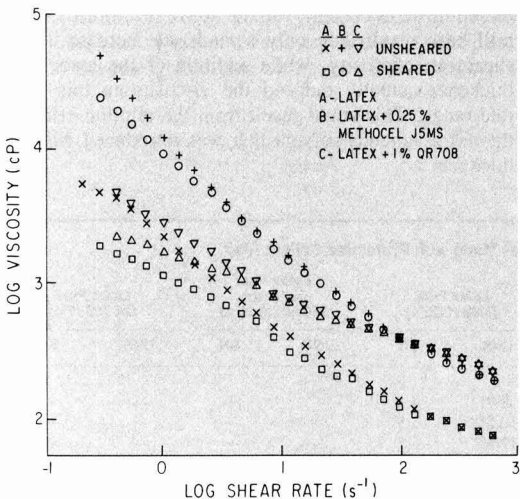


Figure 6—Viscosity-shear rate plots for a thixotropic latex (same latex as in Figure 3) made with cellulosic protective colloid in the emulsion recipe. The data are shown for the as-polymerized latex after dilution with an additional 6.7% water to provide an equivalent polymer concentration to that in the other curves where Methocel J5MS (0.25%) and QR 708 (1%) have been added

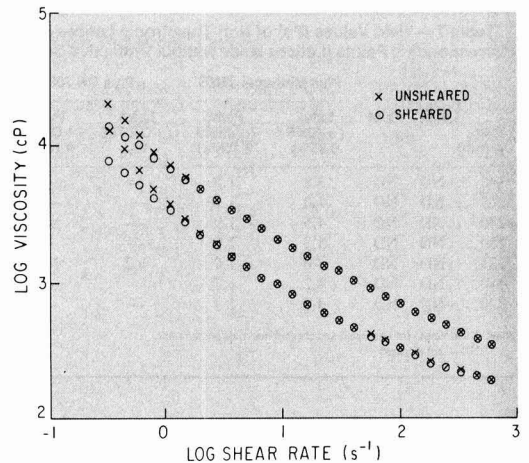


Figure 7—Viscosity-shear rate plots for semi-gloss paint made from thixotropic latex with  $\bar{D}_w$  650 (reaction 4 of Table 4, lower set of symbols) and corresponding plots for paint plus 0.165% extra Methocel J5MS (upper set of symbols). The lower curves are for the paint to which 5% (w/w) water has been added to be equivalent to the dilution in adding thickener

and corresponding points when the emulsion was made without protective colloid in the polymerization reaction: In these cases, the sheared and unsheared materials had the same yield values. The only systems that exhibited yield stresses were those to which hydroxypropyl methyl cellulose was added (over the amount in the mill base, in the case of paint).

Table 8 records yield values for latices that were made with hydroxyethyl cellulose in the emulsion recipe. These thixotropic latices all exhibited yield values in the un-sheared condition. Since the yield stresses are estimated in a rather unsatisfactory manner, we have refrained from

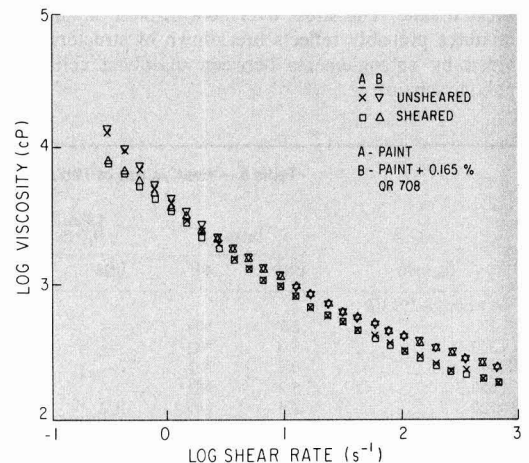


Figure 8—Viscosity-shear rate plot for semi-gloss paint made from thixotropic latex with  $\bar{D}_w$  650 and corresponding plot for paint plus 0.165% QR 708 (upper set of symbols). The lower set of symbols are for paint plus 5% (w/w) water to be equivalent to the dilution in adding the rheology modifier

**Table 7—Yield Values (Pa) of Non-Thixotropic Latices and Corresponding Paints (Latices Made without Protective Colloid)**

Latex $\bar{D}_w$ (nm)	Latex	Paint	Plus Methocel J5MS		Plus QR 708	
			Latex (+ J5MS 0.25%)	Paint (+ J5MS 0.165%)	Latex (+ QR 708 1%)	Paint (+ QR 708 0.165%)
170	ND	ND	3.8	1.5	—	—
205	ND	ND	4.0	1.7	—	—
230	ND	ND	4.5	1.9	—	—
260	ND	ND	5.3	2.0	—	—
280	ND	ND	4.0	1.9	0.2	ND
340	ND	ND	4.2	1.7	—	—
320	ND	ND	3.9	1.7	—	—

Note: Yield values for unsheared and sheared materials are the same.  
ND— not detected

attempts to correlate particle size and the magnitude of the yield value.

Table 9 records yield values of semi-gloss paints made from the thixotropic latices of Table 8. Addition of extra Methocel J5MS to the paint had almost no effect in the yield value. Addition of an equal concentration of the associative thickener QR 708 reduced the yield values in both the sheared and unsheared conditions.

Some paints and latex formulations exhibited a stress overshoot in the unsheared condition. This phenomenon may occur when a material that is initially at rest is suddenly subjected to continuous deformation. The shear stress builds up quickly and then decreases to an equilibrium, lower value. This phenomenon was seen in paints made from latices that were polymerized in the presence of hydroxyethyl cellulose. The original latices themselves did not show stress overshoots. The corresponding paints always contained at least about 1% (on emulsion) of additional hydroxypropyl methylcellulose, from its use in the mill base (Table 1). Addition of hydroxypropyl methyl cellulose to the corresponding latex also resulted in a system that exhibited stress overshoot in the un-sheared state. The stress overshoot in these thixotropic mixtures probably reflects breakdown of structure provided by entanglements between dissolved cellulosic polymer chains.

Typical low shear rate data showing stress overshoot are shown in Figure 9. This should be contrasted with the plot of Figure 10, in which no stress overshoot was observed.

Stress overshoot is generally attributed to the breakdown of associated structures in the system.<sup>16</sup> This explanation appears to fit the present results. Shearing reduced the yield value of all systems.

Some slight pigment settling was observed during storage of all paints made from latices that did not have cellulosic thickeners in the polymerization. This is probably because the materials did not have a yield stress. However, the pigment was easily redispersed, indicating that other factors like surface forces may also be significant.

Low shear rate viscosities are expected to be important in levelling processes. The shear rate operative during levelling has been variously estimated to be in the range 0.1 to 1.0 s<sup>-1</sup>.<sup>13</sup> Our low shear rate measurements were made at 0.6 s<sup>-1</sup> because the particular viscometer did not appear to give reliable readings at lower shear rates with these latices and paints.

Recall that low shear rate viscosities were recorded at 0.6 s<sup>-1</sup> 20 seconds after the cessation of high shear rate stirring and also after the viscosity had reached an equilibrium value at the low shear rate. This is to parallel the levelling and anti-sag properties of a paint.

Equilibrium viscosities were reached very quickly in latices that were polymerized without hydroxyethyl cellulose in the emulsion recipe. Similar behavior was exhibited by the corresponding semi-gloss paints. This is a reflection of the non-thixotropic character of these systems.

Thinning of the paint with moderate amounts of water caused a major reduction in low shear rate viscosity and, presumably, enhanced levelling and flow-out. Addition of cellulosic thickener over and above that already in the mill base resulted in only a moderate increase in low shear rate viscosity, while addition of the associative thickener actually reduced the viscosity in this shear rate range. This could result from the dilution effect of the extra aqueous solvent that was introduced with the thickener.

**Table 8—Yield Values of Thixotropic Latices Made with Protective Colloid (Pa)<sup>a</sup>**

$\bar{D}_w$ (nm)	Latex		Latex Plus H <sub>2</sub> O (6.7%)		Latex Plus J5MS (0.25%)		Latex Plus Natrosol 250 MR (0.25%)		Latex Plus QR 708 (1%)	
	UNS	SH	UNS	SH	UNS	SH	UNS	SH	UNS	SH
<i>Natrosol 250 MR</i>										
270	2.7*	ND	—	—	8.9*	4.5	—	—	—	—
340	3.0	ND	—	—	10.5*	5.5	—	—	—	—
540	6.3	ND	—	—	12.8*	6.5	—	—	—	—
650	5.2	ND	—	—	11.3*	6.0	—	—	—	—
720	3.5	ND	1.2	ND	12.5*	6.0	11.0*	5.5	1.4	0.3
650	4.5	ND	—	—	11.5*	7.0	—	—	—	—
780	4.0	ND	—	—	11.0*	6.0	—	—	—	—

(a) UNS— un-sheared; ND— not detected; SH— sheared.  
(\*) — stress overshoot.

Table 9—Yield Values (Pa) of Paints Made from Thixotropic Latices

	Latex D <sub>w</sub> (nm)	Unmodified Paint		Latex Plus H <sub>2</sub> O (5%)		Latex Plus J5MS (0.165%)		Latex Plus QR 708 (0.165%)		Latex Plus QR 708 (0.165%) J5MS (0.083%)	
		UNS <sup>a</sup>	SH	UNS	SH	UNS	SH	UNS	SH	UNS	SH
Natrosol 250 MR	340	5.0	3.0	—	—	5.5	3.4	3.1	1.7	4.0	2.0
Natrosol 250 MR	540	5.1	3.1	—	—	5.3	3.3	—	—	—	—
Natrosol 250 MR	650	5.5	3.4	3.4	1.9	5.8	3.6	3.4	2.0	—	—
Natrosol 250 MR	720	5.1	3.1	—	—	5.3	3.3	3.1	1.7	—	—
Natrosol 250 MR	650	5.1	3.1	3.2	1.5	5.3	3.3	—	—	—	—
Natrosol 250 MR	780	5.0	3.0	—	—	5.4	3.3	—	—	—	—
QR 708	320	4.8	4.2	—	—	4.9	4.4	—	—	—	—
Commercial PVAC	—	4.5	2.2	—	—	5.0	2.9	3.1	1.6	3.8	1.8
Commercial acrylic	—	1.2	0.8	—	—	1.4	1.0	—	—	—	—

(a) UNS—unsheared; SH—sheared.

Low shear rate (0.6 s<sup>-1</sup>) viscosities of latices made with cellulosic stabilizer in the emulsion recipe were much higher than those of latices made without the use of hydroxyethyl cellulose in the emulsion polymerization. The differences in particle size, if any, are swamped by the consequences of including or eliminating the cellulosic polymer in the emulsion recipe. Not surprisingly, the thixotropic latices arrived at their equilibrium low shear rate viscosities more slowly than their non-thixotropic counterparts. Again, thinning with water reduces the thixotropic latex viscosity at 0.6 s<sup>-1</sup>. Addition of Methocel J5MS or Natrosol 250 MR resulted in almost the same increase in low shear rate viscosity.

Low shear rate viscosities of the corresponding paints were, of course, much higher than those of the original latices. Addition of extra hydroxypropyl methyl cellulose contributed much less of a viscosity increase to the paint than to the latex. Addition of the associative thickener

actually reduced the viscosity of the paint in the low shear rate region for flow and levelling.

High shear viscosity values are compared in this work at 660 s<sup>-1</sup>. As mentioned above, viscosities at this shear rate are about double those exhibited by the same paints under brushing conditions (Table 6).

A useful parameter to condense information on the flow behavior of latices and paints is the shear-thinning factor (ST), defined operationally as:

$$ST = \frac{\text{equilibrium viscosity at } 0.6 \text{ s}^{-1}}{\text{equilibrium viscosity at } 660 \text{ s}^{-1}}$$

It will be recalled that the equilibrium viscosity at 0.6 s<sup>-1</sup> is that reached after recovery from shearing-out of thixotropic structures. The equilibrium viscosity at 660 s<sup>-1</sup>, by contrast, characterizes the sheared system. The best combination of good levelling and adequate hiding (i.e., brush drag or roller resistance) is achieved

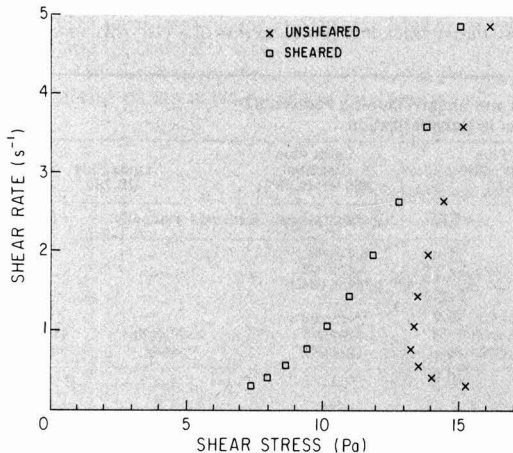


Figure 9—Shear rate-shear stress plot for thixotropic latex made with cellulosic in the polymerization recipe (reaction 5 of Table 4) plus 0.25% Methocel J5MS. Stress overshoot is seen in the unsheared material

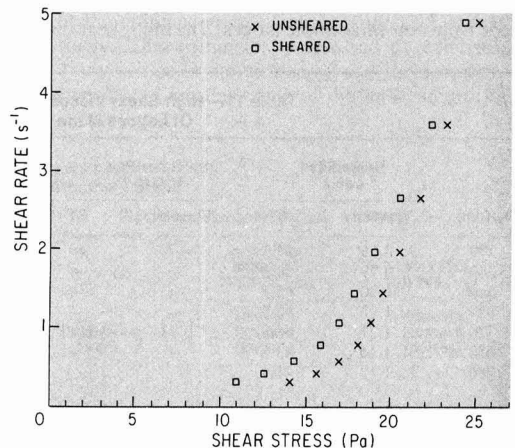


Figure 10—Shear rate-shear stress plot for thixotropic latex made with associative thickener in the polymerization recipe (reaction 8 of Table 4) plus 0.25% Methocel J5MS. There is no stress overshoot in the unsheared material



**Table 10—High Shear Viscosities (poises at 660 s<sup>-1</sup>) and Shear-Thinning Factors (ST) of Latices Made without Protective Colloid**

$\bar{D}_w$ (nm)	Unmodified Latex		Latex Plus H <sub>2</sub> O (6.7%)		Latex Plus Methocel J5MS (0.25%)		Latex Plus QR 708 (1%)	
	Viscosity	ST	Viscosity	ST	Viscosity	ST	Viscosity	ST
170...	0.40	5.9	—	—	1.51	97.0	—	—
205...	0.44	7.2	—	—	1.52	100.2	—	—
230...	0.51	7.8	—	—	1.56	99.1	—	—
260...	0.56	6.4	—	—	1.62	98.8	—	—
280...	0.42	7.6	0.21	5.6	1.40	91.1	3.4	5.5
340...	0.43	7.6	—	—	1.32	95.4	—	—
320...	0.50	6.3	0.22	5.4	1.49	93.5	—	—

with paints that have lower ST's. In other words, for a given Stormer viscosity, the lower ST implies a more Newtonian system and a higher application viscosity.

Latices made without cellulosic protective colloid in the polymerization exhibited low viscosities at 660 s<sup>-1</sup> shear rate and were relatively non-shear-thinning (Table 10). By contrast, thixotropic latices that were polymerized in the presence of hydroxyethyl cellulose had viscosities about 50-100% higher at given particle size (Table 11). Their shear-thinning behavior was also very much greater. That is to say, the effects of cellulosic additives in the polymerization are primarily seen at low shear rates. The thixotropic and non-thixotropic latices differed less at high shear rates. In both latex types, thinning with water caused a significant drop in viscosity across the whole shear rate range and a shift to more Newtonian rheology.

When extra cellulosic was added to the latices, the relative thickening effect was greater for latices made without hydroxyethyl cellulose. The associative thickener augmented the high shear rate viscosity of both latex types and made the material more Newtonian.

The semi-gloss paints were much more shear-thinning than the corresponding non-thixotropic latices. Table 12 records high shear viscosities and ST factors for paints made from non-thixotropic latices. The high shear viscos-

**Table 12—High Shear Viscosities (poises) and Shear-Thinning Factors ST Of Paints Made from Latices with No Protective Colloid In the Polymerization**

$\bar{D}_w$ (nm)	Unmodified Paint		Paint Plus H <sub>2</sub> O (5%)		Paint Plus Methocel J5MS (0.165%)		Paint Plus QR 708 (0.165%)	
	Viscosity	ST	Viscosity	ST	Viscosity	ST	Viscosity	ST
170 ..	2.0	48	—	—	2.6	51	—	—
205 ..	1.7	78	—	—	2.4	71	—	—
230 ..	1.7	70	—	—	2.3	72	—	—
260 ..	1.7	73	—	—	2.3	63	—	—
280 ..	1.7	65	—	—	2.3	60	1.7	52
340 ..	1.7	57	1.2	54	2.3	53	—	—
320 ..	1.7	63	1.1	59	2.2	56	—	—

ities of the paints were three to four fold those of the initial latices, but the shear-thinning factors were about 10 times as great.

A comparison of paints made from thixotropic latices and the original emulsions is made with the aid of Tables 11 and 13. The high shear viscosities of these paints are less than three times those of the progenitor emulsions and the shear-thinning character of the paints is actually less than that of the latices. This is opposite to the behavior of non-thixotropic latices.

Another interesting result from Tables 10-13 is that the non-thixotropic latices produced semi-gloss paints that had higher low shear viscosities and were more shear-thinning than paints made from thixotropic latices. This is despite the fact that the non-thixotropic latices themselves were less shear-thinning than their thixotropic counterparts. This points up the importance of knowledge of thickener-latex interactions as well as of latex behavior in designing paints with desirable rheological behavior.

Addition of extra cellulosic thickener to either the thixotropic or non-thixotropic type of paint has relatively little effect on the shear-thinning character. That is to say, the paint viscosity can be increased by this expedient without making the coating more shear-thinning. The associative thickener produces less of a viscosity increase

**Table 11—High Shear Viscosities (poises) and Shear-Thinning Factors ST Of Latices Made with Thickener in Polymerization**

$\bar{D}_w$ (nm)	Unmodified Latex		Latex Plus H <sub>2</sub> O (6.7%)		Latex Plus Methocel J5MS (0.25%)		Latex Plus Natrosol 250 MR (0.25%)		Latex Plus QR 780	
	Viscosity	ST	Viscosity	ST	Viscosity	ST	Viscosity	ST	Viscosity	ST
270 .....	0.76	37.1	—	—	1.66	73.9	—	—	—	—
340 .....	0.79	38.2	—	—	1.66	86.1	—	—	—	—
540 .....	1.20	65.7	—	—	1.90	91.5	—	—	—	—
650 .....	1.25	46.2	—	—	1.96	78.6	—	—	—	—
720 .....	1.21	46.6	0.73	22.7	2.08	77.3	1.94	76.8	2.31	13.5 <sup>a</sup>
650 .....	1.16	43.8	—	—	2.08	79.1	—	—	—	—
780 .....	1.12	41.1	—	—	1.92	76.9	—	—	—	—
330 <sup>c</sup> .....	1.15	18.3	0.51	10.9	2.04	128.5	—	—	1.41	9.0 <sup>b</sup>

(a) 1% QR 708.

(b) 0.25% QR 708.

(c) Latex made with QR 708 in the emulsion recipe.

**Table 13—High Shear Viscosities (poises) and Shear-Thinning Factors Of Paints Made from Latices with Protective Colloid in Polymerization**

PC	Latex $D_w$ (nm)	Unmodified Paint		Paint Plus H <sub>2</sub> O (5%)		Paint Plus Methocel J5MS (0.165%)		Paint Plus QR 708 (0.165%)		Paint Plus QR 708 (0.165%) J5MS (0.083%)	
		Viscosity	ST	Viscosity	ST	HSV	ST	HSV	ST	HSV	ST
Natrosol 250 MR	340	1.9	48	—	—	2.5	44	1.8	33	2.3	36
Natrosol 250 MR	540	2.0	44	—	—	2.7	41	—	—	—	—
Natrosol 250 MR	650	2.6	35	1.8	29	3.3	34	2.3	25	—	—
Natrosol 250 MR	720	2.2	39	—	—	2.9	36	2.0	29	—	—
Natrosol 250 MR	650	2.4	35	1.7	29	3.1	34	—	—	—	—
Natrosol 250 MR	780	2.1	40	—	—	2.7	38	2.2	27 <sup>a</sup>	—	—
QR 708	320	2.1	60	—	—	3.0	49	—	—	—	—
Commercial PVAC	—	2.2	32	—	—	2.9	32	1.9	27	2.4	29
Commercial acrylic	—	2.4	13	—	—	3.0	14	—	—	—	—

PC—Protective Colloid.  
(a) 0.33% QR 708.

than the same amount of cellulosic, but it also reduces the shear-thinning factor.

The effects of cellulosic and associative thickeners are summarized in the following tables. *Table 14* lists the effects of Methocel J5MS and QR 708 on latices made without thickener in the polymerization. There is no noticeable interaction between the thickener effect and latex particle size. Cellulosic thickeners have a large effect at low shear rates and much less influence at high shear rates.

*Table 15* is a similar summary for latices made with cellulosic in the polymerization recipe. Here the relative effects of post-added cellulosic are much less than with non-thixotropic latices. In both latex types, the additional cellulosic has a greater effect on low shear rate than high shear rate viscosity and this difference is particularly pronounced for latices that are made initially without cellulosic derivatives. The polyurethane block copolymer associative thickener produces a relatively larger increase in the high shear rate than the low shear rate behavior and this is true for both latex types.

*Table 16* summarizes the results of adding rheology modifiers to paints made from non-thixotropic latices.

The relative increase in viscosity is much less than for the addition of cellulosic to the corresponding latex and the discrepancy between relative effects at  $0.6 \text{ s}^{-1}$  and  $660 \text{ s}^{-1}$  is also much less. (Recall that these paints already contain some Methocel J5MS from the mill base.)

The corresponding results for paints made from thixotropic latices are summarized in *Table 17*. Again, comparison with *Table 15* shows that the relative effects of the cellulosic are less in the paints than in the progenitor latices. For paints made from thixotropic latices, the relative increase in viscosity from addition of Methocel J5MS is slightly greater at higher shear rates than at  $0.6 \text{ s}^{-1}$  shear rate. In these paints, also, the addition of associative thickener at the low level used (0.165%) results in a decrease in viscosity at all shear rates. This is probably because of the dilution effect from the solvent carried along with the rheology modifier.

The thixotropic character of the latices made with cellulosic in the polymerization recipe are compared with those of the corresponding paints in *Tables 18* and *19*, respectively. This parameter is assessed by comparing  $0.6 \text{ s}^{-1}$  viscosities in the unsheared and sheared states. The values listed are the ratios of the unsheared to the

**Table 14—Viscosity Increment by Thickener Post-addition To Latices Made without Use of Protective Colloid**

Low Shear Viscosity ( $0.6 \text{ sec}^{-1}$ , poises)				High Shear Viscosity ( $662 \text{ sec}^{-1}$ , poises)			
Latex $D_w$ (nm)	Unmodified Latex	Latex Plus Methocel J5MS (0.25%)	Latex Plus QR 708 (1%)	Unmodified Latex	Latex Plus Methocel J5MS (0.25%)	Latex Plus QR 708 (1%)	
		Viscosity Increase (poises)	Viscosity Increase (poises)		Viscosity Increase (poises)	Viscosity Increase (poises)	
170	2.4	144	—	0.4	1.1	—	
205	3.2	149	—	0.4	1.1	—	
230	3.9	158	—	0.5	1.1	—	
261	3.6	158	—	0.6	1.1	—	
280	3.2	124	15	0.4	1.0	3.0	
340	3.2	123	—	0.4	0.9	—	
320	3.2	135	—	0.5	1.0	—	

**Table 15—Viscosity Increment by Thickener Post-addition To Latices Made with Use of Protective Colloid**

PC	Latex $\bar{D}_w$ (nm)	Low Shear (0.6 sec <sup>-1</sup> , poises)			High Shear (662 sec <sup>-1</sup> , poises)		
		Unmodified Latex	Latex Plus Methocel J5MS (0.25%)	Latex Plus QR 708	Unmodified Latex	Latex Plus J5MS (0.25%)	Latex Plus QR 708
			Viscosity Increase (poises)	Viscosity Increase (poises)		Viscosity Increase (poises)	Viscosity Increase (poises)
Natrosol 250 MR ...	270	28	94	—	0.8	0.9	—
Natrosol 250 MR ...	340	28	114	—	0.8	0.9	—
Natrosol 250 MR ...	540	79	95	—	1.2	0.7	—
Natrosol 250 MR ...	650	58	96	—	1.3	0.7	—
Natrosol 250 MR ...	720	57	104	-25.3 <sup>a</sup>	1.2	0.8	1.1
		[20.5(+ H <sub>2</sub> O 6.7%)] <sup>c</sup>	123	10.7 <sup>b</sup>	[0.73(+ H <sub>2</sub> O 6.7%)]	1.3	1.6
							[(+ QR 708 1%)]
Natrosol 250 MR ...	650	51	113	—	1.2	0.8	—
Natrosol 250 MR ...	780	47	101	—	1.1	0.8	—
QR 708.....	320	17	245	-4	1.2	0.9	0.3
		[5.5(+ H <sub>2</sub> O 6.7%)]	256	7.5	[0.51(+ H <sub>2</sub> O 6.7%)]	1.8	0.9
				[(+ QR 708 0.25%)]			[(+ QR 708 0.25%)]

PC— Protective Colloid.  
 (a) Negative number is due to dilution effect.  
 (b) 1% QR 708.  
 (c) Latex viscosity 25 poises after addition of 6.7% H<sub>2</sub>O.

sheared-out viscosities. In all cases it can be seen that the semi-gloss paint is less thixotropic than the parent latex.

**Latex Particle Size Effects**

The effects of latex particle size are considered here in terms of the latex and paint derived therefrom. The rheological properties of both systems have been seen to differ, depending on whether the latex was synthesized in the presence of a cellulosic protective colloid. Particle size effects are therefore also summarized separately for the two latex types. The latices used were not monodisperse and we have compared different polymers on the basis of  $\bar{D}_w$ , since this average diameter is one of the more reliable averages from hydrodynamic chromatography analyses.

The viscosity behavior is examined at 0.6 s<sup>-1</sup> shear rate (levelling region) and 662 s<sup>-1</sup> shear rate (to parallel application by brush or roller).

Figure 11 shows the 0.6 s<sup>-1</sup> viscosities of latices made without the use of a cellulosic polymer in the emulsion recipe. Data points are given at 0.6 s<sup>-1</sup> 20 seconds after high speed shearing and at the final "equilibrium" at 0.6 s<sup>-1</sup>. These systems are not thixotropic and 20 second and equilibrium viscosities are not significantly different. Trends indicate a maximum viscosity at about 250 nm  $\bar{D}_w$ .

Figure 12 summarizes the corresponding data points for the paints made from the latices of Figure 11. Paint viscosity also peaks with latex  $\bar{D}_w$  diameters between 200-250 nm. The basic paint is not thixotropic. Addition of extra cellulosic, above that in the mill base, does induce some slight thixotropy.

**Table 16—Viscosity Increment by Thickener Post-addition To Paint Made from Latex Polymerized without Use of Protective Colloid**

Latex $\bar{D}_w$ (nm)	Unmodified Paint	Low Shear (0.6 sec <sup>-1</sup> , poises)		Unmodified Paint	High Shear (662 sec <sup>-1</sup> , poises)	
		Paint Plus Methocel J5MS (0.165%)	Paint Plus QR 708 (0.165%)		Paint Plus Methocel J5MS (0.165%)	Paint Plus QR 708 (0.165%)
		Viscosity Increase (poises)	Viscosity Increase (poises)		Viscosity Increase (poises)	Viscosity Increase (poises)
170 .....	95	41	—	2.0	1.7	—
205 .....	135	30	—	1.7	0.6	—
230 .....	119	46	—	1.7	0.6	—
260 .....	120	23	—	1.7	0.6	—
280 .....	111	26	-23 <sup>a</sup>	1.7	0.6	0
340 .....	94	25	—	1.7	0.6	—
320 .....	106	17	—	1.7	0.6	—

(a) Dilution effect.



**Table 17—Viscosity Increment by Thickener Post-addition To Paint from Latices Polymerized with Use of Protective Colloid**

PC	Latex $D_w$ (nm)	Low Shear (0.6 sec <sup>-1</sup> , poises)			High Shear (662 sec <sup>-1</sup> , poises)		
		Unmodified Paint	Paint Plus Methocel J5MS (0.165%)	Paint Plus QR 708 (0.165%)	Unmodified Paint	Paint Plus Methocel J5MS (0.165%)	Paint Plus QR 708 (0.165%)
			Viscosity Increase (poises)	Viscosity Increase (poises)		Viscosity Increase (poises)	Viscosity Increase (poises)
Natrosol 250 MR	340	89	21	-29	1.9	0.6	0
Natrosol 250 MR	540	91	21	—	2.0	0.7	—
Natrosol 250 MR	650	92	19	-34	2.6	0.7	-0.3
		[52.9(+H <sub>2</sub> O 5%)]	58	6	[1.8(+H <sub>2</sub> O 5%)]	1.5	0.5
Natrosol 250 MR	720	87	182	-29	2.2	0.7	-0.3
Natrosol 250 MR	650	85	21	—	2.4	0.7	—
Natrosol 250 MR	780	84	20	-25	2.1	0.6	0.1
				[0.33% QR 708]			[0.33% QR 708]
QR 708	320	123	26	—	2.1	1.0	—
Commercial PVAC	—	71	21	-19	2.2	0.7	-0.3
Commercial acrylic	—	30	13	—	2.4	0.6	—

PC—Protective colloid.  
(a) Negative numbers are due to the dilution effect.

**Table 18—Thixotropy Indices of Latices Made with Use Of Protective Colloid Viscosities at 0.6 s<sup>-1</sup> Shear Rate**

PC	Latex $D_w$ (nm)	Unmodified Latex	Latex Plus H <sub>2</sub> O (6.7%)	Latex Plus Methocel J5MS (0.25%)	Latex Plus Natrosol 250 MR (0.25%)	Latex Plus QR 708
Natrosol 250 MR	270	2.7	—	1.7	—	—
Natrosol 250 MR	340	2.5	—	1.7	—	—
Natrosol 250 MR	540	2.1	—	1.5	—	—
Natrosol 250 MR	650	2.2	—	1.5	1.6	2.1 (1%)
Natrosol 250 MR	720	2.4	2.5	1.6	—	—
Natrosol 250 MR	650	2.4	—	1.6	—	—
Natrosol 250 MR	780	2.4	—	1.6	—	—
QR 708	320	1.5	1.4	1.2	—	1.2 (0.25%)

PC—Protective colloid.

**Table 19—Thixotropy Indices of Paints Made from Latices Polymerized With Use of Protective Colloid Viscosities at 0.6 s<sup>-1</sup> Shear Rate**

PC	Latex $D_w$ (nm)	Paint	Paint Plus H <sub>2</sub> O	Paint Plus Extra Methocel J5MS (0.165%)	Paint Plus QR 708 (0.165%)	Paint Plus QR 708 (0.165%) J5MS (0.83%)
Natrosol 250 MR	340	1.3	—	1.2	1.2	1.2
Natrosol 250 MR	540	1.3	—	1.2	—	—
Natrosol 250 MR	650	1.2	1.3	1.1	1.2	—
Natrosol 250 MR	720	1.3	—	1.2	1.2	—
Natrosol 250 MR	650	1.3	1.3	1.2	—	—
Natrosol 250 MR	780	1.3	—	1.2	1.2	—
					(0.33%)	
QR 708	320	1.0	—	1.0	—	—

PC—Protective colloid.

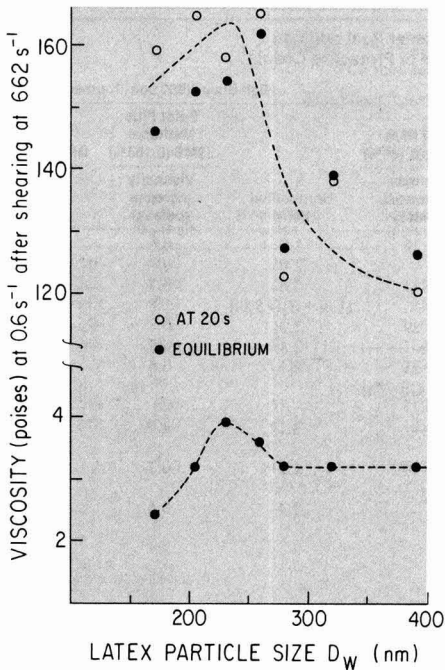


Figure 11—Viscosities in the levelling region ( $0.6\text{ s}^{-1}$  shear rate); lower curve—latices made without protective colloid in the emulsion recipe; upper curve—corresponding latices plus 0.25% Methocel J5MS

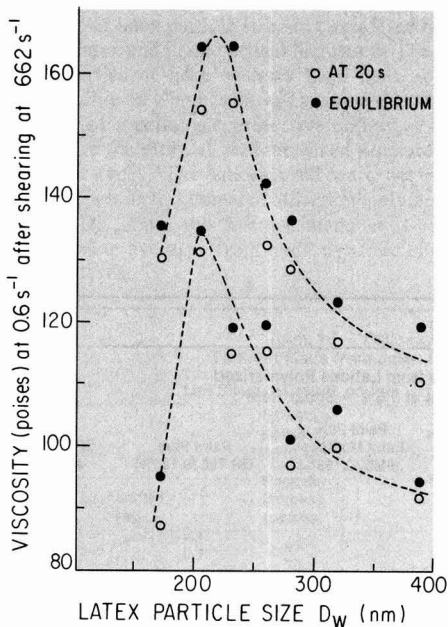


Figure 12—Viscosities in the levelling shear rate region ( $0.6\text{ s}^{-1}$ ). Lower curve—paints made according to Table 1 formulation from latices produced without protective colloid in the emulsion recipe; upper curve—paints to which were added an extra 0.165% Methocel J5MS

Plots of low shear rate viscosity are given in Figure 13, for latices polymerized in the presence of Natrosol 250 MR. Here, thixotropy is exhibited by both the primary latex and the version that had been thickened with Methocel J5MS. A strong maximum in viscosity is seen in this case with  $D_W$  particle diameters about 550 nm. The corresponding paints (Figure 14) show very little effect of particle size on levelling behavior, however. The degree of thixotropy is also much less than in the primary latices.

We look now in Figure 15 at the high shear rate ( $662\text{ s}^{-1}$ ) viscosities of latices made without cellulosic in the emulsion recipe. The high shear rate viscosity also appears to peak in the 250-300 nm  $D_W$  region (cf. Figure 11).

The high shear rate viscosities of the corresponding non-thixotropic paints are depicted in Figure 16. Here one sees essentially no effect of latex particle size, except for the smallest diameter sample, which probably has  $D_W$  lower than commercial poly (vinyl acetates) for this application.

Figures 17 and 18 show the respective high shear rate viscosities for thixotropic latices (cellulosic in the polymerization recipe) and the corresponding paints. The latices appear to have a peak viscosity with  $D_W$  near 600 nm but the paint viscosity is fairly well independent

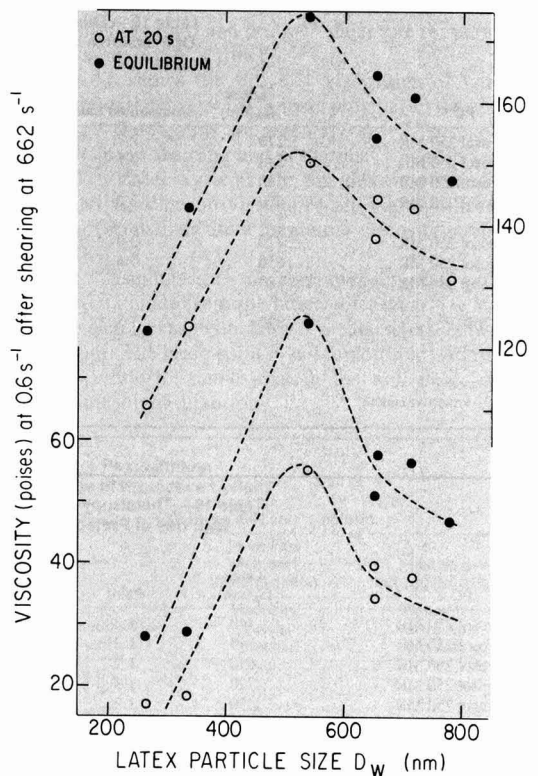


Figure 13—Viscosities in the levelling shear rate region ( $0.6\text{ s}^{-1}$ ). Lower pair of curves—latices made with cellulosic in the emulsion recipe; upper pair of curves—corresponding latices plus 0.25% Methocel J5MS

of latex particle size in the 700-800 nm range. It is important, also, to note that the formulations of *Figure 18* are generally close to the systems that are used for most retail paints.

## DISCUSSION

This work was confined to the use of a particular hydroxyethyl cellulose (Natrosol 250 MR) in the emulsion polymerization recipes and to a single hydroxypropyl methyl cellulose (Methocel J5MS) as a thickener in paints and latices. The conclusions from this research are, however, unlikely to be restricted to these materials alone. The cellulosic used in the latex polymerization is rapidly degraded in the process and its initial molecular weight is unlikely to be of great importance in this case.<sup>15</sup> The molecular weight of Methocel brand cellulose ethers is reported to have no effect on the low shear viscosity of vinyl acrylic based paints.<sup>17</sup> Variations of the thickener from the particular cellulosic used here will, similarly, affect the high shear rate viscosity only to a maximum of about 20%.<sup>13</sup> (Such comparisons are normally made at equal Stormer viscosities. The greater apparent efficiency of low molecular weight cellulose at high shear rates may be simply due to the higher concentrations needed to achieve a given Krebs units level. This may be important in particular formulations, but it is a weak effect compared to the variables which are the focus of this report.)

Within the constraints of the systems studied here, it can be seen that a major factor is the presence or absence of cellulosic thickener in the latex polymerization.

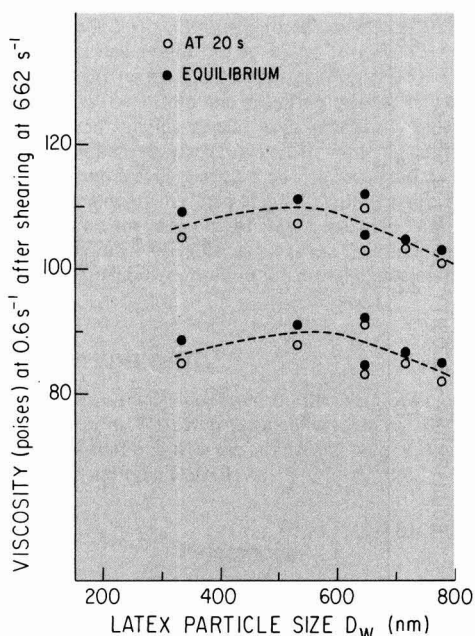
An important feature of the results reported here is that the latices produced were thixotropic only if the polymerization had been performed in the presence of dissolved cellulosic polymer. Post-addition of a cellulosic polymer thickened the latex but did not make it thixotropic. In parallel, paints made according to the formulation of this work were thixotropic only if the precursor latex was also thixotropic. These observations provide insights into the thickening action of cellulose.

Thickening by water-soluble polymers such as cellulose derivatives has been shown not to be a simple process of increasing the viscosity of the aqueous medium.<sup>18</sup> The ability of the thickener to flocculate or deflocculate the latex appears to be an important part of the thickening mechanism.

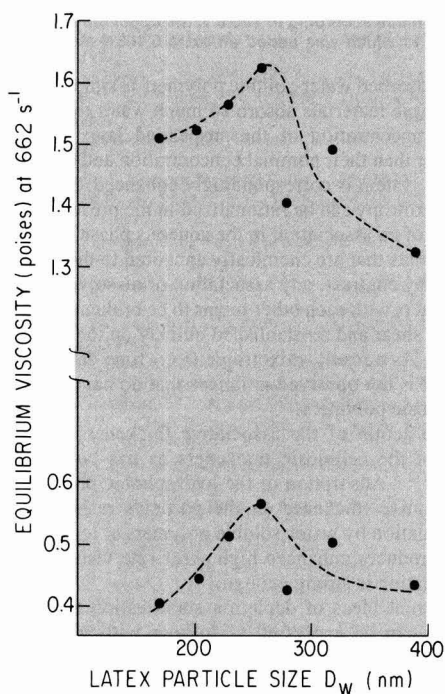
Such flocculation has been explained in terms of "bridging"—i.e., adsorption of the soluble polymer onto several latex particle surfaces.<sup>19,20</sup> More recently, it has been demonstrated to occur in the absence of adsorption.<sup>21</sup> Nonadsorbed soluble polymers can, in fact, cause flocculation by a mechanism in which particles are forced further together when the distance between them is too small to accommodate the hydrated dissolved polymer coils. The details of this "depletion flocculation" mechanism are described qualitatively later in this section.

Current thinking appears to favor the depletion flocculation mechanism over the bridging hypothesis. Our results reinforce this conclusion since there is no noticeable effect of latex particle size on thickener effectiveness.

Another way of rationalizing the thickening action of



**Figure 14**—Viscosities in the levelling shear rate region ( $0.6 \text{ s}^{-1}$ ). Lower curve—paints (*Table 1*) made from the latices of *Figure 13*; upper curve—paints to which were added an extra 0.165% Methocel J5MS



**Figure 15**—Sheared-out viscosities ( $662 \text{ s}^{-1}$ ) for latices produced without cellulosic in the emulsion recipe (lower curve). The upper curve is for the corresponding latices to which were added 0.25% (w/w) Methocel J5MS



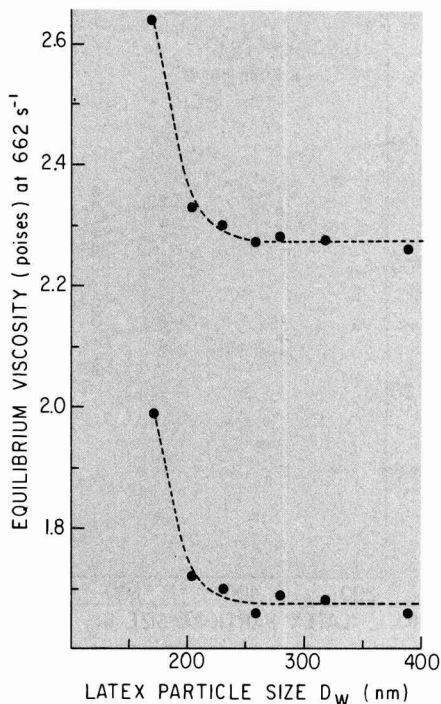


Figure 16—High shear rate ( $662 \text{ s}^{-1}$ ) viscosities of paints made from the latices of Figure 15. The lower curve is for paints made according to Table 1; the upper curve is for these paints to which was added an extra 0.165% Methocel J5MS

non-adsorbed water-soluble polymers is simply to assume that these materials absorb so much water that the effective concentration of the suspended latex particles is greater than their nominal concentration and the viscosity of the system is correspondingly enhanced.

Thixotropy can be rationalized in the present system in terms of an association in the aqueous phase of cellulosic fragments that are chemically anchored to the latex particles. By contrast, any association of dissolved cellulosic polymers with each other seems to be broken very quickly under shear and reestablished quickly on the cessation of shear. As a result, thixotropic (i.e., time dependent) behavior is not observed in latices that do not carry grafted cellulosic polymers.

The action of the associative thickener differs from that of the cellulosic thickeners as has been noted by others.<sup>2,3</sup> Adsorption of the hydrophobic portions of the associative thickener on the particles resists depletion flocculation by water-soluble polymers at low shear rates and produces enhanced high shear rate viscosities if the association is strong enough.

Current ideas of depletion stabilization and flocculation<sup>22</sup> can be expressed as follows with respect to the systems of this report. When two latex particles happen to approach each other to within a distance that is smaller than the hydrodynamic diameter of the hydrated cellulosic molecule, the latter will be expelled from the region between the particles. This polymer-free volume is one in which the local osmotic pressure is less than in the bulk

medium. (In thermodynamic terms, the relatively pure solvent in the interparticle region has a higher chemical potential than solvent containing dissolved celluloses.) The net tendency is for solvent to flow out of the interparticle region and for the latex particles to be pushed together.<sup>23</sup> The reversible flocculation of polymer latices by hydroxyethyl cellulose has been shown to proceed by the mechanism described.<sup>21</sup>

The flocs that are formed in this manner are loose, since the particles will still stand off from each other because of electrostatic repulsion from adsorbed and grafted anionic surfactants on their surfaces.

This flocculation process will have less importance when the latex particles are sterically stabilized by grafted cellulosic polymer fragments that are produced by emulsion polymerization in the presence of the protective colloid. In that case, the hydroxyethyl cellulose is chemically anchored to the poly(vinyl acetate) particle surface and the effective density of the water-soluble polymer in interparticle regions will be little affected by the concentration of added, extra cellulosic derivative.

Restabilization of loosely flocculated latices can occur in more concentrated solutions of water-soluble polymers.<sup>24</sup> With increasing concentration of polymer, the hydrodynamic size of the hydrated coils decreases<sup>25,26</sup> and the water-soluble molecules are more readily able to insert themselves into the interparticle regions. As a con-

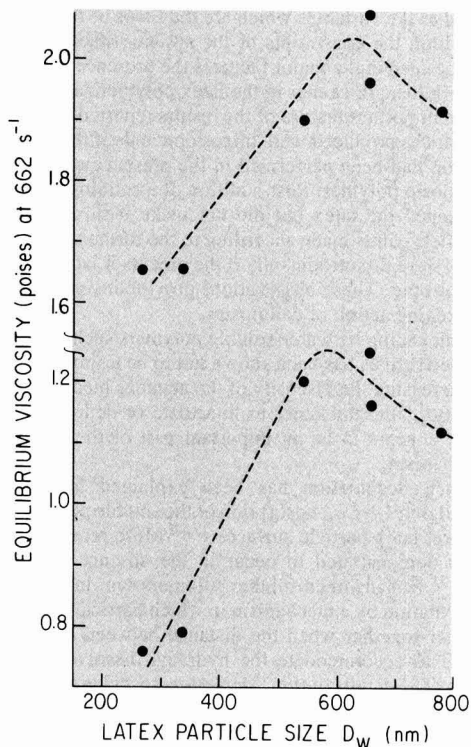


Figure 17—High shear rate viscosities ( $662 \text{ s}^{-1}$ ) of latices produced with cellulose in the emulsion recipe (lower curve) and the same latices to which was added an extra 0.25% (w/w) Methocel J5MS

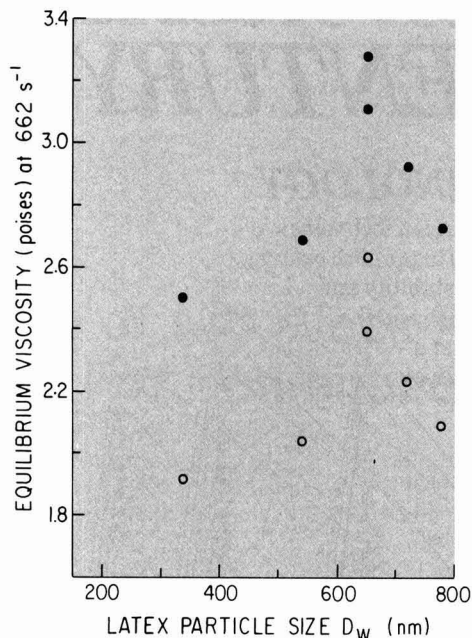


Figure 18—High shear rate viscosities (662  $s^{-1}$ ) of paints made from the latices of Figure 17 (open curves) and the same paints with an extra 0.165% Methocel J5MS

sequence, the flocs are broken and the latex is restabilized.

Addition of a water-soluble cellulosic thickener to a thixotropic latex increases the viscosity of the system, with some reduction in thixotropic character. The same general effects are observed when these thickeners are added to thixotropic paints. The reductions in thixotropy are weak effects, however (cf. Figure 7). These observations can be explained as follows.

The viscosity increase that results from addition of water-soluble cellulose is thought to result partly from hydrogen bonding with water molecules, with some contribution from entanglement of cellulosic polymer coils. That is, this viscosity increase reflects the increased viscosity of the aqueous medium. In addition, of course, the effective concentration and the viscosity of the latex suspension is increased by the crowding effect mentioned above.

The decrease in thixotropy on addition of water-soluble cellulosic thickeners may be due to the reduction in effective particle-particle distances. This facilitates the recovery of interparticle associations and decreases the time dependence of the recovery of thixotropic structures.

In support of this hypothesis, we note that the urethane block copolymer associative thickener decreases the thixotropic character of the latex less than an equivalent weight concentration of cellulosic polymer. This is to be expected, since the associative thickener is believed to function by bridging particles, rather than by forcing them together.

The particle size of a latex influences its flow behavior. We focus in this discussion on thixotropic latices, since these correspond more closely to commercial products for

the coatings industry. Strong particle size effects are seen in the shear rate region for levelling (Figure 13), with weaker effects on brushing behavior (Figure 15). These do not carry over into the resulting paints, however. Levelling and sagging appear to be practically independent of latex particle size (Figure 14), while the brushing viscosity is independent of particle size for all latices with mean diameters greater than about 250 nm (Figure 17).

Generally, the effects of latex particle size are swamped by other variables such as the type and amount of polymeric stabilizer used in the actual emulsion polymerization.

## ACKNOWLEDGMENT

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# "CREATIVE COLORSHIP FOR OVER A CENTURY"



## COLORSHIP is TECHNOLOGY

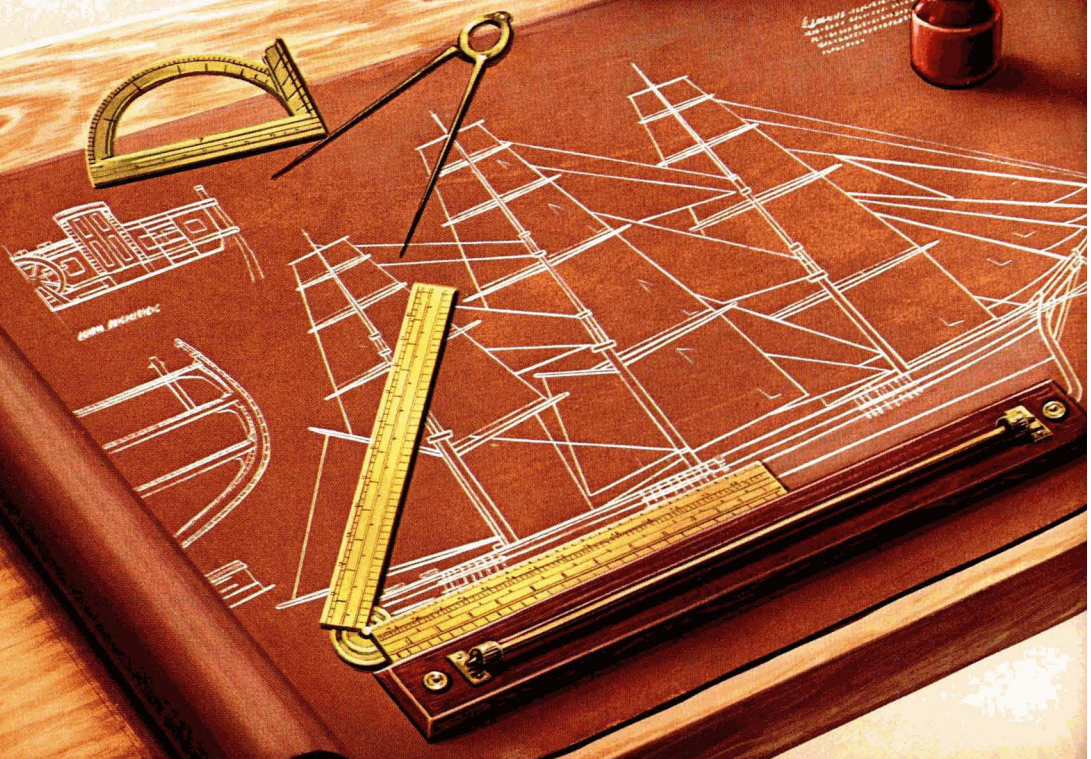
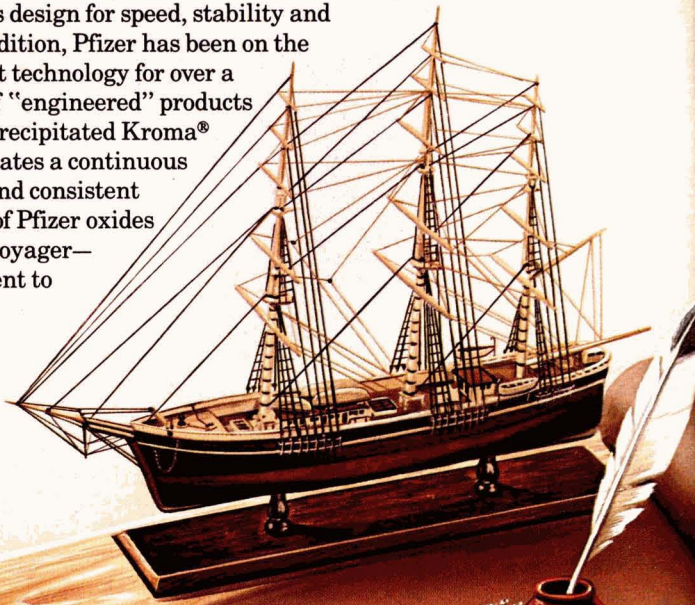
The Yankee Clipper represented American technological leadership as it roamed the world's oceans in the 19th century. Nothing could match its design for speed, stability and stature. In the same tradition, Pfizer has been on the leading edge of Pigment technology for over a century. Introduction of "engineered" products such as Pferrisperse™ Iron Oxide Slurry, Precipitated Kroma® Reds, and Low Viscosity Yellows demonstrates a continuous commitment to technology. High purity and consistent quality have resulted in the specification of Pfizer oxides in the rocket motors of America's newest voyager—The Space Shuttle. Colorship is commitment to technology. At Pfizer, there's no other way.



### MINERALS, PIGMENTS & METALS DIVISION

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# Use of A-B Block Polymers as Dispersants For Non-aqueous Coating Systems

Henry L. Jakubauskas  
E.I. du Pont de Nemours & Co., Inc.\*

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Theoretical considerations involved in the dispersions of pigments are reviewed, including a new class of pigment dispersants for non-aqueous solvent systems that has been developed by Du Pont. These dispersants are polymer molecules containing "polar clusters" attached to one end of non-polar polymeric chain. The stability of dispersed pigment particles is achieved by adsorption of the "polar cluster" on the pigment surface with the non-polar portion of the molecule providing a barrier for pigment-to-pigment interactions. The mechanism of stabilization of particles is steric (entropic).

Pigment deflocculation is shown to be a function of dispersant structure, molecular weight polar and non-polar segments, pigment polarity, and solvent polarity used.

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## INTRODUCTION

Most surface coatings contain solid particles such as pigments and fillers. The dispersion of these particles in organic media is of considerable importance to the overall properties of surface coatings. Such coating properties as opacity, color intensity, gloss, color reproducibility, transparency of metallic colors, and mechanical properties such as tensile strength, hardness, flexibility, etc., depend on the degree of agglomeration and flocculation of particles. The same can be said about application properties such as flow, leveling, and sagging. Therefore, the selection of dispersants used to maintain the particles in a deflocculated state is of the utmost importance for controlling coating properties.

Surface coating formulations can be considered as a composite of three basic systems:

(1) Pigment system consisting of high concentration of pigment dispersed in solvent, surfactant/dispersant, and polymer commonly referred to as "mill-base" or color concentrate;

(2) Film-forming system to obtain desirable physical properties; and,

(3) Solvent system to obtain desired application latitude.

The maintenance of stable pigment dispersion in all of these three systems is a must for achieving the desired coating properties. The use of dispersing agents (dispersants, surfactants) in aqueous coating systems for preparation of mill-bases has been well documented. However, for non-aqueous systems, dispersant technologies on sound scientific principles are presently emerging. In general, for dispersing pigments in non-aqueous media, the main film-forming vehicles are used by coating manufacturers as pigment dispersants. This approach may or may not achieve stable pigment dispersions. As the coating technology shifts from low solids, high molecular weight polymers to high solids and low molecular weight polymers, the effectiveness of these polymers as pigment dispersants in most cases is reduced. Furthermore, a greater burden is placed on pigment dispersions to provide higher solids and higher pigment content dispersions for these high solids systems in an environment conducive to flocculation. Commercial dispersants have been developed by chemical companies such as ICI Americas Inc. (SOLSPERSE®), Byk-Chemie (DISPERBYK®), and others which are aimed at non-aqueous systems.

The purpose of this paper is to present additional evidence for the use of A-B block type polymers as effective pigment dispersants for non-aqueous systems. The effectiveness of these dispersants depends on the size and chemical nature of the adsorbing group and specific interactions of solvent-dispersant, and chemical nature of pig-

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Presented at the 63rd Annual Meeting of the Federation of Societies for Coatings Technology, in St. Louis, MO, on October 9, 1985.

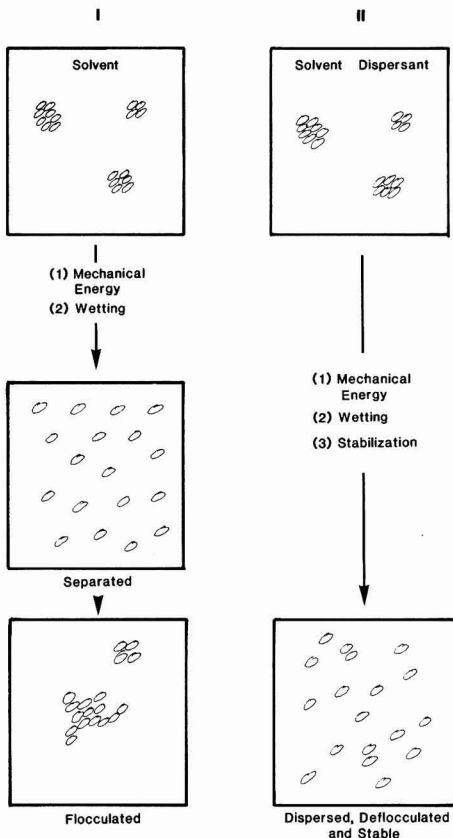
\*Marshall R&D Laboratory, 3500 Grays Ferry Ave., Philadelphia, PA 19146.

ment surfaces. To achieve stable dispersions for multi-pigment systems, more than a single dispersant is needed.

**DISPERSION THEORY**

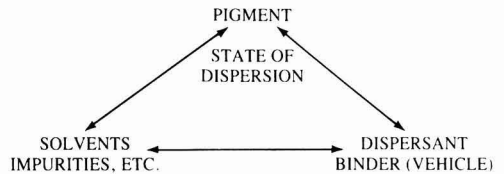
In general, all particles in a void attract each other as a result of the London van der Waals forces as advanced by Hamaker.<sup>1</sup> In addition as pointed out by the same author, when particles are in finite medium, they not only attract one another, but they also attract the molecules of the medium (solvent, gas, electrolytes, polymers). Therefore, the pigment stability depends on the ability of the electrolyte or polymer to concentrate at the solid-liquid interfaces and form a solvated layer of sufficient thickness to overcome the attractive forces between the solid particles.

Most of the pigments supplied are composed of aggregates and agglomerates of small pigment particles (1-10,000nm). The preparation of mill-bases requires mechanical energy (grinding process) to break up the pigment aggregates and agglomerates and to expose the maximum surface area for wetting by solvents and dispersants. To achieve a complete dispersion of pigments, the following factors must be present:



Case II has all the factors needed to achieve stable pigment dispersions. Stable dispersions in non-aqueous

systems are usually produced by the use of dispersants in addition to major film forming vehicles, and only in rare cases without any additives. For some cases, the vehicle itself can act as dispersing agent for pigments. It is desirable to have mill-bases with high pigment concentrations (5-80% by weight) for cost and formulation reasons. The preparation of the final coating is accomplished by a let-down of these mill-bases with vehicles (same or different) and solvents to yield the desired pigment/binder compositions. The state of the dispersion (degree of flocculation) at any of these steps in the preparation of coating compositions plays an important part in the overall performance of the coatings. The state of dispersion can be viewed as a composite of pigment-dispersant/binder, pigment-solvent, and solvent-binder interactions as illustrated.

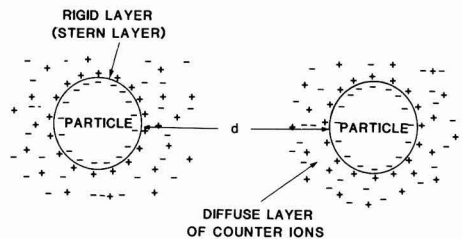


The importance of these interactions will be illustrated with specific A-B block polymers. As already emphasized, for preparation of reproducible and stable dispersions, one needs the wetting, size reduction, and stabilization factors. Wetting and size reduction factors are adequately discussed in dispersion literature.<sup>2</sup> Stabilization, which is defined as the process by which the particles can be prevented from flocculation and agglomeration, is of utmost importance in producing high concentration stable pigment dispersions.

There are two basic mechanisms ("charge double layer" and "steric") and a third proposed ("depletion") for stabilizing pigment particles in a solvent, polymer, electrolyte media.

**Charge Double Layer**

This mechanism is based on establishing a charge double layer at particle liquid interfaces, known as the Deryaguin, Landau,<sup>3</sup> Verwey, and Overbeek<sup>4</sup> (DLVO) theory. A charged species is preferentially adsorbed on the surface producing a charge layer with the immediate surrounding liquid, forming diffuse parts of a double layer. The mechanism can be illustrated by the following:

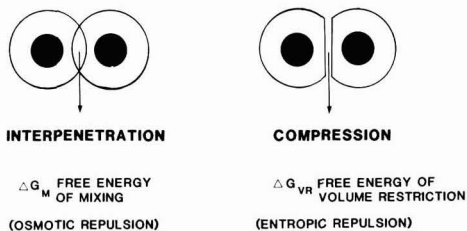


When two particles approach the diffuse parts of the double layer they begin to interpenetrate, giving rise to strong coulombic (electrostatic) repulsive forces. The re-

pulsive forces increase in magnitude as the distance between the particles decreases. However, if the particles are forced to approach each other closer, this repulsive barrier can be surmounted with London van der Waals attractive forces predominating, resulting in coagulation (irreversible flocculation) of particles. The magnitude of this barrier is strongly a function of ion concentration of media and, in turn, the dielectric constant of the media. Dispersions only using charge double layers as stabilizing mechanisms suffer from stability problems due to pH, ion concentration, multivalent ion contamination, and shear variables. This mechanism explains stabilization of pigments in aqueous media and, in many cases, is the predominant mechanism where electrolytes or polyelectrolytes are used as dispersants. However, for non-ionic dispersants and dispersants in organic solvents (low-dielectric constant), this mechanism may contribute to pigment stability as recognized by Fowkes,<sup>5</sup> but it is not the major mechanism.

**Steric Stabilization Mechanism**

The mechanism is based on adsorption of polymers on the pigment surfaces to reduce pigment-pigment attractive forces. This mechanism accounts for the reduction of attractive forces between particles in aqueous media with non-ionic type dispersants and in organic media for all dispersants used. The mechanism was advanced by Clayfield and Lumb<sup>6</sup> and later by Vincent<sup>7</sup> and Napper.<sup>8</sup> The adsorbed matter can be gas, solvent, or polymeric. However, the adsorbed barriers to prevent particle attractions should be in the order of 100 Å or higher<sup>9,10</sup> which then excludes gases and solvents. Stable dispersions (no appreciable increase in size of particle clusters, which cannot be broken down by gentle mixing) can be achieved with adsorbed or "anchored" (signifies strong attachment of adsorbing groups such as by hydrogen bonding or chemisorption as advanced by Vincent<sup>7</sup>) polymers of sufficient molecular weight. The major contents of steric stabilization can be illustrated as follows:



The steric stabilization can be described in terms of  $\Delta G_r =$  free repulsive energy.

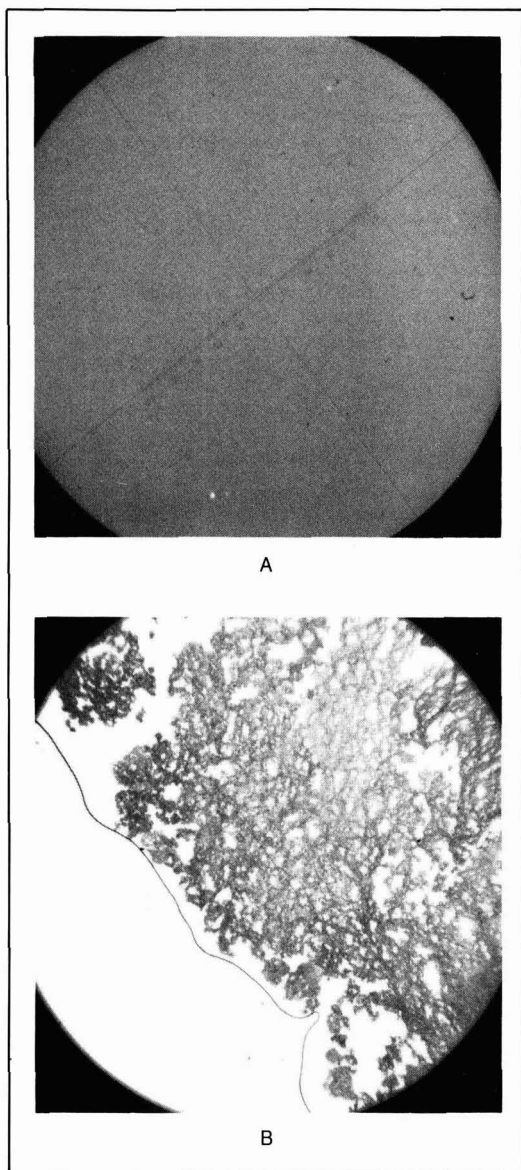
$$\Delta G_r = \Delta H_r - \Delta S_r$$

A positive value for  $\Delta G_r$  is necessary to achieve stability, and it can arise for  $\Delta H_r$  (enthalpic) and  $\Delta S_r$  (entropic) components.

Napper<sup>11,12</sup> has classified these two contributions in the manner presented in Table 1.

**Table 1—Components of Steric Stabilization**

Enthalpic	Entropic	Combined
$\Delta H_r, \Delta S_r$ positive	$\Delta H_r, \Delta S_r$ negative	$\Delta H_r$ , positive $\Delta S_r$ , negative
$H_r > \Delta TS_r$	Dispersions flocculate on cooling	↑
Dispersions flocculate on heating	Predominates in non-aqueous systems	Dispersions stable over wide temperature range



**Figure 1—Light microscopy. A—deflocculated dispersion (425X); B—flocculated dispersion (425X)**



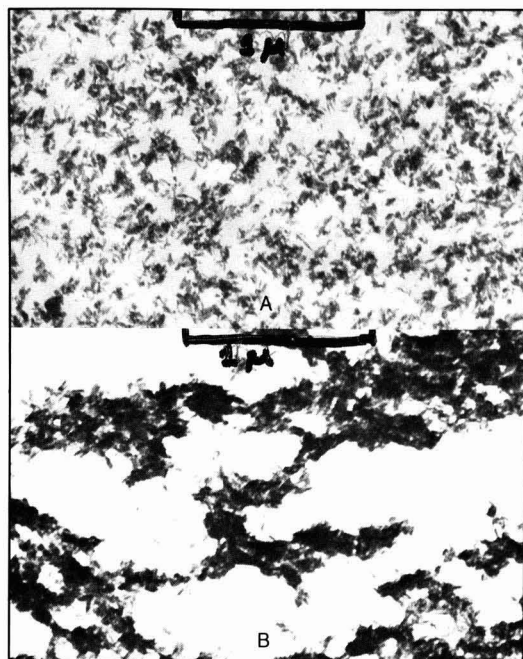


Figure 2—Electron microscopy (19000X). A—deflocculated iron oxide; B—flocculated iron oxide

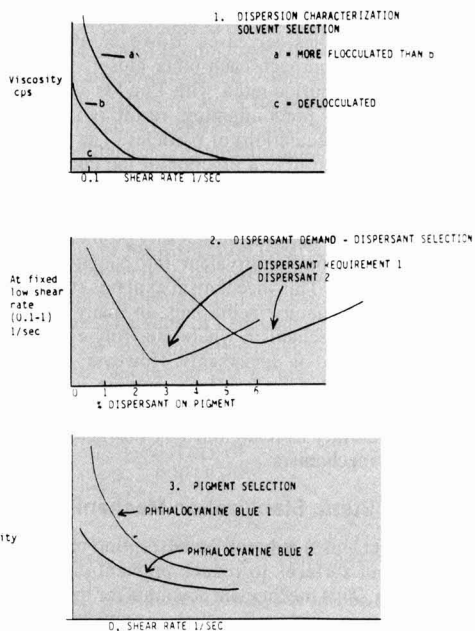


Figure 4—Rheological characterization of dispersion components

$$\eta = \frac{\text{SHEAR STRESS}}{\text{SHEAR RATE}}$$

$$\tau = \frac{\text{FORCE}}{\text{AREA}} = \text{DYNES/CM}^2$$

$$D = \frac{V}{t} = \frac{\text{VELOCITY}}{\text{THICKNESS}} = \text{SEC}^{-1}$$

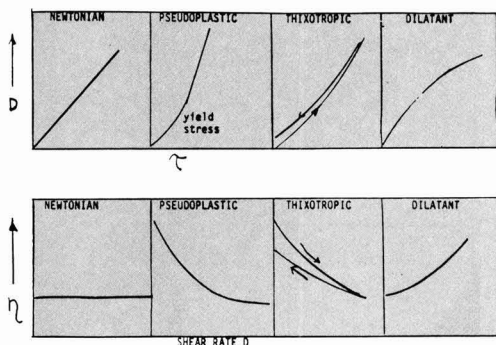


Figure 3—Rheological assessment of 15-20% pigment dispersions. Dispersions close to Newtonian were deflocculated/stable. This method is used extensively to define dispersant levels, pH, and temperature effect, and screening of various pigment surfaces. Levels: Very thixotropic—flocculated; Slight thixotropy—slight interaction on flocculation; Newtonian—deflocculated; Dilatant—deflocculated but not desirable state

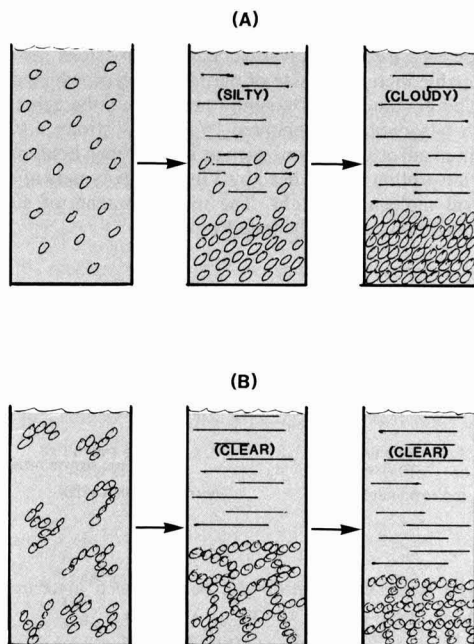


Figure 5—Settling and dispersion stability. A—settling of dispersed (deflocculated pigment), shows slow persistent settling to a hard packed cake that resists redispersion; B—settling of flocculated pigment, shows rapid settling to soft voluminous bulk pigment that is easily redispersed. (Patton, T.C., "Paint Flow and Pigment Dispersions," Wiley & Sons, Inc., New York, 1979, pp. 541-545)

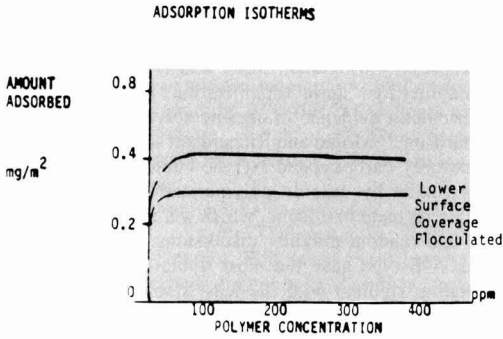


Figure 6—Adsorption isotherms

For steric stabilization to be effective we need polymers/dispersants (1) that can be strongly attached to pigment surfaces, (2) that maintain complete coverage of surface (high efficiency/unit weight), and (3) in which the solvated part must extend into medium to form barriers of sufficient thickness. The initial theories predicted that stability can only be achieved with dispersing polymers of very high molecular weight (100,000 ±). However, more recent evidence<sup>9,13</sup> indicates that 100 Å thickness is sufficient. The most important factor is the formation of stable and strong "anchors." For dispersants from 1000 to 10,000 Mw, stability does increase with molecular weight. For practical reasons, such as viscosity, compatibility, etc., most pigment dispersants used in non-aqueous systems are of molecular weight less than 10,000 unless the vehicle polymers itself is used as dispersant.

**Depletion Stabilization**

The depletion stabilization mechanism proposed by Napper<sup>14</sup> is based on a concept that repulsion can result due to depletion of polymer concentration between two approaching surfaces, resulting in lower polymer concentration between the surfaces than in the bulk. This mechanism does not need adsorption of polymers and it should apply to aqueous and non-aqueous systems. However, at this point, very little work has been done to determine its contribution to the overall stability of pigment dispersions.

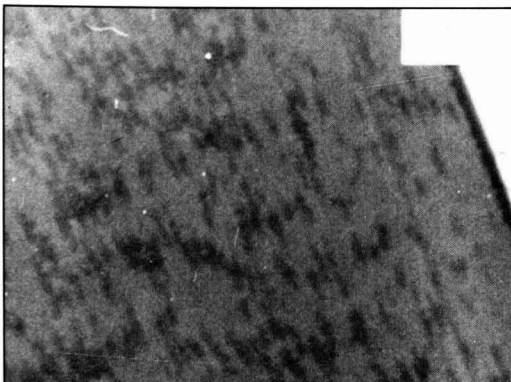


Figure 7—Incompatible dispersant used in a coating system

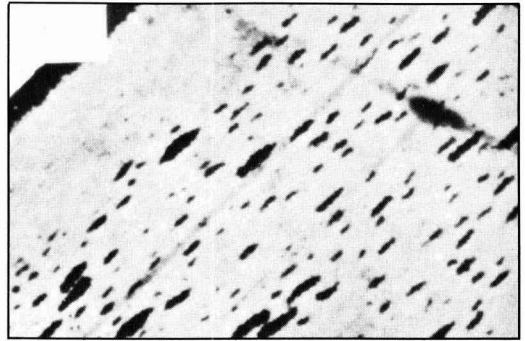


Figure 8—Incompatible dispersant used with transparent iron oxide and polymer

**PIGMENT DISPERSION STABILITY ASSESSMENT**

To what extent the particles have been separated in concentrated dispersions and how stable these dispersions are on standing and during subsequent processing are the critical questions to be answered from physical measurements of these dispersions. At this time, there is no single method or measuring device for obtaining this assessment for concentrated dispersions. Most measuring devices involve dilutions of concentrated dispersions which can change dispersion characteristics. For assessing concentrated dispersions, rheological<sup>15,16</sup> measurements are the most reliable route. However, to obtain a total picture, a variety of measuring devices and techniques are used for characterization of dispersions. The assessment of the state of dispersion can be determined by the following experimental techniques:

- (1) Microscopy (light, electron)
- (2) Rheology<sup>15,16</sup> (low shear viscosity, "yield stress")
- (3) Sedimentation (rates of settling, and settling volumes)
- (4) Adsorption isotherms (amount of dispersant on pigment surface)

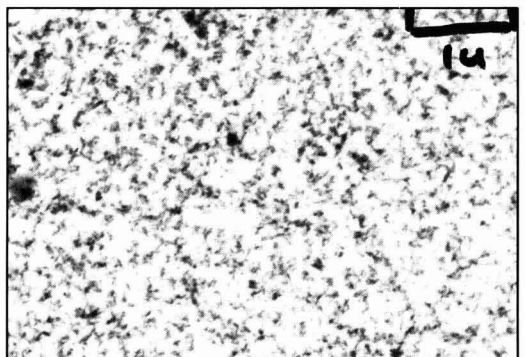


Figure 9—Compatible dispersant with transparent iron oxide and polymer

- (5) Particle size analysis<sup>17</sup>
- (6) Tinting strength (color development)
- (7) Fineness gauges (large particles)
- (8) State of dispersion upon letdown in solvents and vehicles using above techniques.

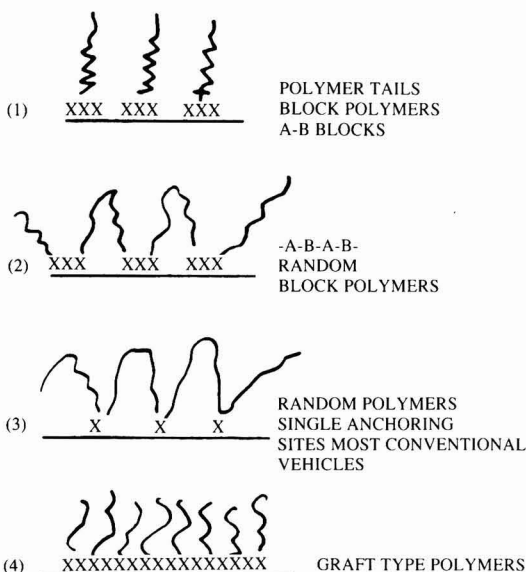
Figures 1-6 illustrate the differences between dispersions using the above techniques.

### CHEMISTRY AND DISPERSANT DESIGN CONSIDERATIONS

In organic media the "steric stabilization" mechanism predominates. In the design consideration of dispersant, one must consider two separate positions of dispersant or polymers:

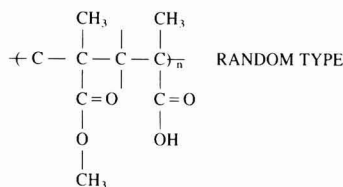
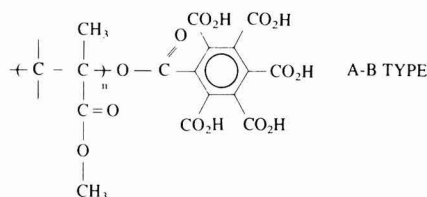
- (1) "Anchoring" site which adsorbs or bonds to pigment surface
- (2) Solvated portion (loops and tails) of sufficient thickness to prevent attraction of pigment particles. The solvated portion must be compatible (no significant phase structure) with film-forming vehicles. This requirement of compatibility is important in maintaining coating properties. Figures 7-9 illustrate when incompatible dispersant is used in a coating system. Figure 7 is dispersant plus film-former, showing incompatibility (phase structure-electron micrographs). If this dispersant is used with fine pigments, such as transparent iron oxide, and incorporated in the same polymer, the pigment is concentrated in the dispersant phase (Figure 8). Figure 9 is the same pigment with a compatible dispersant and polymer. This last requirement in many cases forces the formulators to use the film-forming polymers as dispersants for pigments.

For dispersing pigments, the following type polymers with their configurations at surfaces have been used as dispersants:



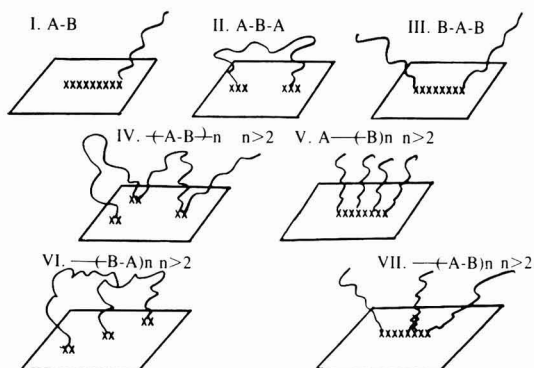
Clayfield and Lumb<sup>6</sup> and Meier<sup>18</sup> from theoretical considerations predicted that polymers with loops and tails and especially with tails (Case 1 and 4) should be favored for stability by "steric" stabilization. There is also some experimental evidence in organic solvent by Molan and Richardson.<sup>19</sup> Molan and Richardson studied dispersions of partially carboxylated styrene-butadiene block polymers (made by anionic polymerization) with titanium dioxide pigment in toluene, which were compared to fatty acid and random polymer dispersions. They found that block A-B type gave the most stable dispersions (sedimentation studies) with the least stable being the fatty acid.

Further evidence for A-B type over random polymers was obtained by investigators at Du Pont from a comparison polymethyl methacrylate carboxy terminated with polymethyl methacrylate-methacrylic acid random polymer dispersions (titanium dioxide and quinacridone pigments):



The A-B type (1) gave the lower viscosity (low shear, Brookfield) pigment dispersions.

In addition to the case above, a series of polymers were prepared with the following surface configurations:





The chemistry used in preparation of these dispersions was based on hydroxyl, amine isocyanate reactions and graft polymerization as shown in *Scheme 1*.

The polymers were used to prepare titanium dioxide (R-902) and quinacridone (RT-796 D Du Pont) dispersions. From microscopy and rheology (Brookfield LV, spindle #2 at 20 rpm) measurements, the following conclusions were made:

The polymers examined having structures I, III, V, VII will function as dispersants. The most efficient dispersant is of structure A-B type I. Polymers of structure type II, IV, and VI (postulated) did not behave as dispersants and acted as flocculants. No attempt was made to enlarge or optimize the loop size vs the size of A-groups for II, IV, VI structures where they would act as dispersants. The size of loops for random type or random block polymers is a very important factor in obtaining sufficient thickness of the solvated layer. Lubbock and Kershaw<sup>20</sup> studied (rheological method) the state of TiO<sub>2</sub> dispersions in xylene using lauryl methacrylate-glycidyl methacrylate random polymers. They have shown that dispersant efficiency passes through a maximum as anchor concentration increases. At low concentration of anchors, they found low degree of adsorption and at high concentration (postulated), a very flat adsorption (insufficient thickness). The present thinking is that structures II, IV, and VI can also be made to behave as dispersants if the size of loops and the size of A-groups were optimized. However, the "bridging" of pigments by A-groups for structures II, IV, VI cannot be entirely eliminated, giving rise to higher low shear viscosities than for structures I, III, V and VII. We elected to pursue further A-B type dispersants because of their greater efficiency (less dispersant needed) and ease of synthesis (large selection of A-groups) using

combinations of free radical, polyester, and polyurethane chemistry, as shown:

#### A-B TYPE DU PONT

3,684,771	1975
3,788,996	1974
4,070,388	1976
4,032,698	1977

#### BRITISH

1,339,930	1973
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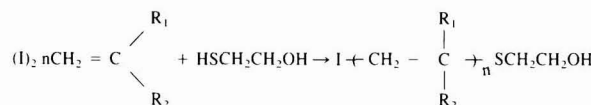
For an example, see *Table 2*.

Dispersant components, pigments, and solvents and their contribution to the state of dispersion are:

**A-SEGMENTS:** A large variety of A-adsorbing groups were examined for A-B type structures. Some of these groups and their interactions (flocculation, deflocculation) with titanium dioxide (R-902, Du Pont) and quinacridone (RT-796D, Du Pont) are shown in *Table 3*. The most important function of A-segment is its ability to adsorb and strongly interact (hydrogen bonding, acid-base type interactions) with pigment surface. The ability of an A-segment to perform its required function not only depends on its own structure, but also on the kind of pigment surfaces and solvents used. The stronger the interaction of A with the pigment surface, the more stable the dispersion will be with respect to polar impurities, and in turn to flocculation. The interaction of an A-group is high in multifunctional or large A-groups as shown in *Table 3*, (tribenzoic vs pentabenzic acid) and weak in small or few adsorbing groups. The importance of A-group size on the state of dispersions for polycaprolactone-polyethyleneimine type A-B polymers is illustrated by *Figure 10*. The lowest dispersion viscosities

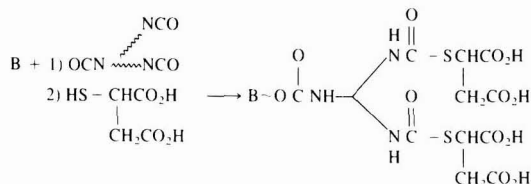
**Table 2—Chemistry of Dispersants Having Terminal Polar Groups  
(A-B, TYPE-I)**

#### Acrylics



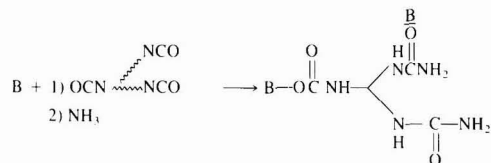
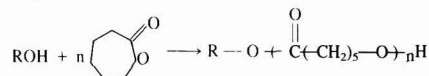
$$R_1 = H, CH_3$$

$$R_2 = -CN, -CO_2CH_3$$

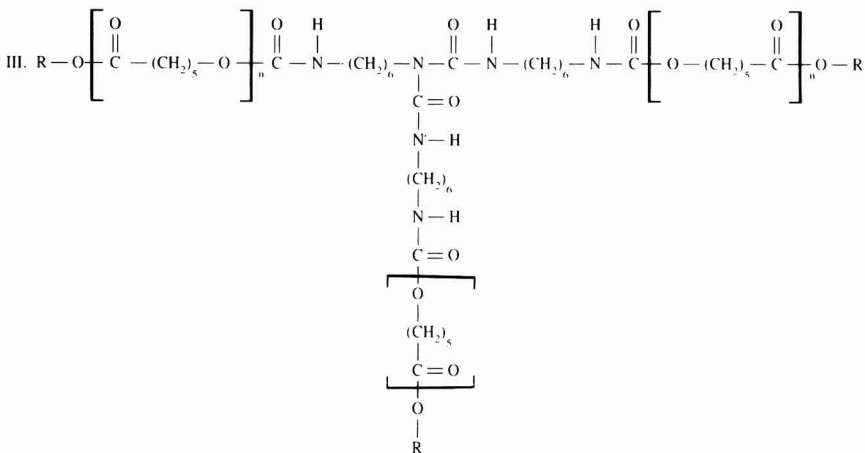
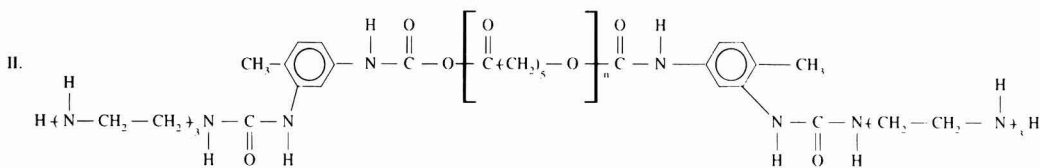
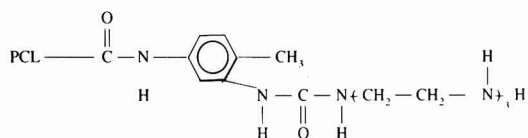
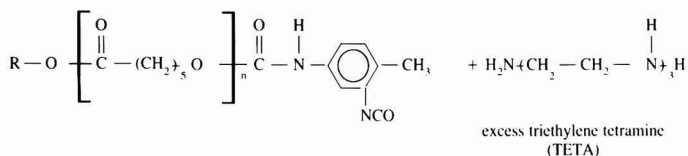
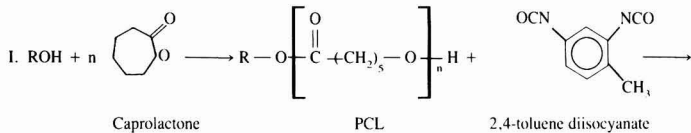


B-A

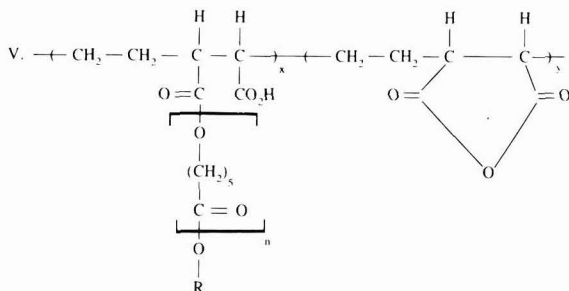
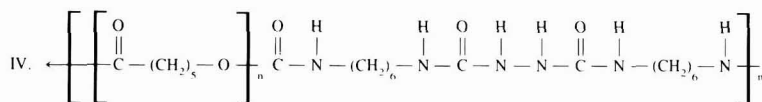
#### Polyesters



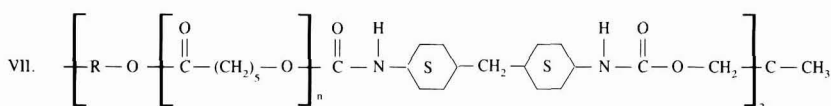
**Scheme 1**



## Scheme 1 (Continued)



VI. NOT MADE BUT ASSUMED TO BE LIKE II.



were obtained with A-segment between 146-600 molecular weight for B-segments of 3000 molecular weight. At lower levels than 146 and higher than 600 for polyethyleneimine A-segments, the dispersions were flocculated. Similar observation was made by Molan and Richardson<sup>19</sup> that A-segment size vs dispersion stability goes through a maximum. The explanation for this observation is that low levels of functionality in A-segment result in weak adsorption and low surface coverage. Conversely, at very high levels, too few tails or B-segments are present on the surface to prevent pigment attraction forces. Also, it is evident from Table 3 that more than one type of A-group is needed to satisfy a variety of pigments and pigment surfaces presently used. The interaction of A-segments with surface can be weakened or even displaced by stronger adsorbing groups that may be present in solvents, catalysts, polymers, etc. More will be said about this under discussion of solvents. In general, poor solvents are preferred for A-segments to maximize the interactions between surfaces and A-segments.

**B-SEGMENTS:** As already stated, the function of the B-segment is to provide strongly solvated tails of sufficient length and, in turn, barriers of sufficient thickness to counteract the attractive forces of particles. The B-segment should have very good solubility in the solvents

used and good "compatibility" (void of phase structure) with vehicle resins. The molecular weight of B-segment depends on the size of pigment particles. However, for most automotive type pigments (10-500 nm size) the molecular weight of the B-segment should be no less than 500-1000 and for all practical purposes no higher than 10,000. The most critical parameter in achieving stable dispersions is the strength of A-segment interactions. The effect of molecular weight of B-segment vs rheology for

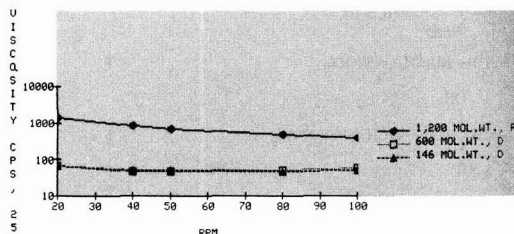
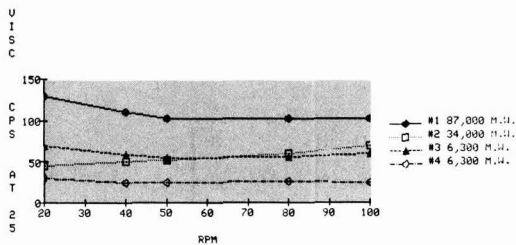


Figure 10—Molecular weight of A-segment viscosity of dispersions (15% BT-796D Monastral red, 5% dispersant, 80% toluene) vs shear rate. Dispersant: B-segment—3000 mol wt polycaprolactone, A-segment—polyethyleneimine (1200, 600, 146). D—deflocculated; f—flocculated; viscosity—Brookfield LV, spindle #1



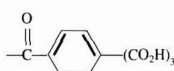
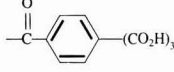




**Figure 11**—Molecular weight of B-segment. Viscosity of dispersions (15% RT-796D Monastral red, 5% dispersant, 80% toluene) vs shear rate. Dispersant: PMMA (87,000; 34,000; 6,300; 6,300) B-segment; triethylene tetramine A-segment—No. 4 Desmodur-N-teta A—segment. D—deflocculated; viscosity—Brookfield LV, spindle #1 at 25°C

**Table 3**—Efficiency of Adsorption Promoting Groups in A-B Dispersants<sup>a</sup>

B = poly(caprolactone-3000)  
Deflocculation Stability of Dispersion<sup>b</sup>

Segment A—	5% 819 Toluene <sup>c</sup>	5% 185 Toluene <sup>d</sup>
None	F	F
—TDI—OCH <sub>3</sub>	F	L.F
—TDI—NH—CH <sub>2</sub> CH <sub>2</sub> — 	D	D
—TDI—NH—CH <sub>2</sub> CH <sub>2</sub> — 	D	D
—TDI—(OCH <sub>2</sub> CH <sub>2</sub> ) <sub>7</sub> —OH	F	D
—TDI—(OCH <sub>2</sub> CH <sub>2</sub> ) <sub>7</sub> —O—TDI—P(OL)	F	D
—TDI—NH(CH <sub>2</sub> CH <sub>2</sub> NH) <sub>3</sub> —H	D	D
—TDI—NH—(O—CH—CH <sub>2</sub> ) <sub>n</sub> —NH <sub>2</sub> —“400”   CH <sub>3</sub>	D	L.F.
—TDI—O—(CH <sub>2</sub> CH <sub>2</sub> O) <sub>m</sub> —CH <sub>3</sub> —“350”	F	D
 —(CO <sub>2</sub> H) <sub>3</sub>	F	F
 —(CO <sub>2</sub> H) <sub>3</sub>	F <sup>e</sup>	D
—TDI—NH(CH <sub>2</sub> ) <sub>3</sub> —Si(OEt) <sub>3</sub>	F	F
—CH <sub>2</sub> CH(OH)CH <sub>2</sub> —O(CH <sub>2</sub> ) <sub>3</sub> —Si(OCH <sub>3</sub> ) <sub>3</sub>	F	F
—SO <sub>3</sub> H	F	L.F.
—TDI—NH(CH <sub>2</sub> CH <sub>2</sub> NH)—H ~14	D	D

(a) 5% surfactant

(b) F = Flocculated L = Lightly

V.L. = Very Lightly D = Deflocculated

(c) 819—Pigment-Quinacridone, Monastral<sup>®</sup> Red B, Du Pont RT-796D. “Polar component” = 0.22

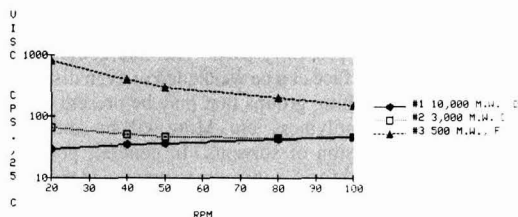
(d) 185—Pigment-Titanium dioxide—Du Pont R-902

(e) Originally deflocculated; on standing polymer & dispersion flocculated

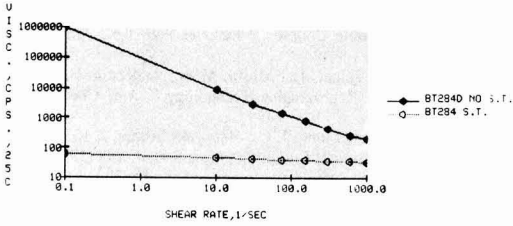
two systems based on polymethyl-methacrylate-triethylene tetramine, polymethyl methacrylate-Desmodur-N<sup>®</sup> (Mobay aliphatic polyisocyanate) and polycaprolactone-polyethyleneimine A-B block polymers is illustrated by Figures 11 and 12. The polymethacrylate polymers (Figure 11) compared had molecular weights between 6,300-87,000. All dispersions of PMMA were deflocculated. The lowest viscosities were obtained with 6,300-34,000 molecular weights. The lowest viscosity was obtained with PMMA-Desmodur type dispersant (PMM = 6,300 Mw). The polycaprolactone polymers had molecular weight ranging between 500-10,000. The most flocculated dispersion was with B-segments of 500 molecular weight. Above 3000 molecular weight, very little difference was noted.

**PIGMENTS:** To form strong attachment of A-groups or anchors of A-B block polymers on the pigment surface, one must have a good knowledge of the specific pigment surface. The chemical structure of pigment (phthalocyanine, quinacridone, etc.) itself is important, but what compounds are on its surface (surface treatments, process impurities, etc.) will determine the bonding strength of these A-blocks to the surface. The pigment manufacturers have recognized that the pigment surface treatments can change the level of flocculation of pigment dispersions and are using this approach to manufacture “flocculation resistant,” “easily dispersed,” and “improved flow-rheology” type pigments. The effect of pigment surface treatment on the state of dispersion is illustrated by Figure 13. Approximately a 1000 fold decrease in viscosity at 0.1 l/sec shear rate can be achieved by a surface treated pigment as compared to a non-treated one. The A-segment, in the case of surface treated pigment, strongly interacted, providing a “steric” barrier to flocculation. Therefore, the characterization of pigment surfaces is necessary if one is to understand the polymer-pigment-solvent interactions, and how they relate to dispersion stability and the degree of flocculation.

Pigments have been characterized by particle size and shape, chemical structure of surfaces,<sup>21</sup> surface energy,<sup>22-24</sup> solubility parameter,<sup>25,26</sup> acid-base,<sup>27</sup> pH, etc. The type of interactions for obtaining strong bonding of polar A-block with pigment surfaces are “hydrogen bonding,” “acid-base,” and in some cases “covalent.” It has been observed that pigments containing hydroxyl,



**Figure 12**—Molecular weight of B-segment viscosity of dispersions (15% RT-796D Monastral red, 5% dispersant, 80% toluene) vs shear rate. Dispersant: polycaprolactone (10,000, 3,000, 500 MW) B-segment, polyethyleneimine 146 MW A-segment. D—deflocculated; f—flocculated; viscosity—Brookfield LV, spindle #1 at 25°C



**Figure 13—Phthalocyanine pigment dispersions rheology—surface treatment. Dispersion—pigment (BT-284D no treatment, BT-284D surface treatment) 16.5% wt, A-B type dispersant—2.5%, polymer—26%, solvent—xylene; process—attritor. Data: Haake roto, at 25°C**

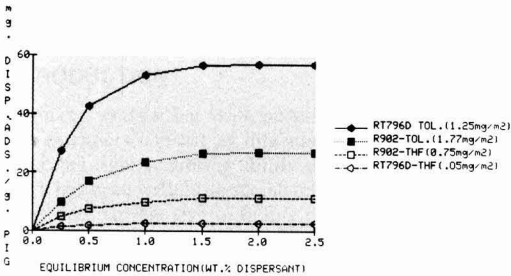
carboxyl, oxides, and other polar groups can be deflocculated to a greater degree than pigments without these groups. It has been postulated that this greater degree of deflocculation is due to polar-polar interactions between groups on the surface and polymers. Further evidence that adsorption of A-B block polymers is enhanced by polar pigment surfaces was obtained by S. Wu and K. J. Brzozowski<sup>2,3</sup> from determination of the surface free energy of pigments, and more importantly, the determination of the polar component of surface free energy. The total surface free energy is:

$$Y = Y^d + Y^p$$

where  $Y$  = surface free energy,  $Y^d$  = dispersion component of surface free energy and  $Y^p$  = polar component of surface free energy. The results were obtained by two contact angle measurements (water and methylene iodide) on pressed pigment discs. The results for some of the pigments are given in Table 4. Pigments having high polar component of surface free energy show greater degree of deflocculation than pigments with low polar component of surface free energy in a non-polar solvent and A-B block polymer. Adsorption isotherms show very little adsorption of A-B block polymer on pigments having low polar surface free energy.

**SOLVENT:** The major requirement of solvents used in preparation of non-aqueous dispersions with A-B type polymers are:

(1) Solubilization of the B-segment which provides polymer tails and steric barrier to pigment flocculation.



**Figure 14—Adsorption of PCL-teta from toluene and tetrahydrofuran on quinacridone (RT-796D) and titanium dioxide (R902)**

**Table 4—Surface Free Energy of Pigments**

Pigment Type	Surface Free Energy, Dynes/cm <sup>2</sup>		Polarity Y	State of Dispersion
	Y <sup>d</sup>	Y <sup>p</sup>		
α phase Copper phthalocyanine BT-425D	39.4	10.1	49.5	DF-LF
α phase Copper phthalocyanine BT-284D	40.0	6.9	46.9	F
Maroon, Naphthoic, RT-695D	27.8	24.1	51.9	D

Dispersant = A-B Block PMMA/2EHA—Desmodur-N Type,  
Solvent = Toluene, 5% pigment in dispersion

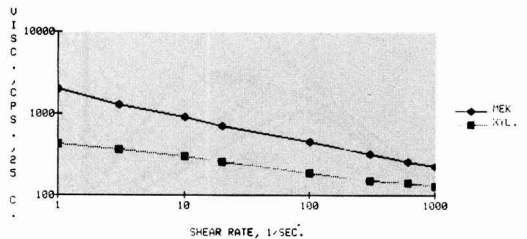
F = flocculated; DF = deflocculated; LF = lightly flocculated.

The solvent should be a good solvent for the B-segment. If a poor solvent is used, the tails can collapse, resulting in coagulation of pigment dispersion. This process is reversible and stable dispersions can be regenerated by addition of good solvent to the dispersion.

(2) Enhancement of A-polar polymer block adsorption to the pigment surface. The solvents should not compete with A-groups for adsorption sites on pigment surfaces.

In general, “poor” solvents for solubility of A-segments are required. The solvent can affect the level of dispersant adsorbed on the surface and is illustrated by Figure 14. The amount of A-B block polymer adsorbed on pigment surface is much greater from solvents like toluene than for tetrahydrofuran. Furthermore, for pigments with weaker or fewer adsorbing sites, as in case of quinacridone, almost all of the A-B is displaced by tetrahydrofuran. Figure 15 shows the effect of solvent on rheology. Again, the highest low shear viscosities were obtained with methyl ethyl ketone than xylene containing pigment dispersions.

The control of solvent and other additives (catalysts, stabilizers, etc.) is very critical in maintaining stability of pigment dispersions and formulated coatings.



**Figure 15—Xylene and methyl ethyl ketone solvents rheology of hostaperm yellow H3G dispersions. Pigment—35 wt %, A-B type dispersant—5.25%, polymer—18.0%; solvent—41.75%. Data: Haake roto, at 25°C**

## SUMMARY

Pigments are stabilized in non-aqueous coating systems predominantly by steric stabilization with loops and tails from polymers or dispersants providing the repulsive barriers to flocculation. A-B type block polymers having polar A-groups and non-polar B-groups are superior pigment dispersants. A-B structures provide an opportunity in designing dispersants with a variety of A-groups and B-groups to accommodate a variety of pigment surfaces and film-forming polymers. Strong adsorption and interaction (hydrogen bonding, acid-base) of A-groups with pigment surface and total surface coverage is critical in maintaining stable dispersions. The interaction of A-polar block with the pigment surface is of polar nature. The size and type of A-group determines the strength of this interaction. The B-group provides the tails. The more polar the pigment surface (polar surface free energy), the greater is the deflocculation potential of pigment dispersions. Solvents and additives can compete for the adsorption sites on the pigment surface and thus alter the state of dispersion to a more flocculated state. The pigment dispersion in an organic media is not a simple situation. Interactions of dispersant (polymer)-pigment surface, polymer-solvent, pigment-solvent, and all other additives must be well defined before the state of the dispersion can be predicted or understood.

## ACKNOWLEDGMENT

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# Influence Of Fine-Particle Size Extenders On the Optical Properties Of Latex Paints

Luigi Cutrone  
Tioxide Group PLC\*

The "spacer" theory suggests that coarse extenders crowd titanium dioxide particles together, reducing the ideal spacing at which the pigment affects maximum light scattering. When extender particles of a size closer to that of titanium dioxide are used, they move between the titanium dioxide particles and space or separate them for greater scattering efficiency. This paper gives examples of light scattering phenomena which are better explained by a new model involving air encapsulation. For example, fine-particle size extenders were found not to enhance the scattering ability of titanium dioxide but merely maintain the same level of opacity as binder is removed at low concentration of titanium dioxide (10% PVC), and a reduction in scattering efficiency as the titanium concentration was increased (20 and 30% PVC). A number of fine-particle size extenders were used in the study and none improved the scattering efficiency of titanium dioxide.

## INTRODUCTION

For many years it has been postulated that fine-particle size extenders can act as diluents or "spacers" to enhance the light scattering ability of titanium dioxide.<sup>1-6</sup> Much has been written about the relationship of refractive index and particle size to hiding power,<sup>7-9</sup> but to aid in the understanding of the spacer theory, background information about light scattering will be given. Light scatter-

ing by particulate matter can be considered to consist of three separate phenomena: surface reflection, refraction, and diffraction.<sup>10</sup>

Surface reflection and refraction impart opacity due to the difference in refractive index between the pigment and the medium in which the pigment is dispersed in accordance with Fresnel's equation: [equation (1)].

$$F = \frac{(n_1 - n_2)^2}{(n_1 + n_2)^2} \quad (1)$$

where  $F$  is reflectivity,  $n_1$  is the refractive index of the pigment and  $n_2$  is the refractive index of the medium. Scattering by diffraction, however, contributes to opacity by a more complex mechanism. For this purpose light is considered as a wave phenomenon. An important characteristic of a wave is the bending from its original path when it passes through an obstructing medium. With

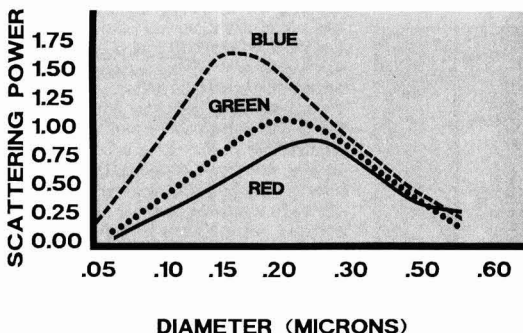


Figure 1—Relative scattering power of TiO<sub>2</sub>

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\*Central Laboratories, Stockton-on-Tees, Cleveland TS18 2NQ, England.

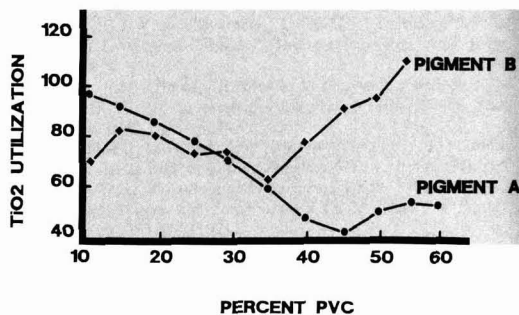
**Table 1—Titanium Dioxide and Extenders Particle Size Determined by Transmission Electron Microscope**

	Particle Size ( $\mu\text{m}$ )
Calcium carbonate (fine)	0.5
Calcium carbonate (coarse)	0.7
Aluminum silicate (delaminated)	0.4
Sodium silico aluminate	0.3
Hollow synthetic beads	0.5
Vesiculated beads	0.25
Titanium dioxide	0.24

large objects, diffractive bending of light is of no significance to a pigment manufacturer, since the angle by which it is bent is less than the ratio of wavelength to diameter. As the particle becomes smaller, two phenomena occur. First, the dimensions of the particle and the wavelength of light become comparable, so that the scattered angle increases; and second, as the mass of the particle decreases, the charges in the particle oscillate in concert with the electromagnetic wave of the passing light and, in turn, these oscillating charges produce a change in the electromagnetic field and change the direction of the light.<sup>11</sup> Since these two phenomena are additive, the effect of particle size on scattering power is very important, as shown in Figure 1.<sup>10</sup> As the particle size is reduced even further, it becomes so small that it is not recognized by the beam of light, hence the light scattering power is reduced. The optimum scattering occurs at approximately half the wavelength of light.<sup>12</sup>

Particles of optimum size can scatter approximately four times as much light as actually impinges on them, because the scattering cross section is about four times as great as the geometrical cross section of the particle. From this it may be said that maximum scattering is proportional to pigment content as long as the particles are more than half the wavelength of light apart, but above this level closer packing renders them less effective as scattering units.

Therefore, as the pigment loading in a paint film increases, the scattering power (which for white paints may be regarded as opacity) of the film increases linearly up to a pigment volume concentration (PVC) of about 10%. Beyond this point, although there is still an increase in opacity as the pigmentation level is increased, the scattering efficiency of the pigment is no longer being maintained at the maximum with respect to the scattering of

**Figure 3—Pigment utilization of two rutile grades**

light per particle. Several authors have derived expressions that quantify the loss in scattering efficiency.<sup>13-17</sup>

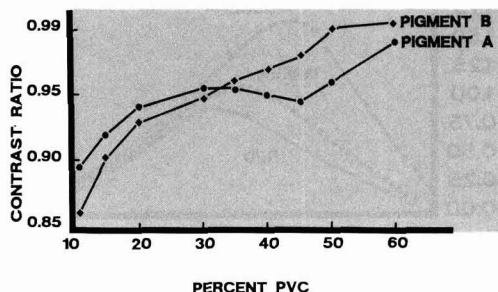
From the above, the theory evolved that fine-particle size extenders act as diluents or "spacers" to improve the optical efficiency of titanium dioxide.<sup>1</sup> There have been several claims stating that a given amount of titanium dioxide can be replaced by fine-particle size extenders and retain the same optical properties.<sup>2-6</sup> According to our findings this phenomenon does not occur below CPVC. What does happen however, is that the CPVC has been exceeded and dry hiding is obtained. Above CPVC, the opacity increases again, but the Fresnel equation is no longer applicable.<sup>18-19</sup> Density measurements show that above CPVC air is introduced into the film and this contributes to an increase in opacity.<sup>20</sup> Fine-particle size extenders have a large surface area and a high oil absorption. Therefore, when they are used, the effective PVC is moved into a higher range, and this increases the contribution of dry hiding. More opacity is obtained because more pigment/air interfaces have been introduced. This will adversely affect other film properties.

Another shortcoming of the spacer theory is that nearly all fine pigment particles such as titanium dioxide or similar size extenders are present mainly in the paint film as agglomerates, and not as discrete crystals.<sup>21-22</sup> For example, Hornby and Murley determined that, in a well dispersed system, only a small proportion (20%) of the titanium dioxide crystals is present as simple crystals, the remainder being present in groups of two, three, and more crystals.<sup>23</sup> Despite the above comments, it is still widely believed that the spacing concept is valid.<sup>4-6</sup>

An evaluation of different types of extenders in latex paint was carried out, and it was found that there were limitations to the current spacer model in explaining results.

## EXPERIMENTAL

Several classes of extenders were evaluated, including fine-particle size delaminated aluminum silicate, sodium silico aluminate, fine and coarse calcium carbonate, hollow synthetic beads, and pigmented vesiculated beads, (Table 1). The particle size of each extender was determined by examining a dilute dispersion of polyvinyl alcohol in a transmission electron microscope. Each extender was evaluated at PVC levels from 10% to 60% in an

**Figure 2—Opacity of two rutile grades**

acrylic emulsion system pigmented with 10%, 20%, and 30% titanium dioxide. The effect of blending coarse and fine-particle size extender on the spacing of titanium dioxide particles was also evaluated.

### Measurements

**OPACITY:** To assess the opacity of a given paint sample, plots of contrast ratio against spreading rate were drawn. Thus, four films of each sample, each of a different thickness, were applied to polyester film using wire wound applicator bars. After drying for 24 hours, the reflectance of each sample film over standard black and white tiles was measured using a HunterLab colorimeter equipped with a Y tristimulus filter. Contrast ratios were then calculated using equation (2).

$$\text{Contrast Ratio} = \frac{\text{reflectance over black tile}}{\text{reflectance over white tile}} \quad (2)$$

Using measurements of film weight, specific gravity, and total paint solids, a spreading rate for each film was calculated and plotted against contrast ratio. By graphical interpolation, the contrast ratio for each sample at constant dry film thickness of 1 mil was determined.

### RESULTS AND DISCUSSION

In a paint film containing no extender, the separation of titanium dioxide particles is directly related to the formula PVC, the higher the pigment volume concentration, the smaller the spacing between individual pigment particles. Furthermore, the higher the formula PVC of such a paint film, the lower the amount of white hiding power produced for a given weight of titanium dioxide, or the lower its optical efficiency.<sup>1,15</sup> This is seen in Figure 2, where the pigmentation level of two markedly different grades of coated rutile titanium dioxide pigment is varied from 10% to 60% PVC. Increasing the concentration of pigment A (TiO<sub>2</sub> content = 95%) to a PVC of about 30%, produces an increase in opacity, even though the pigment utilization<sup>24</sup> decreases (Figure 3) due to the closer proximity of the pigment particles to each other. Pigment utilization is defined as the area of paint film that contains one kilogram of pigment (m<sup>2</sup>/Kg) when it is applied at a spreading rate which produces a contrast ratio of 0.90,

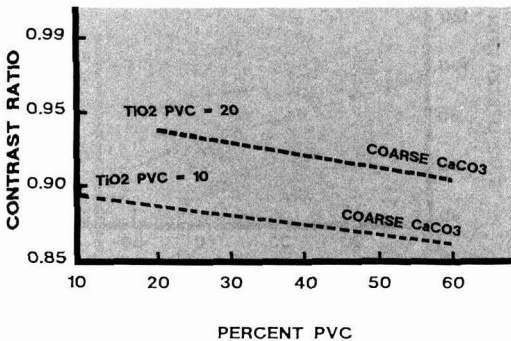


Figure 4—Effect of fine particle size extender on opacity at fixed TiO<sub>2</sub> PVC

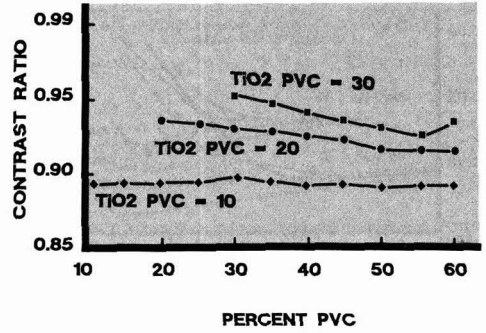


Figure 5—Effect of pigmented vesiculated beads on opacity at fixed TiO<sub>2</sub> PVC

and can be calculated using the following expression [equation (3)]

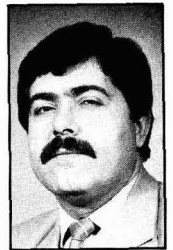
$$\text{Pigment utilization} = \frac{\text{SR } 0.90 \times 100}{p \times Z} \quad (3)$$

where SR 0.90 = spreading rate at 0.90 contrast ratio  
 p = density of wet paint  
 Z = percent pigment by weight of wet paint.

At a PVC in excess of 30%, the crowding effect becomes so strong that the opacity actually decreases, until a state is reached where just sufficient binder is present to satisfy the absorption properties of the pigment surface (critical pigment volume concentration). Beyond the CPVC, air is introduced into the film and therefore an increase in scattering power will occur (dry hiding).

For pigment B (TiO<sub>2</sub> content = 82%) below 30% PVC, the opacity is lower than for pigment A; this is primarily due to the lower titanium dioxide content per given volume. However, as the PVC is increased, pigment B shows an advantage over pigment A and, more importantly, it does not show a reduction in opacity. This is due to the nature of the coating on the pigment which produces a large surface area, and thus a higher oil absorption and a lower CPVC. Consequently, air is present

LUIGI CUTRONE obtained a B. Sc. degree in Chemistry in 1974 at Loyola College in Montreal and he continued his graduate studies at McGill University also in Montreal in Synthetic Organic Chemistry of Biologically Active Compounds. For several years he was at M.F. Paints in Laval where he headed the Research and Development Department. For five years he held the position of Supervisor of the Application Development at Tioxide Canada Inc., where he was responsible for Research and Development work in such areas as Rheology, Electrokinetic properties of pigments and Hi-solids. Presently, he holds the position of Manager of the Application Development section at Tioxide UK Ltd. in England. He has held several chairs in the Montreal Society and he has published several papers.





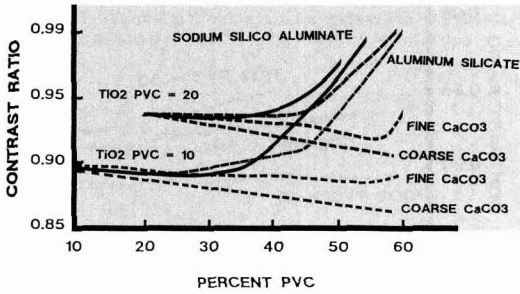


Figure 6—Effect of fine particle size extender on opacity at fixed  $TiO_2$  PVC

in the film at a lower  $TiO_2$  concentration, giving an increase in opacity.

Introducing an extender into the matrix, depending on particle size and refractive index, will have a marked effect on the opacity profile. According to the Fresnel equation, most extenders, having approximately the same refractive indices as polymer binders, should have no effect on hiding, but because the particle size of most extenders is considerably greater than that of  $TiO_2$ , they actually reduce the scattering efficiency of titanium dioxide, hence reducing opacity. This is seen in Figure 4, where two latex paint samples containing constant amounts of titanium dioxide, 10% and 20%, respectively, give poorer opacity as the concentration of coarse calcium carbonate extender, of mean diameter 2.5 microns, increases.

An exception to this is pigmented vesiculated beads<sup>25,26</sup> which do not seem to reduce the scattering efficiency at low concentrations of  $TiO_2$  (Figure 5). In actuality, the relatively large pigmented vesiculated beads do crowd the small  $TiO_2$  particles, but the loss in scattering efficiency is compensated for by the internal

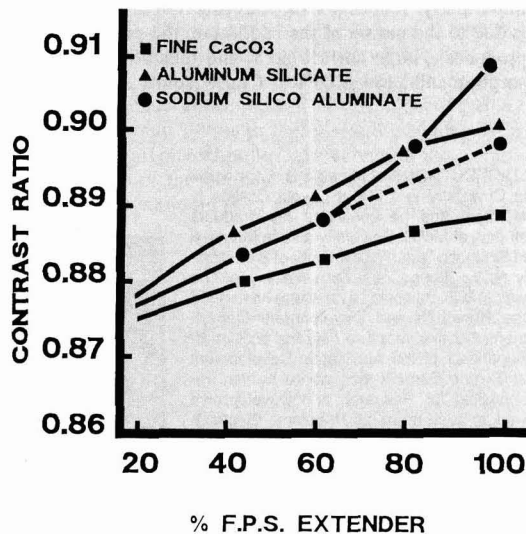


Figure 7—Replacement of large particle with fine particle size extender

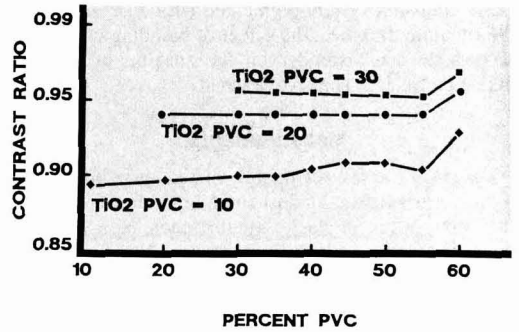


Figure 8—Effect of hollow synthetic beads on opacity at fixed  $TiO_2$  PVC

pigment and air. This is more apparent when the titanium dioxide content is increased to 20 and 30% PVC, where the internal pigment and air cannot compensate for the greater crowding effect, hence a slight reduction in scattering efficiency is observed.

If, on the other hand, a finer particle size extender such as calcium carbonate, delaminated aluminum silicate, or sodium silico aluminate extender is used (Figure 6), the opacity at low concentration of  $TiO_2$  is maintained at a value originally imparted by the unextended titanium dioxide. A slight increase in opacity is observed below CPVC (45%) with delaminated aluminum silicate. This is due to the platy character of the particles, which encapsulate air as the extender concentration is increased, giving improved scattering.<sup>27</sup> This increase, however, disappears as the  $TiO_2$  concentration is increased, due to the crowding effect and the inability of the fine particle size extender to efficiently separate the titanium dioxide particles. At high  $TiO_2$  concentration, there is actually a slight reduction in scattering efficiency with fine particle size calcium carbonate and sodium silico aluminate. In the past, because of the fact that the particle size of most paint extenders was larger than the titanium dioxide, any mixture of the two would result in the packing of the finer titanium dioxide particles into the voids existing between the coarser extender particles. When, however, an extender of the same particle size as titanium dioxide was

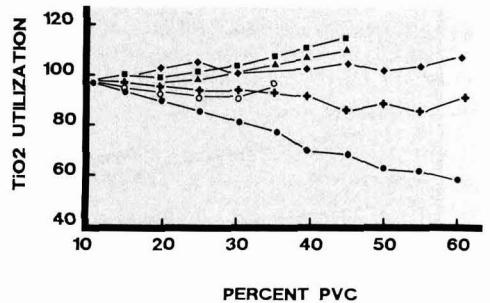


Figure 9—Effect of fine particle size extender on pigment utilization at 10%  $TiO_2$  PVC. Legend: ●—coarse  $CaCO_3$ ; ○—sodium silico aluminate; ◆—vesiculated pigmented beads; +—fine  $CaCO_3$ ; ▲—aluminum silicate; ■—hollow synthetic beads

**Table 2—Opacity at a PVC/CPVC Ratio of 1.10**

Extender	TiO <sub>2</sub> Level (PVC)		
	10%	20%	30%
Calcium carbonate (fine)	0.921	0.940	0.959
Hollow synthetic beads	0.928	0.956	0.966
Aluminum silicate (delaminated)	0.921	0.955	0.963
Sodium silico aluminate	0.912	0.953	0.968

**Table 3—Opacity Rate Increase Above CPVC**

Extender	TiO <sub>2</sub> Level (PVC)		
	10%	20%	30%
Calcium carbonate (fine)	1.62	1.64	1.76
Hollow synthetic beads	2.07	1.62	1.65
Aluminum silicate (delaminated)	1.81	1.46	1.36
Sodium silico aluminate	1.60	1.70	1.40
TiO <sub>2</sub> only	—	1.86	—

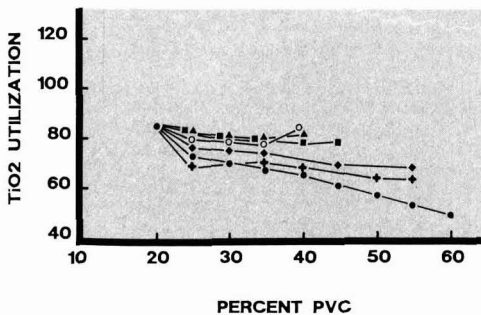
introduced, the titanium dioxide present in the voids was spaced and the optical efficiency was improved.<sup>2</sup>

According to the findings of this evaluation, the use of fine-particle extenders will improve the optical efficiency of a system whose optical properties have been impaired by the use of particles larger than that of titanium dioxide. This is shown in *Figure 7* where a sample at 40% PVC with 10% PVC titanium dioxide and extended with an extender of 2.5 microns is blended with another sample of the same PVC but extended with a fine-particle size extender. The opacity increases linearly as the amount of the fine-particle size extender is increased. The only exception is sodium silico aluminate, which upon increasing its concentration, CPVC is encountered and the contrast ratio increases markedly. When all of the coarse extender is replaced with the fine extender, the level of opacity has increased to its maximum, suggesting that larger particle size extender should not be used in coatings because they crowd the smaller titanium dioxide particles, hence reducing the scattering power. With the newer types of extenders, such as hollow synthetic beads and pigmented vesiculated beads, as the PVC is increased at low TiO<sub>2</sub> concentration, there is a slight increase in opacity, more in the case of hollow synthetic beads as seen in *Figures 5* and *8*. The slight increase in opacity which is observed is not due to the spacing of titanium dioxide, but is produced by the internal reflections of the spherical voids filled with air.<sup>28</sup> The increase in opacity diminishes as the TiO<sub>2</sub> concentration is increased, and increases more markedly in the case of pigmented vesiculated beads, because of their larger particle size. At higher TiO<sub>2</sub> concentrations, where fine-particle size extenders should separate the TiO<sub>2</sub> particles, there is no improve-

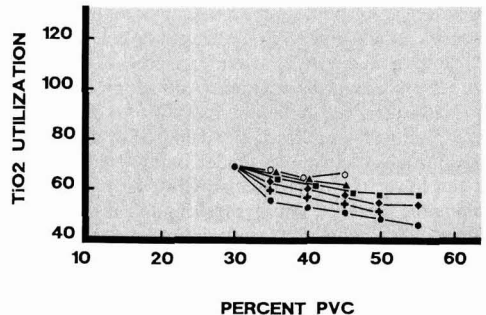
ment in opacity. In most cases, there is a reduction in opacity, hence a reduction in scattering efficiency.

Above CPVC, it is unfair to compare opacity at a constant PVC. Each extender, having a different particle size and medium demand, will inevitably exhibit a different critical PVC. A more meaningful comparison is to compare the opacity at constant PVC/CPVC ratio.<sup>29</sup>

Referring to *Table 2*, it can be seen that when fine-particle size extenders are compared at constant PVC/CPVC ratio, the opacity values for a given level of titanium dioxide are comparable. The only exception is fine calcium carbonate which shows a slight reduction in opacity. The reduction is due to its broader particle size distribution and the larger particles cause slight flocculation. Another phenomenon occurring above CPVC is the fast rate of increase in opacity. According to *Table 3*, the rate of increase in opacity<sup>18</sup> (defined as the greatest rate of change in opacity PVC after CPVC—e.g., see *Figure 6*) seems to be independent of the pigment with which the PVC is increased. Theoretically, according to Fresnel's equation, there should be a difference in scattering power depending on whether the air surrounds a titanium dioxide particle or an extender particle. In practice, however, this does not happen. The rate of increase in opacity is constant whether the matrix is rich in titanium dioxide or in extender. One possible explanation is: larger voids or bubbles formed after the CPVC contribute significantly to opacity, and because the scattering curve for bubbles does not fall off as fast as compared with titanium dioxide, crowding between bubbles is not a dominant factor.<sup>28</sup> Obviously the shape of the bubble is another factor which will affect both crowding and light scattering efficiency. This last point is very important because, accord-



**Figure 10—Effect of fine particle size extender on pigment utilization at 20% TiO<sub>2</sub> PVC. Legend: same as *Figure 9***



**Figure 11—Effect of fine particle size extender on pigment utilization at 30% TiO<sub>2</sub> PVC. Legend: same as *Figure 9***

ing to Ross,<sup>28</sup> the scattering power of flat bubbles parallel to the surface at 50% PVC is four times greater than that of spherical bubbles. In reality, the scattering power is probably even greater because of the greater number of reflections due to the irregular shapes of the voids. This also explains why the air inside spherical synthetic hollow beads and vesiculated pigmented beads shows only a slight increase in scattering power below the CPVC (*Figures 5 and 8*).

Further evidence was found that fine-particle size extenders do not enhance the scattering efficiency of titanium dioxide. This was done by employing "pigment utilization"<sup>24</sup> values for the two rutile grades and the six extenders as a function of their concentrations.

A decrease in pigment utilization represents a loss of scattering efficiency for titanium dioxide. Analysis of the results in *Figures 9-11* indicates that, for coatings containing 10% TiO<sub>2</sub> and extended with the various extenders up to a PVC of 60%, the pigment utilization is either less (for coarse calcium carbonate) or comparable (for fine calcium carbonate, sodium silico aluminate, pigmented vesiculated beads, hollow synthetic beads, and delaminated aluminum silicate). Above the CPVC (for sodium silico aluminate at 35% PVC and delaminated aluminum silicate at 45% PVC), the pigment utilization increases because of the introduction of air into the film. Increasing the TiO<sub>2</sub> concentration to 20% PVC, the pigment utilization is reduced, and it is reduced even further when the TiO<sub>2</sub> concentration is increased to 30%. This means that the pigment utilization depends primarily on titanium dioxide concentration. The presence of an extender either reduces the pigment utilization or maintains the same value, but in no instance does the pigment utilization increase below the CPVC. When the pigment utilization remains the same it means that some binder can be replaced with extender without affecting the scattering efficiency of the pigment. It can also be seen from *Figure 3*, that reducing the titanium dioxide content results in an increase in pigment utilization or scattering efficiency and this is simply due to a reduction in crowding as a consequence of increasing the particle-particle distance.

## CONCLUSION

Exceptions have been found to the claim that fine-particle size extenders can act as diluents or spacers to enhance the light scattering ability of titanium dioxide. Fine-particle size mineral extenders at low concentrations of titanium dioxide can, to a certain degree, replace binder without reducing the scattering efficiency of the pigment. However, at higher concentrations of titanium dioxide, the fine-particle size extenders fail to maintain the scattering efficiency as binder is removed. Because they can lower the refractive index of the medium surrounding the pigment, microvoids should cause a considerable increase in pigment scattering power. This occurs only above CPVC where air is randomly distributed in the matrix. Below CPVC, the encapsulated air is equivalent to fine-particle size mineral extenders.

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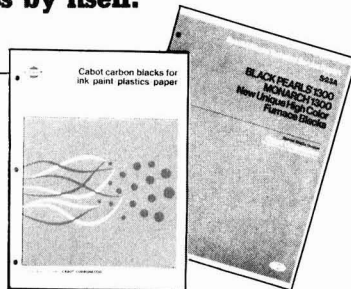
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# Structure and Properties Of Amorphous Silica Gel In Coatings Applications

Leon Kutik

W.R. Grace & Company, Davison Chemical Division\*

Amorphous silica gel is a unique type of synthetic silica which normally exhibits a high surface area, low density, and a porous structure. Each of these properties is controlled by the manufacturing process so that highly specialized products for a variety of coating applications result. Physical properties such as pore volume, surface area, and density are important considerations during the manufacturing process. Examples are shown how these parameters are modified to optimize the adsorption properties of silica gels which are used to remove moisture from water sensitive coating materials. Similarly, the effect of particle size, density, pore volume, and surface properties on the flattening efficiency of silica gel pigments is described. Flattening efficiency curves demonstrating the use of silica gel pigments in various industrial coating systems are presented. The use of recently developed silica gel thixotropes in various nonaqueous systems is also described, together with a comparison with other types of synthetic silicas.

## INTRODUCTION

Amorphous silica gel is a unique type of synthetic silica characterized by high surface area, porosity, structural integrity, and chemical inertness.<sup>1,2</sup> These properties have created a significant market for silica gel in the coatings and plastic industry as well as the food, personal products, and petrochemical industry.

To gain a better understanding of this product, let us review how silica gel is manufactured and illustrate how,

by selectively controlling or modifying specific processing conditions, one can obtain highly specialized and efficient products for the coatings industry. These products can be designed for either moisture adsorption, gloss control, and most recently, rheological properties.

Silica gel is normally manufactured by a procedure as shown in *Figure 1*. Sodium silicate is mixed with sulfuric acid to form a hydrosol. The hydrosol sets and undergoes a phase change to a gel-like structure known as a hydrogel. The hydrogel is broken into relatively small sections and washed to remove soluble salts and impurities. During the wash cycle the pore structure of the silica gel is developed.

Following the wash cycle heat is applied to remove water. By carefully controlling the heating and washing conditions, aerogel or xerogel structures are obtained with controlled physical properties. By definition a xerogel is a gel in which the liquid medium has been removed with the structure being compressed and the pore volume reduced. Surface area is generally high for these materials and the pore volume is lower than an aerogel. This type of structure generally results from slow drying. An aerogel

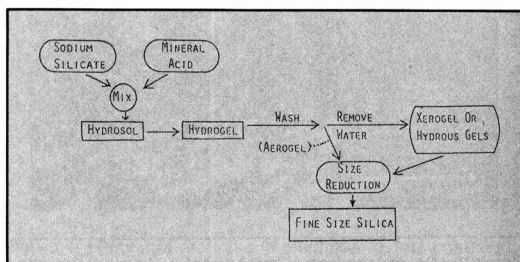


Figure 1 — General production scheme for silica gels

Presented at the 63rd Annual Meeting of the Federation of Societies for Coatings Technology, October 9, 1985, in St. Louis, MO.

\*Technical Center, 5500 Chemical Rd., Baltimore, MD 21226.



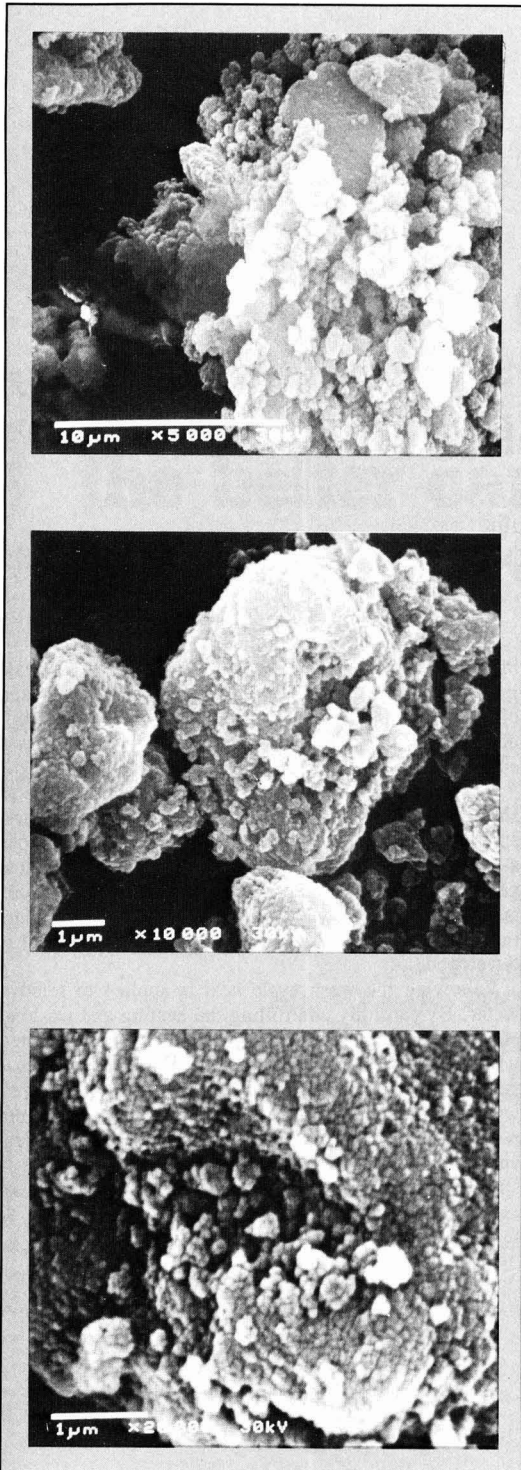


Figure 2—Scanning electron micrographs of a representative silica aerogel (Syloid 244) at increasing magnifications

is a gel in which the liquid is removed quickly to prevent shrinkage of the pores. Products of this type are characterized by high pore volume and low specific area.

## PROPERTIES OF SILICA GEL

Typical properties of two different types of xerogel as compared to a standard aerogel are shown in *Table 1*.

The final processing step involves grinding the gel to a specific particle size distribution and in some cases applying a surface treatment to provide lubricity or to improve suspension properties.

Key manufacturing parameters include: chemical nature of the wash, temperature of the wash, duration of wash, drying rate, size reduction, and surface treatment. These in turn affect the morphology of the final product with regard to bulk density, pore volume, pore diameter, adsorption properties, particle size distribution, surface area, and surface properties.

Currently, commercially available silica gels are available with various ranges of properties (*Table 2*).

### Pore Volume and Surface Area

Photomicrographs of a micronized silica gel illustrate the three dimensional structure of this product showing its high pore volume and surface area (*Figure 2*).

The surface of silica gel when manufactured and untreated has an affinity for many chemical substances with a preference for most polar compounds such as water. However, by varying the pore volume and surface area, the adsorption capacity of silica gel can be modified as indicated by *Figure 3*.

This difference in performance can be explained by the fact that the xerogel structure (Syloid AL-1) possesses a large surface area combined with a relatively small pore diameter. Capillary action causes condensation of the water vapor in the pores. Similarly, products having a lesser surface area but a larger pore volume and pore diameter exhibit reduced capillary action (Aerogel A and Xerogel B—Syloid 244 and Syloid 74).

To summarize, small pore diameter results in: smaller total pore volume, greater surface area, and more selective adsorption. Large pore diameter results in: greater total pore volume, smaller surface area, and reduced condensation of water in pores.

### Moisture Adsorbance

In the coatings industry, the product referred to in the Figures as Syloid AL-1 is widely used to remove even

Table 1—Comparison of Properties Of Two Types of Xerogel and Standard Aerogel

	Desiccant Type Xerogel Syloid AL-1	Flattig Type Xerogel Syloid 74	Standard Type Aerogel Syloid 244
Surface area, M <sup>2</sup> /g.....	675	340	310
Pore volume, cc/g.....	0.40	1.1	1.4
Average pore diameter (A).....	20	150	200
Oil adsorption, lbs/100 lbs.....	60	200	300
pH (5% slurry).....	4	7	7

trace amounts of moisture from paints containing metallic pigments such as aluminum. Without the presence of this moisture adsorber, the metallic pigment would react with the moisture to liberate hydrogen, causing a dangerous buildup of pressure in the container. This would cause the paint to seep out of the can and eventually pop the lid with potentially disastrous results (Figure 4).

**Flattening Efficiency**

With regard to the influence of pore volume and surface area, let us see how these parameters combined with particle size distribution interact to produce highly efficient flattening agents based on silica gel for industrial and consumer based paints, printing inks, and plastics.

The ideal flattening agent should combine the following properties: high pore volume; narrow pore size distribution; appropriate particle size for the application and film thickness of the coating; narrow particle size distribution; and particle integrity—maintain both pore volume and particle size distribution during processing.

Silica gel based flattening agents encompass all of these properties and are the most highly efficient flattening agents currently employed by the coatings industry.

Since flattening or gloss reduction is a result of micron-sized particles scattering rather than reflecting light and pore volume is inversely proportional to density, products with higher pore volume and lower density can provide greater flattening efficiency since they contain a larger number of particles per unit weight.

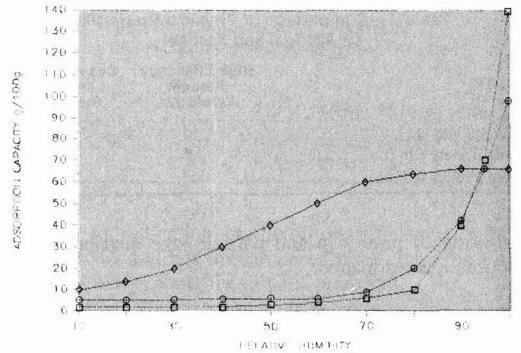
Improved aerogels are now available for trade sales varnishes, coil coatings, vinyl topcoats, and general industrial finishes based on high pore volume, lower density products.

Representative physical properties of the higher efficiency, lower density aerogel as compared to a conventional xerogel are listed in Table 3.

Examples of how these products perform as flattening agents can be seen in Figures 5-7 which clearly demonstrate the superior performance of this type of silica gel in major industrial applications such as a thermosetting acrylic coil coating enamel (Figure 5), nitrocellulose wood lacquer (Figure 6), and a vinyl topcoat lacquer (Figure 7).

In addition to improved flattening efficiency, the use of these products also results in:

- (1) Films with improved physical properties such as flexibility and impact resistance because of lower pigment loading as compared to other types of flattening agents;



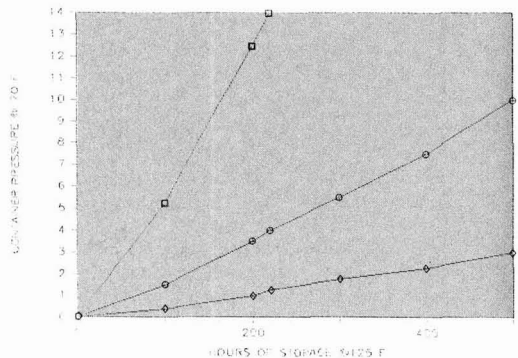
**Figure 3—Moisture adsorption capacity of silica gel at 25°C. Legend: □ — Syloid 244; ⊕ — Syloid 74; ◇ — Syloid AL-1**

- (2) Improved suspension properties; and
- (3) No change in rheological properties.

**Particle Size**

Particle size is another important criteria in designing and selecting particular silica gels. Generally speaking, in thin films such as vinyl top coats and paper coatings, products with an average particle size of 2-4µm are required. For films applied at a normal thickness of 0.8 to 1.2 mils an average particle size of 4 to 6µm is usually acceptable. Generally speaking, the particle size should be 20 to 30% of the total film thickness. Particle size distribution is equally as important and must be carefully controlled. An excess of fines can cause agglomeration of the particles and may cause film defects. It can also cause a turbidity in clear films or a lack of jetness in opaque colors. On the other hand, even a small amount of over-size particles can disturb the smooth texture of the coating and contribute to roughness.

Although pore size and particle size distribution are key factors influencing flattening efficiency, overall efficiency can be seriously decreased if these pigment parameters are altered during the pigment dispersion process or during the actual coatings application method. As pointed out previously, silica gel process variations allow the



**Figure 4—Pressure increase in aluminum paints containing 0.3% moisture (capacity at 25°C). Legend: □ — Control; ⊕ — 0.4% Syloid AL-1; ◇ — 1% Syloid AL-1**

**Table 2—Silica Gel Morphology**

Surface area .....	200-700 M <sup>2</sup> /g
Pore volume .....	0.4 to 2.5 cc/g
Median pore diameter.....	20 to 300 Å
Particle size .....	2 to 25 microns
Water content .....	4-7%
Impurities .....	<0.5%
Oil adsorption .....	50-300 lbs/100 lbs
Bulk density .....	5-30 lbs/cu ft
Surface treatments .....	Wax, fluoride

**Table 3—Comparison of Physical Properties Of Aerogel and Xerogel**

	High Efficiency Aerogel Syloid 221	Conventional Xerogel Syloid 74
Surface area (M <sup>2</sup> /g) .....	250	340
Bulk density (lb/ft <sup>3</sup> ) .....	7	16
Pore volume (cc/g) .....	1.7	1.1

tailoring of pore size and particle size distributions to optimize performance.

**Other Advantages**

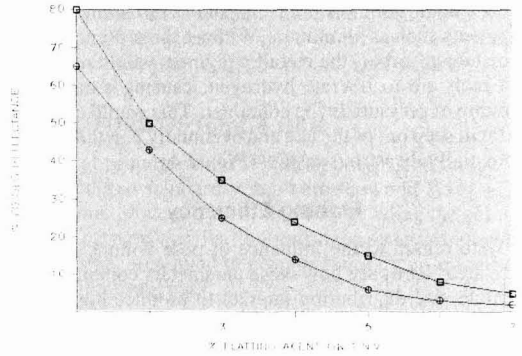
**STABILITY:** In addition, the unique silica gel process results in a stable particle which will maintain its size and porosity during the dispersion and application process to produce the desired level of gloss in spite of significant variations in shear rates. This is particularly important when production batches are subjected to extra dispersion processing during manufacturing as is frequently required during color matching or in high shear application methods such as high speed coil coatings.

**REFRACTIVE INDEX:** Another important advantage of silica gel is its refractive index, which is 1.46. This value is similar to the refractive index of most resins, therefore, silica gel does not detract from the clarity of clear coatings or plastic films as would be the use of most other extenders.

**INERTNESS:** Its chemical inertness is also of great importance as it has outstanding resistance to chalking and leaching in exterior applications. It is the flattening agent of choice in warranted architectural coil coatings where gloss and chalking rates are warranted for as long as 20 years.

**Thixotropy**

Recently, silica gel thixotropes have been developed based on a novel manufacturing process. In contrast to conventional silica gel processing which produces a stable product with regard to particle integrity, this new process is aimed at developing particles which are ex-



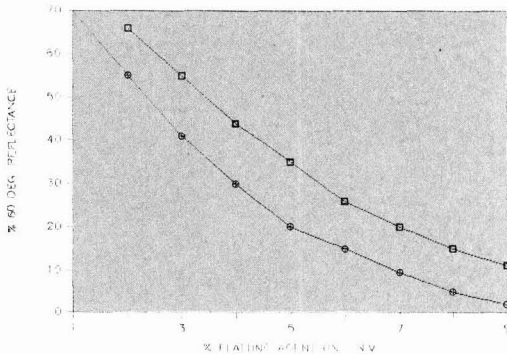
**Figure 6—Flattening of high efficiency aerogel (Syloid 169) in clear nitrocellulose lacquer for wood. Legend: □ — normal aerogel; ⊕ — Syloid 169**

tremely friable. This is accomplished by producing particles with significantly higher pore volumes. These high pore volume particles break down to submicron particles during the dispersion process and are effective thixotropes. These products have been found to be cost effective as compared to precipitated and fumed silicas.

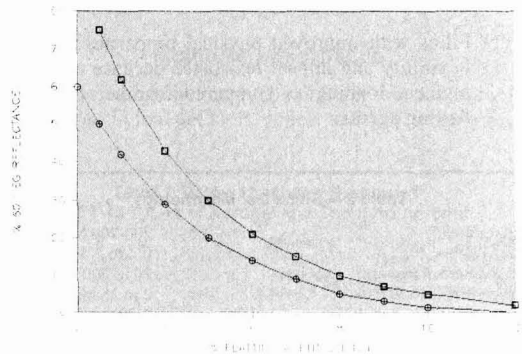
The mechanism by which silica gel functions as a thixotropy is quite complicated but is related to its surface chemistry (silanol group concentration) as well as to its physical state, which includes the following parameters:

- (1) Size of ultimate particle;
- (2) Degree of dispersion of particles;
- (3) Size of aggregates;
- (4) Relative wetting of particles to each other and to the liquid interface; and
- (5) Surface chemistry of particle (hydrophilic or hydrophobic nature).

Thickening and thixotropy are produced by small aggregates linked together through non-wetted areas into an extensive three-dimensional gel network throughout the liquid medium. The non-wetted areas are formed by fracture when highly porous aggregates are milled and broken apart into smaller aggregates. Maximum structure and thixotropy occur when these three-dimensional networks



**Figure 5—Flattening of high efficiency aerogel (Syloid 221) in thermosetting acrylic coil coating enamel. Legend: □ — normal xerogel; ⊕ — Syloid 221**



**Figure 7—Flattening of high efficiency aerogel (Syloid 234) in vinyl topcoat lacquer. Legend: □ — normal aerogel; ⊕ — Syloid 234**



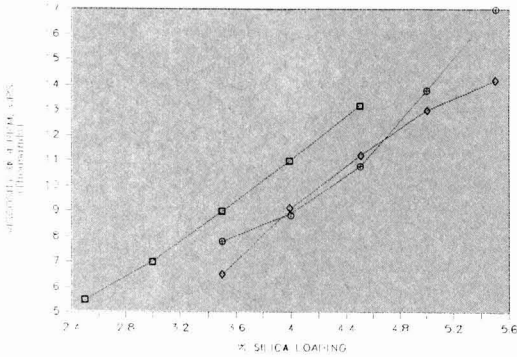


Figure 8—Effect of thixotropes on polyester gel coat viscosity. Legend: □ — Sylox TX; ⊕ — PPT silica "A"; ◇ — PPT silica "B"

link together into larger networks through hydrogen bonding. Linkages involving hydrogen bonding are easily broken by shearing forces but quickly reestablish when polymer thickener systems are at rest.

Silica gel thixotropes such as Sylox TX are finding use in automotive body patching compounds, styrene polyester gel coats, epoxy coal tar coatings, and a variety of caulking and adhesive systems. A specific example of how Sylox TX performs as a thixotrope as compared to a precipitated silica in a styrene polyester gel coat is shown in Figure 8.

A further comparison of Sylox TX to a precipitated silica at different shear rates in a styrene polyester gel coat at equal loadings of 3% thixotrope clearly demonstrated the improved efficiency of Sylox TX. The viscosity was measured in centipoises with a Brookfield RVF viscometer, a number 4 spindle (Table 4). The thixotropic

Table 4—Comparison of Thixotropy Of Precipitated Silica and Sylox TX

	2 RPM	4 RPM	20 RPM	Thixotropic Index
Precipitated silica . . . . .	12,700	9,000	4800	2.65
Sylox TX . . . . .	19,700	13,250	6180	3.19

index is an indication of thixotrope efficiency and is obtainable by dividing the low speed viscosity by the high speed viscosity. A higher value is preferred.

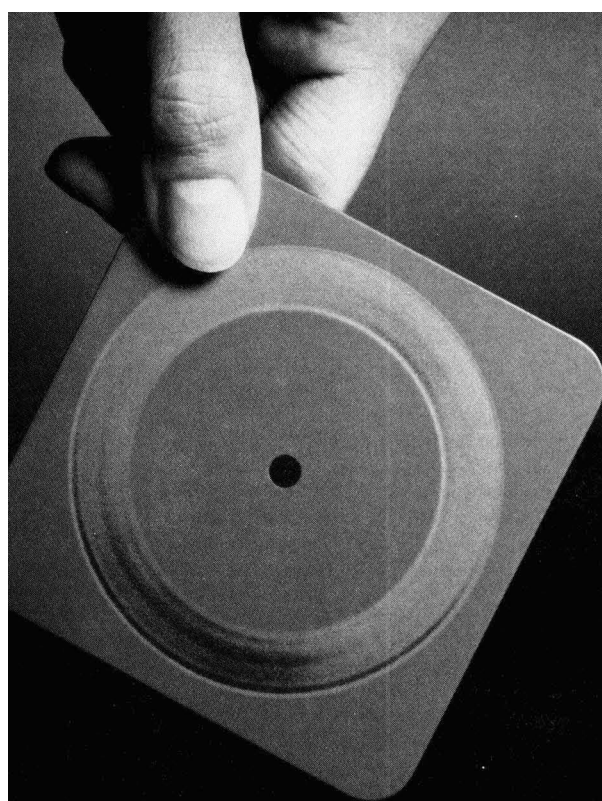
In addition to outstanding thixotropic properties, Sylox TX disperses easily, does not settle, and has excellent storage stability. This is especially important in consumer based products where long shelf life is essential.

SUMMARY

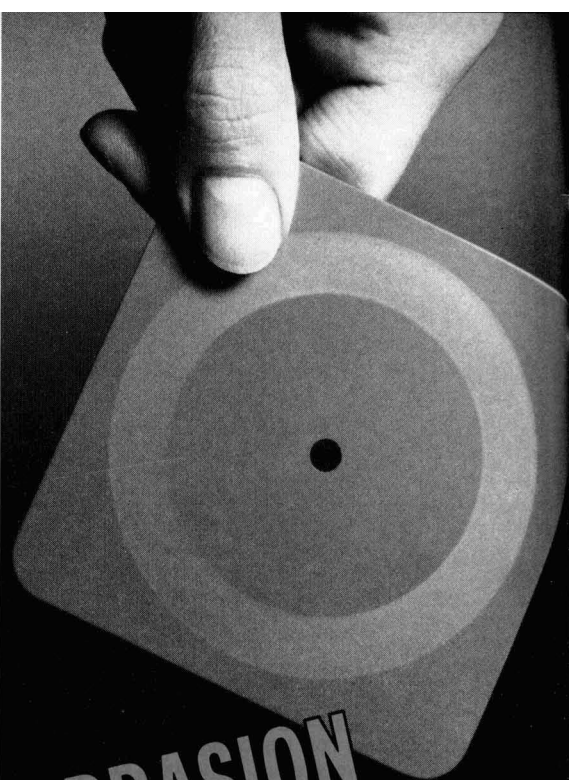
It has been shown that synthetic amorphous silica gel possesses a complex and variable morphology which can be controlled or modified by a sophisticated manufacturing process. An understanding of how specific aspects of this morphology such as pore diameter, pore volume, surface area, density, and particle size distribution affect silica gel performance is essential to both the manufacturer of this product and the ultimate customer if improved products are the result.

References

- (1) Iler, R. K., "The Chemistry of Silica," John Wiley & Sons, New York, NY, 1979, Chap. 5, pp 462-599.
- (2) Tischer, H., "Silica, Synthetic Aerogels and Hydrogels," in "Pigment Handbook, Volume 1," Edited by Temple C. Patton, John Wiley & Sons, New York, NY, 1973, pp 189-199.



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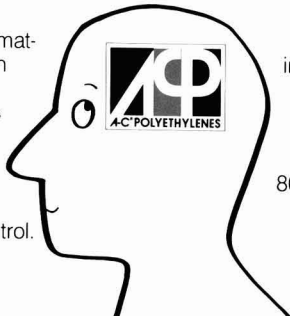
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# Addendum to An Infrared Spectroscopy Atlas For the Coatings Industry

Scandinavian Paint and Printing Ink  
Research Institute\*

"An Infrared Spectroscopy Atlas for the Coatings Industry" by the Chicago Society is used extensively and is greatly appreciated even by the Scandinavian coatings laboratories. These laboratories have combined their individual corrections and additions in the present publication. The information originates from raw material suppliers and reference spectra, mainly those from Hummell

Scholl's collections and those recorded in the individual laboratories.

Main participants were analytical chemists from:

Køge Chemical Work Ltd.  
Sadolin & Holmblad Ltd.  
Scandinavian Paint and Printing Ink Research Institute

POLYMERS	Spectrum No.	Sample Identification	
Acrylics	4	Acryloid AT-50	Contains melamine
	5	Acryloid AT-70	EA/S/maleic acid 2:1:0,4 + N-containing compound (imidazoline?)
	7	Acryloid B 66	BMA/MMA 73:27
	10	Butyl acrylate homopolymer	1090 cm <sup>-1</sup> not from butyl acrylate homopolymer
	20	ICI-2018	Contains styrene
	21	Ionac-JLW-A1	Contains styrene
	43	Neocryl B-700	Isobutyl methacrylate homopolymer
	44	Neocryl B-705	IBMA/VT + something else (1100-1250 cm <sup>-1</sup> )
	45	Neocryl B-723	BMA/MMA
	46	Neocryl B-725	BMA/MMA
	47	Neocryl B-726	BMA/MMA
	48	Neocryl B-728	MMA-homopolymer
	49	Neocryl B-734	BMA/MMA
	51	Pliolite ACL	Styrene/acrylate copolymer
	†53	Modifier 0245 (not acrylic, not Resimene RF 0245-OE)	54% tall oil ester of RJ 101 (= styrene allyl alcohol)

\*The Infrared Spectroscopy Atlas for the Coatings Industry was prepared by the Infrared Spectroscopy Committee of the Chicago Society for Coatings Technology. Published by the Federation of Societies for Coatings Technology, 1980.

\*Analytical Contact Group, Agern Alle 3, 2970 Horsholm, Denmark.

†Erratum

\*\*Reprints are available from the Federation office, 1315 Walnut St., Philadelphia, PA 19107.



	<b>Spectrum No.</b>	<b>Sample Identification</b>	
<b>POLYMERS</b>			
<b>Alkyds</b>			
	59	Adipic acid modified o-phthalic alkyd	Contains benzoic acid
	71	Gelkyd 331 w	Contains phthalic anhydride
	76	Macopol 3017	Styrenated isophthalic alkyd
	77	Macopol 3077	Vinyl-toluenated isophthalic alkyd
	86	Methyl methacrylate modified o-phthalic alkyd	Contains styrene
	91	P-T-Butyl benzoic acid modified o-phthalic alkyd	No visible PTBB. Contains benzoic acid
<b>Melamine Formaldehydes</b>			
	183	Cymel 248-8	Butylated melamine formaldehyde resin
	184	R-1129	Butylated melamine formaldehyde resin
	185	Resimene 731	Methylated melamine formaldehyde resin
	186	Resimene 740	Methylated melamine formaldehyde resin
	187	Resimene 745	Methylated melamine formaldehyde resin
	188	Resimene 750	Butylated melamine formaldehyde resin
	189	Resimene 1720	Methylated melamine formaldehyde resin
	*190	Uformite MM-55 (not urea formaldehyde)	Butylated melamine formaldehyde resin
<b>Urea Formaldehydes</b>			
	193	Resimene U 920	Butylated urea formaldehyde resin
	194	Resimene U 964	Etherified urea formaldehyde resin (probably mixed alcohols)
	195	Resimene U 975	Methylated urea formaldehyde resin
<b>Benzoguanamine Formaldehydes</b>			
	197	Cymel 1123	Etherified benzoguanamine formaldehyde resin
	198	QR-336	Butylated benzoguanamine formaldehyde resin
	199	RF-7358	Butylated benzoguanamine formaldehyde resin
	200	RF-7359	Butylated benzoguanamine formaldehyde resin
	201	Uformite MX-61	Butylated benzoguanamine formaldehyde resin
<b>Natural Resins</b>			
	242	Unirez 1014 resinat	Zinc resinat
<b>Polyesters</b>			
	276	Aquamac 1100	OPA-polyester
	277	Aquamac 1300	Isophthalic-polyester
	278	Atlac 382 E	Based on bisphenol A
	279	Chemacoil TA-100	Contains fatty acid esters and amide
	280	Chemacoil TA-101	Contains fatty acid esters and amide
	281	Desmophen 650 A	Contains OPA
	282	Desmophen 651 A	Contains OPA
	284	Multron R-12A	Contains adipic acid
	285	Multron R 221	Contains OPA
	286	Oxyester Z-1439	Contains isophthalic acid
	287	Paraplex RGA-2	Oil modified sebacic alkyd
	288	PC-600	Contains terephthalic acid
	289	PE-310	Contains isophthalic acid
	290	PE-320	Contains isophthalic acid
	303	Resin HS-450	Contains isophthalic acid
	304	Resin HS-550	Contains isophthalic acid
	306	Vitel PE 207	Contains terephthalic acid
	307	WS-300	Contains isophthalic acid
	308	WS-549	Contains isophthalic acid
<b>Vinyls</b>			
	369	Airflex A-500	Vinylacetate/ethylene copolymer
	370	Airflex A-728	Vinylacetate/ethylene copolymer
	371	Amoco 18-290	Poly- $\alpha$ -methylstyrene

\*Erratum

	Spectrum No.	Sample Identification	
<b>POLYMERS</b>			
	376	Butuar B-79	Polyvinylbutyral
	396	Flexbond 811	Vinylacetate/dibutyl maleate copolymer
	403	Gelvatol 3000	Polyvinylalcohol (not completely hydrolyzed)
	404	Gelvatol 6000	Polyvinylalcohol (not completely hydrolyzed)
	405	Gelvatol 9000	Polyvinylalcohol (not completely hydrolyzed)
	407	Hycar ATBN	Butadiene/acrylonitrile copolymer with secondary amine end groups. 16.5% AN
	408	Hycar CTBN	Butadiene/acrylonitrile copolymer with carboxyl end groups. 10% AN
	409	Kolima 35	Contains polyvinylacetate
	410	Piccolastic D-150	Polystyrene possibly with a small amount of butadiene
	411	Piccotex 100	$\alpha$ -methyl styrene/vinyltoluene copolymer
	421	RJ-101	Styrene allyl alcohol copolymer
	*422	Saran F-120	Contains a ketone (solvent)
	423	Saran resin F-310	Contains vinylidene chloride
	*431	Union Carbide VAGH	Contains a ketone (solvent)
	444	Wave 345	Mainly PVA
	445	Wave 375	Mainly PVA
<b>Miscellaneous</b>			
	447	Contact cement	Polychloroprene + other components (p-tert.-butyl phenol resin?)
	*455	Parlon P-10	Contains toluene
	*456	Parlon S-10	Contains toluene
	467	Rubber cement	Cis-polyisoprene
	469	Surlyn D	Poly (ethylene-co-acrylic or methacrylic acid) partly on salt form
	470	Unichlor 40-150	Chlorinated aliphatic hydrocarbon, probably polyethylene
<b>MONOMERS</b>			
<b>Acids &amp; Anhydrides</b>			
	*508	Stearic acid, 12-hydroxy-	Mainly dimethyl sulfoxide
<b>Acrylics</b>			
	*526	Blemmer "G"	Not glycidyl methacrylate (Compare Sp 537) Mainly some alcohols
<b>Isocyanates</b>			
	555	Desmodur N-75	Aliphatic isocyanate, probably based on HDI
	556	Desmodur VP	Probably based on IPDI
<b>Natural Oils</b>			
	*577	Dehydrated castor oil	Not dehydrated castor oil. Blown castor oil?
	598	Spencer Kellogg 1066	Absorptions at 1060 $\text{cm}^{-1}$ and 870 $\text{cm}^{-1}$ must originate from a modification
<b>SOLVENTS</b>			
<b>Ether Alcohols</b>			
	*704	Carbitol	2-ethoxy ethanol (Not 2(2-ethoxy ethoxy ethanol))
<b>INORGANIC PIGMENTS</b>			
<b>Whites &amp; Extenders</b>			
	802	Goelhite	$\alpha$ -FeOOH, yellow iron oxide (not white or extender)
	809	Hydrocarb	$\text{CaCO}_3$ + organic component
	810	Hydrocarb L	$\text{CaCO}_3$ + organic component
	813	Minex-7	Aluminium silicates

\*Erratum

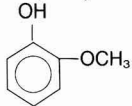
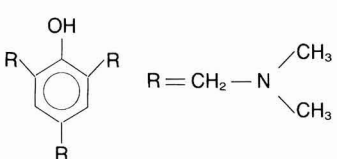
Spectrum No.	Sample Identification	
<b>INORGANIC PIGMENTS</b>		
815	Omya BLR-3	CaCO <sub>3</sub> + organic component
816	Omyalite 90	CaCO <sub>3</sub> + organic component
817	Omyalite 95-T	CaCO <sub>3</sub> + organic component
818	Peerless coating clay	Aluminium silicates
<b>Yellows &amp; Oranges</b>		
*908	Chrome orange (medium)	Wrong name and composition. Lead silicochromate e.g. Onchor M 50
914	Mapico yellow 300	Yellow iron oxide
920	Primrose yellow	PbCrO <sub>4</sub> • PbSO <sub>4</sub>
<b>Blue (Violet)</b>		
946	Carbazole violet blue shade	C.I. pigment violet 23 type
947	Carbazole violet red shade	C.I. pigment violet 23 type
948	Graphol blue	C.I. pigment blue 15 $\alpha$
949	Hostaperm blue 3RF	C.I. pigment blue 60
950	Hostaperm blue A3R	C.I. pigment blue 15 $\alpha$ coated
951	Hostaperm blue AFN	C.I. pigment blue 15 $\alpha$ (15:2)
952	Hostaperm blue AR	C.I. pigment blue 15 $\alpha$ (15:1)
953	Hostaperm blue B2G-A	C.I. pigment blue 15 $\alpha$ (15:3)
954	Hostaperm blue B2GAJ	C.I. pigment blue 15 $\alpha$ (15:3)
955	Hostaperm blue BFN-A	C.I. pigment blue 15 $\alpha$
956	Hostaperm violet RL	C.I. pigment violet 23
957	Hostaperm violet RL EX strong	C.I. pigment violet 23
958	Indofast blue	C.I. pigment blue 22
959	Indofast violet	C.I. pigment violet 23
960	Perrindeau violet	C.I. pigment violet 29 type (perylene pigment)
962	Phthalo blue G-NCNF	C.I. pigment blue 15 $\alpha$ coated
963	Phthalo blue lake R-NCNF	C.I. pigment blue 15 $\alpha$ (15:2) + monochlorinated—coated
964	Phthalo blue R-NC	C.I. pigment blue 15 $\alpha$ + monochlorinated
965	Phthalo blue R-NCNF	C.I. pigment blue 15 $\alpha$ + monochlorinated— coated
966	Quindo magenta	C.I. pigment red 122
967	Quindo violet	C.I. pigment violet 19-coated
968	Sandorin blue BNK	C.I. pigment blue 15 $\alpha$ (15:1)
969	Sandorin blue RL	C.I. pigment blue 60
970	Sandorin violet blue pigment	C.I. pigment violet 23 type
971	Sunfast blue NC NF	C.I. pigment blue 15 $\alpha$
972	Sunfast violet	C.I. pigment violet 23
<b>ORGANIC PIGMENTS</b>		
<b>Brown</b>		
973	Ginger brown	C.I. pigment brown 5
974	Hostaperm brown HFL	C.I. pigment brown 32
975	Hostaperm brown HFR	C.I. pigment brown 25
<b>Green</b>		
976	Hostaperm green 6G	C.I. pigment green 36-coated
977	Hostaperm green 8G	C.I. pigment green 36
978	Hostaperm green 8G-A	C.I. pigment green 36-coated
979	Hostaperm green BG-A	C.I. pigment green 7 (+ pigment blue 15 $\beta$ ?)
980	Hostaperm green GG	C.I. pigment green 7 type
981	Phthalo green blue shade	C.I. pigment green 7-coated
982	Phthalo green G-5018	C.I. pigment green 7 type
983	Phthalo green G-5400	C.I. pigment green 36 type
984	Phthalo green med blue shade	C.I. pigment green 7 type
985	Phthalo green resinated	C.I. pigment green 7-coated
986	Phthalo green yellow shade	C.I. pigment green 36 a.o.
987	Sandorin green 3GLS	C.I. pigment green 7 type

\*Erratum

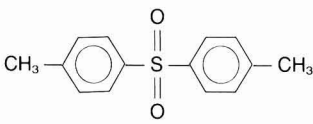
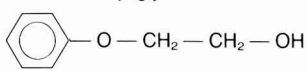
Spectrum No.	Sample Identification	
<b>ORGANIC PIGMENTS</b>		
988	Sunfast green 264-8137	C.I. pigment green 36 type
989	Sunfast green 264-8142	C.I. pigment green 7 type
<b>Red (Maroon)</b>		
990	Alizarine maroon	C.I. pigment violet 6
991	Barium lithol med.	C.I. pigment red 49/Ba
992	Big macatawa red medium dark	C.I. pigment red 52/?
993	Bon maroon	C.I. pigment red 58/Mn
994	Bright red	C.I. pigment red 4
995	Calcium lithol dark	C.I. pigment red 49/Ca
996	Canyon red	C.I. pigment red 48/?
997	Electra red	C.I. pigment red 37
998	Flash red	C.I. pigment red 2
999	Graphtol red	C.I. pigment red 149
1000	Hansa red 3B	C.I. pigment red 3
1001	Hansa red B	C.I. pigment red 3
1002	Hansa scarlet RNC	C.I. pigment red 3
1003	Hostaperm pink E	C.I. pigment red 122
1004	Hostaperm red E3B	C.I. pigment violet 19 $\beta$
1005	Hostaperm red E5B	C.I. pigment violet 19 $\beta$
1006	Hostaperm red EG	C.I. pigment red 209
1007	Hostaperm red violet ER-02	C.I. pigment violet 19
1008	Indofast brilliant scarlet R-6335	C.I. pigment red 123
1009	Indofast brilliant scarlet R-6500	C.I. pigment red 190
1010	Indofast double scarlet	—
1011	Lake red toner LCLL-02	C.I. pigment red 53:1/Ba
1012	Lithol rubine lt.	C.I. pigment red 57/Ca
1013	Molora red	C.I. pigment red 52/Ca
1014	Montclair red 235-0006	C.I. pigment red 17
1015	Montclair red 235-7700	C.I. pigment red 17
1016	Para red	C.I. pigment red 1
1017	Permanent bordeaux FGR	C.I. pigment red 14
1018	Permanent bordeaux HF 3R	C.I. pigment violet 32
1019	Permanent carmine FB-01	C.I. pigment red 5
1020	Permanent carmine FBB	C.I. pigment red 146
1021	Permanent carmine FBB-3D	C.I. pigment red 146
1022	Permanent carmine HF 3C	C.I. pigment red 176
1023	Permanent carmine HF 4C	C.I. pigment red 185
1024	Permanent maroon HFM	C.I. pigment red 171
1025	Permanent red 2BM	C.I. pigment red 48/Mn
1026	Permanent red BL	C.I. pigment red 149
1027	Permanent red F 3RK-70	C.I. pigment red 170
1028	Permanent red F 4RH	C.I. pigment red 7
1029	Permanent red F 5RK	C.I. pigment red 170
1030	Permanent red FGR	C.I. pigment red 112
1031	Permanent red FRLl	C.I. pigment red 9
1032	Permanent red GG	C.I. pigment orange 5
1033	Permanent red HF 2B	C.I. pigment red 208
1034	Permanent red HF 3S	C.I. pigment red 188
1035	Permanent red HF 4B	C.I. pigment red 187
1036	Permanent red HFG	C.I. pigment orange 38
1037	Permanent red HFT	C.I. pigment red 175
1038	Permanent red TG-01	C.I. pigment red 194
1039	Permanent red violet MR	C.I. pigment red 88
1040	Permanent scarlet O-A	—
1041	Permanent vat red RRN	C.I. vat red 15
1042	Perrindo maroon deep	C.I. pigment red 179
1043	Perrindo red	—
1044	Perylene red dark	C.I. vat red 29
1045	Pyrazolone red	C.I. pigment red 38
1046	Quindo red	C.I. pigment violet 19 type
1047	Ranger red 2B lt.	C.I. pigment red 48/Ba
1048	Sandorin brilliant red 5BL	C.I. pigment red 192
1049	Thiofast red	C.I. pigment red 88



<b>Spectrum No.</b>	<b>Sample Identification</b>	
<b>ORGANIC PIGMENTS</b>		
1050	Toluidine red lt.	C.I. pigment red 3
1051	Toluidine red	C.I. pigment red 3
1052	Toluidine red (light)	C.I. pigment red 3
1053	Toluidine red dark	C.I. pigment red 3
1054	Toluidine red ex. lt.	C.I. pigment red 3
1055	Toluidine red med	C.I. pigment red 3
<b>Yellow &amp; Orange</b>		
1056	Benzidine orange	C.I. pigment orange 13
1057	Benzidine yellow AOT	C.I. pigment yellow 14
1058	Benzidine yellow AAA	C.I. pigment yellow 12
1059	Crayon orange	C.I. pigment orange 15
1060	Diane orange	C.I. pigment orange 16
1061	Dianisidine orange	C.I. pigment orange 16
1062	Diarylide OA yellow	C.I. pigment yellow 17
1063	Diarylide A yellow	C.I. pigment yellow 12
1064	Diarylide orange	C.I. pigment orange 13
1065	Diarylide OT yellow	C.I. pigment yellow 14
1066	Diarylide yellow	C.I. pigment yellow 13
1067	Dinitraniline orange	C.I. pigment orange 5
1068	Dinitroaniline orange	C.I. pigment orange 5
1069	Fanchon orange	C.I. pigment orange 1
1070	Fanchon yellow 10G	C.I. pigment yellow 3
1071	Fanchon yellow G	C.I. pigment yellow 1
1072	Fanchon yellow RN	C.I. pigment yellow 65
1073	Fanchon yellow YH-5757	C.I. pigment yellow 74
1074	Fanchon yellow YH-5770	C.I. pigment yellow 73
1075	Golden yellow R	C.I. pigment yellow 83-coated
1076	Golden yellow RS	C.I. pigment yellow 83
1077	Graphitol yellow RCL	C.I. pigment yellow 83
1078	Hancock yellow 10-G	C.I. pigment yellow 3
1079	Hancock yellow 73	C.I. pigment yellow 73
1080	Hancock yellow G	C.I. pigment yellow 1
1081	Hansa 10G	C.I. pigment yellow 3
1082	Hansa brilliant yellow 10GX	C.I. pigment yellow 98
1083	Hansa brilliant yellow 56X	C.I. pigment yellow 74
1084	Hansa G	C.I. pigment yellow 1
1085	Hansa yellow 10G	C.I. pigment yellow 3
1086	Hansa yellow 2R-A	C.I. pigment yellow 65
1087	Hansa yellow G-02	C.I. pigment yellow 1
1088	Hansa yellow H-A	C.I. pigment yellow 74
1089	Hostaperm orange GR	C.I. pigment orange 43
1090	Hostaperm yellow H 4G	C.I. pigment yellow 151
1091	Indofast orange OV-5964	C.I. vat orange 3
1092	Indofast orange OV-5983	C.I. pigment orange 43
1093	Lemon metallic yellow	C.I. pigment yellow 17
1094	Luna LF yellow	C.I. pigment yellow 74
1095	Luna yellow	C.I. pigment yellow 74-coated
1096	Oriole yellow AAOA	C.I. pigment yellow 17
1097	Permanent orange G	C.I. pigment orange 13
1098	Permanent orange GRR	C.I. pigment orange 16
1099	Permanent orange HL	C.I. pigment orange 36
1100	Permanent orange HL-70	C.I. pigment orange 36
1101	Permanent orange RK-C	C.I. vat orange 3
1102	Permanent orange RL-01	C.I. pigment orange 34
1103	Permanent orange RL-3R	C.I. pigment orange 34
1104	Permanent yellow DGR	C.I. pigment yellow 126
1105	Permanent yellow DHG	C.I. pigment yellow 12
1106	Permanent yellow FGL	C.I. pigment yellow 97
1107	Permanent yellow G	C.I. pigment yellow 14
1108	Permanent yellow G3R-01	C.I. pigment yellow 114
1109	Permanent yellow GG	C.I. pigment yellow 17
1110	Permanent yellow GR	C.I. pigment yellow 13
1111	Permanent yellow H1 OG	C.I. pigment yellow 81

Spectrum No.	Sample Identification	
<b>ORGANIC PIGMENTS</b>		
1112	Permanent yellow H1 OGL	C.I. pigment yellow 113
1113	Permanent yellow H2G	C.I. pigment yellow 120
1114	Permanent yellow HR	C.I. pigment yellow 83-coated
1115	Permanent yellow HR-70	C.I. pigment yellow 83
1116	Permanent yellow NCG	C.I. pigment yellow 16
1117	Permanent yellow NCG-70	C.I. pigment yellow 16
1118	Permanent yellow R-A	—
1119	Permanent yellow YR	—
1120	Pyrazolone orange	C.I. pigment orange 13
1121	Sunbrite yellow	C.I. pigment yellow 74
1122	Velvetized yellow	C.I. pigment yellow 1
<b>MISCELLANEOUS</b>		
<b>Anti-skinning &amp; Anti-oxidants</b>		
1124	Anti-skinning agent	Methyl ethyl ketoxime
1127	Exkin 2	Methyl ethyl ketoxime
1128	Guaiacol	OH 
1130	Skinfoil KE	Methyl ethyl ketoxime
1134	Troykyd anti-skin odorless liquid	Contains ethyl glycol?
<b>Catalysts &amp; curing agents</b>		
1140	DMP 30	
1141	Epicure 8525	Polyaminoamide
1144	Irgacure 651	Contains probably water
<b>Defoamers</b>		
1161	Byk-0	Mainly cyclohexanone
1162	Byk-010	Mineral oil + different additives
1163	Byk-069	Mineral oil + special alcohols, metal soaps and emulsifiers
1164	Byk-073	Mineral oil + metal soaps and emulsifiers
1165	Byk-W	Mineral oil + metal soaps, esters and emulsifiers
1166	DC-230	Silicone
<b>Dispersants</b>		
1173	Anti-terra P	Phosphoric acid salt of polyaminoamide
1174	Anti-terra U	Salt of unsaturated polyaminoamides and high molecular acid esters
1176	Darvan 1	Mainly sulphonic acid or salt
1177	Darvan 7	Carboxylic acid salt
1178	Disperbyk	Alkylolamine salt of polymeric carboxylic acid
1180	Intersperse	Long oil alkyd
1181	Polywet ND-1	Carboxylic acid salt
1182	Polywet ND-2	Carboxylic acid salt
<b>Plasticizers</b>		
*1250	Flexol 380	Di (2-ethyl hexyl) isophthalate (not di(2-ethyl hexyl) phthalate)

\*Erratum

	<b>Spectrum No.</b>	<b>Sample Identification</b>	
<b>MISCELLANEOUS Preservatives</b>			
	1288	Amical-48 dispersion	
<b>Surfactants</b>			
	1346	Surfynol TG	2,4,7,9-tetramethyl-5-decyne-4,7-diol + alkyl phenyl ether of polyethylene glycol + ethylene glycol
	1353	Triton GR-5M	60% Dioctyl sodium sulfosuccinate aq + 20% 2-propanol
<b>Thickeners</b>			
	1376	Thixatrol ST	Amidized and hydrogenated castor oil
<b>Waxes</b>			
	1407	Polyem 20	Contains alkyl aryl sulfonate (compare 1327 and 1344)
<b>Miscellaneous Additives</b>			
	1412	Byk 301 (mar and slip agent)	Contains butyl glycol
	1416	Dalpad A (coalescing agent)	
	1419	Paint additive 3 (leveling and flow agent)	Propyl silicone
	1420	Paint additive 11 (mar and flow agent)	Silicone
	1421	Paint additive 14 (mar and slip agent)	Silicone
	1422	Paint additive 16 (hammer tone agent)	Methyl silicone
	1423	Paint additive 21 (adhesion promotor)	Silicone
	1424	Paint additive 56 (curtain coating agent)	Silicone
	1425	Paint additive 57 (mar and flow agent)	Silicone
	1426	Puffing agent	Modified vegetable oil
	1429	Troykyd 366 (anti-crater agent)	Alkyd + silicone or silicone modified alkyd

These subcommittee reports are for the use of the membership of ASTM Committee D-1 in continuing its work and for the interest of readers in the activities of ASTM Committee D-1. The reports are not official actions of ASTM and may not be quoted as representing any action or policy of the American Society for Testing and Materials.

# January 1986 Subcommittee Reports Of ASTM Committee D-1

The January meeting of ASTM Committee D-1 on Paint and Related Coatings and Materials was held on January 12-15, 1986 at the Sheraton Hotel in New Orleans, LA. In the three and one-half days preceding the final session and general meeting of Committee D-1, 147 members and guests met in 135 scheduled meetings of D-1 subcommittees and working task groups. The present membership of Committee D-1 is 498.

*Special Highlight* of the meeting was a session of three talks on biodeterioration chaired by D.L. Campbell, chairman of Subcommittee D01.28. Presentations included: "The Enzyme Degradation of Cellulosic Latex Paint Thickeners" by D.P. Leipold, "Wood Preservation" by W.B. Woods, and "Microbiological Corrosion" by R.T. Ross.

*Certificate of Appreciation* was presented to Harold D. Marshall.

*Appointments* were made as follows: Sub. D01.07 on Government Contacts—R.J. Martell to replace A.A. Chasan as Chairman; Sub. D01.51 on Powder Coatings—D. Wilverding to replace C. Grenko as Chairman and H.S. Fairman as Vice-Chairman; Sub. D01.94 on Awards and Memorials—S.B. Levinson to replace H.D. Hammond III as Chairman.

## Highlights

The following items of major interest to the coatings industry warrant special emphasis:

*D 3980 Interlaboratory Testing Practice, Software Program*—Sub. D01.20 reports on a basic computer program to analyze results.

*Transfer Efficiency*—Sub. D01.21.13 reports details on development of TE test method.

*Static Friction of Coatings*—Sub. D01.23.15 working on this problem for rough coatings.

*Zahn Cup Precision*—Sub. D01.24.19 running a round-robin to collect data on this instrument.

*Purchasing of Paints*—Sub. D01.41 proposed a simplified source revision.

*Evaluating Coating Repair Materials*—Sub. D01.46.03 will start work on this in the future through test patch applications.

*Viscosity of Liquid Inks*—Sub. D01.24 and Sub. D01.56 working together on this problem. Joint task group to meet.

## Future Meetings

June 15-18, 1986—Louisville, KY (Galt Hotel).  
January 25-28, 1987—Tampa, FL (Hyatt Regency).  
June 21-24, 1987—Dearborn, MI (Hyatt Regency).  
January 24-29, 1988—Tampa, FL (Hyatt Regency).

## New Standards

New D-1 standards since the July 1985 meetings of D-1 approved by the ASTM Committee on Standards in the months shown: (subcommittee jurisdiction)

### SEPTEMBER 1985

D4518-85 "Methods for Measurement of Static Friction of Coating Surfaces" (Sub. D0.23)

### OCTOBER 1985

D4457-85 "Test Method for Analysis of Dichloromethane and 1,1,1, Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph" (Sub. D0.21)

D4462-85 "Specification for Zinc Hydroxy Phosphite Pigment" (Sub. D0.31)

### NOVEMBER 1985

D4540-85 "Guide for Testing Interior Latex Semigloss and Gloss Paints" (Sub. D0.42)

D4541-85 "Method for Pull-off Strength of Coatings Using Portable Adhesion-Testers" (Sub. D0.46)

### JANUARY 1986

D4563-86 "Test Methods for the Determination by Atomic Absorption Spectroscopy of Titanium Dioxide Content of Pigments Recovered from Whole Paint" (Sub. D0.21)



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## DIVISION 1 ADMINISTRATIVE

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### SUBCOMMITTEE D01.07 GOVERNMENT CONTACTS

#### R.J. Martell, Chairman

The present status of Federal Standard 141 was reviewed based on the chairman's report of an interview at the Fort Belvoir R & D Center, the agency responsible for coordination. Plans were discussed for a comparison of tests in the 'C' version, scheduled for publication in 1986, with comparable ASTM tests, with the aim of avoiding duplication where possible.

The multiplicity of test methods in government specifications was brought up as an industry problem, particularly for the small producer who does not have all types of test equipment available. This problem surfaced when GSA started to enforce the requirements for full testing of every batch. J.C. Weaver pointed out some of the difficulties encountered over the years in obtaining agreement on such basic considerations as constant temperature and humidity. Developing a consensus on a battery of tests to be used across the board for testing of government specification paints could prove to be a difficult task.

The chairman reviewed the DOD/SAE/ASTM conference held in December, 1985 in Williamsburg, VA, which covered the various aspects of the DOD AD-Cop program. This program is an extension of the earlier Commercial Acquisition Program since it involves adoption of voluntary standards in place of government standards, wherever appropriate.

Recent organizational and personnel changes in various agencies were discussed. The most sweeping change is the assignment of all Federal Sales Class 80 products to Auburn, WA. Adhesives and sealants have previously been handled by Kansas City.

### SUBCOMMITTEE D01.08 ENVIRONMENTAL CONCERNS

#### J.C. Weaver, Chairman pro tem

The approved scope for Sub. D01.18 is: "To assess the need for, recommend the initiation of, and coordinate the timely development and effective utilization of test methods relating to environmental matters including current and potential

federal and state regulations to insure that methods of compliance and the impact on the community are understood. Compatibility with ASTM will be effected through membership in the Environmental Coordinating Committee."

Transfer efficiency measurement in factory painting, e.g., of automobile bodies, etc., is now recommended by E.A. Prashan of General Motors as ready for method standardization in D-1 as another of the list of methods needed for state compliance with EPA regulation of volatile organic content (VOC). Three years of study by EPA via its contractor CENTEC in eight paint laboratories in the Detroit area are described hereafter in the Sub. D01.21 report. Sub. D01.08 recommends that Sub. D01.55 begin a draft of a standard test method for Transfer Efficiency based on data from those laboratory tests.

Standards Technology Training (STT) two-day short courses on diverse subjects across ASTM's 140 technical committees were initiated in 1985 under ASTM staff management by Kenneth C. Pearson. STT's are expanding rapidly, e.g., a list of eight in the January issue of Standardization News. Sub. D01.08 recommends that D-1 design and operate an STT short course on VOC Compliance Proficiency, using relevant ASTM standards and supporting documents as classroom texts. Attendees may be analysts, formulators and applications engineers from state regulatory agencies, paint producers, and paint users. Much preparation is needed toward an Autumn 1986 target for a first session of this STT.

Special Technical Publications (STP's) by ASTM as supplements to its primary consensus standards enjoy wide sales. STP 500, D-1's Paint Testing Manual, sold out a few years after its 1972 issue, a tribute to its dedicated editor, G.G. Sward. Reissue in toto is an awesome challenge, while segments may be completed sooner. A D-1 STP on VOC Compliance Standards and Proficiencies is recommended for both direct sale and use as an STT textbook. Contents would comprise (1) the D 3980 VOC Practice and its many satellite standards, (2) U.S. EPA Method 24 on VOC and extracts from relevant EPA and CARB documents and (3) exposition of proficiency and other variables of compliance procedures.

### SUBCOMMITTEE D01.13 CONSUMER AFFAIRS

#### R.S. Shane, Chairman

The chairman noted the offer of Committee F-15 to provide consumer input to any ASTM technical committee desiring such help. At his suggestion, Sub.

D01.13 agreed to recommend to Sub. D01.90 that it bring this F-15 offer to the attention of all D-1 subcommittees and task groups to be used as seems appropriate.

In a discussion on labeling practices, the chairman reported that he had made presentations to Sub. D01.41 and the Delaware Valley Consumer Sounding Board (sponsored by F-15). He stated that from a consumer point of view, the consumer needed chiefly the following information:

(1) What kind of a surface the paint should be applied to and what the expected results are (type of finish, estimate of coverage).

(2) How the surface should be prepared for painting.

(3) How the coating should be applied, including any information on the preparation of the coating (e.g., dilution practices).

(4) How the coating should be maintained.

It was pointed out that the large manufacturers prepare and monitor their labels carefully. Perhaps a guide for consumer-oriented paint labels should be developed by Sub. D01.13 or Sub. D01.41. Doubt was expressed that consumers even read the labels. The chairman noted that the label is the last opportunity that the manufacturer has to communicate definitively with the ultimate user. This opportunity should be carefully taken advantage of in order to achieve maximum possible customer satisfaction and repeat business in the marketplace.

### SUBCOMMITTEE D01.18 EDITORIAL

#### H.E. Ashton, Chairman

*D01.18.02, Indexing.* S. LeSota, Chairman, discussed the progress on revision of the index of the 06 parts of the Book of Standards. Pigments, which are a big problem, took up most of the meeting time.

It costs money to make these changes. Therefore, the aim is to make as many improvements as possible within the limits of the assigned ASTM budget. It will be recommended that this budget be increased, considering the importance of this endeavor. Additions are being given priority, since deletions also cost money.

It is being recommended that key words be mandatory for all future standards and at the time of the five-year revisions subject for balloting and that future standard titles be carefully scrutinized to make sure they truly describe the test.

Changes in the recent indexes show that definite progress is being made.

However, this will have to be a continuing effort in D-1 and more work will have to be done by the individual subcommittees.

## **SUBCOMMITTEE D01.94 AWARDS & MEMORIALS**

**S.B. Levinson, Chairman**

The subcommittee was reorganized to reduce the total membership as a result of the completion of their terms by four former members, to reduce the membership of each task group, and to assign responsibility for awards to members who previously have received the specific award. The task group memberships are being revised.

The following members have been recommended for the Henry A. Gardner Award: Joseph M. Behrle (1986) and Laurence R. Thurman (1987).

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## **DIVISION 20 RESEARCH AND GENERAL METHODS**

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### **SUBCOMMITTEE D01.20 SAMPLING, STATISTICS, ETC.**

**H.E. Ashton, Chairman**

The chairman reported that D 3924, "Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquers, and Related Materials," and D 3925, "Practice for Sampling Liquid Paints and Related Pigmented Coatings," had been approved on Society ballot. The final copies were editorially reviewed.

As agreed at the January, 1985 meeting, the D-1 rating system (10-perfect, 0-complete failure) had been put in the format of a practice. After reviewing the document, it was agreed to submit it to concurrent Sub. D01.20/D-1 ballot and to send a copy to the chairman of Sub. D01.25 on Pictorial Standards of Coating Defects because several pictorial standards have rating schemes that do not agree with the D-1 scale.

Provisional Sub. D01.10 had submitted a proposed practice for accreditation of paint testing laboratories and requested a simultaneous Sub. D01.20/D-1 ballot. This has not been possible because it was a new topic, but copies had been made for review at the meeting. The chairman said

he would check with D.F. Savini on whether it is necessary to occupy the first several pages with applicable documents, when they are also listed in the table of relevant test methods (it is). It was agreed to submit the practice together with related E-36 document, E 994, "Guide for Laboratory Accreditation Systems," to ballot of the subcommittee and other interested parties.

As decided at the Orlando meeting, the definitions section of D 3980, "Practice for Interlaboratory Testing of Paint and Related Materials," had been submitted to the Committee on Terminology. The comments received were discussed. The chairman agreed to review the definitions to see whether those already covered by several definitions from other committees could be slightly modified to eliminate duplication. After the July meeting, the chairman had approached one computer programmer who agreed to write a basic software program to analyze results in accordance with D 3980. This was completed just prior to the meeting and two copies were distributed for evaluation. It was decided that since two other D-1 methods, D 1535 (Munsell Color) and D 2805 (Hiding Power), now contain such information, a footnote on the availability of the program would be added. This will be submitted to concurrent D01.20/D-1 ballot.

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

**R.W. Scott, Chairman**

*D01.21.13, Coordination of VOC Standards and Information.* H. Fujimoto, Chairman, reported that D. Crumpler of the U.S. EPA encourages development of the gas pycnometer for the determination of volume nonvolatile (VNV) or a good reliable VNV method. To determine the mass of VOC per volume of solids, it is necessary to know the VNV of the coatings. Since other data used in the equation to calculate this value is determined, the use of the theoretical "as supplied" VNV, as stated in U.S. EPA's Reference Method 24 (RPM 24), is not satisfactory to the state regulatory bodies.

In response, Sub. D01.21 has requested TG 24 on Volume Nonvolatile Matter of Clear or Pigmented Coatings to reorganize under the title of "Volume Nonvolatile Method Development" and to evaluate the gas pycnometer.

*Transfer Efficiency Test Method*—Much has been heard about the development of a TE method by the U.S. EPA via Centec. Since the Centec-supervised TE study was done at Inmont's Southfield

Automotive Research Center, a report is given here on some aspects of the test method.

All of the equipment for the test and the paint used are supplied. The parameters for the test are set and specified for: (1) fluid delivery; (2) air pressure; (3) gun to work distance; (4) spray viscosity; (5) line speed; (6) electrostatic conditions for electrostatic spraying, and (7) wet paint film thickness.

Two spray guns are used for the three types of spraying techniques tested: (1) airless conventional; (2) air atomized electrostatic (on), and (3) air atomized conventional (used electrostatic gun-off).

Ten 6" × 48" galvanized panels, or targets, are used, each of which is covered with a piece of weighed aluminum foil. In the test, the two leading and the last two panels are not used (scavenger). The inner six aluminum foil panels are weighed before and after the test run. Six runs were made for each type of spraying technique. The sprayed aluminum foils are baked and weighed.

*VOC in Inks*—S. Balistreri of the Bay Area Air Quality Management District (BAAQMD) in California is interested in a method to determine the VOC of inks. A copy of the recently developed Sub. D01.56 method to determine nonvolatiles in inks will be sent to him. He has developed a method for the regional EPA to determine the VOC of waste or salvaged materials. In California, waste products for landfills cannot have more than 1% VOC or the material must be treated to lower the VOC.

In summary, the method developed is a steam distillation technique where a weighed specimen is mixed with an excess of water and then the volatiles distilled over with the steam. C<sub>13</sub> hydrocarbons or products having a vapor pressure lower than 0.1 mm Hg has been arbitrarily set for the limit of VOC volatility.

All these fractions were treated with a 1% potassium dichromate in a concentrated sulfuric acid solution for a colorimetric measurement (orange to green-brown). The correlation between the concentration of solvent and color intensity has been determined. The volatiles in the distillate can now be determined colorimetrically; not an exact color match, but close enough. Time required for the test is said to be one hour.

The proposed method will be written up and presented at the VOC workshop sponsored by the Golden Gate Society for Paint Technology, June 16, 1986 in San Francisco. Before proposing new regulations or test methods, the California air regulatory bodies hold informal workshops at which it flows out a proposed draft which can be critiqued informally. This allows industry input to the staff. The comments received are studied and the regulation or test meth-

od modified. The edited method then follows regular channels through public hearings.

Some of the task group members' questions on the determination of VOC in waste products were: (1) how does one get a good representative sample; (2) how is the sample dispersed in water; (3) are all the solvents extracted; (4) the proposed color test is sulfonation of the solvents and the color varies with the solvent tested; (5) why do we need another method to measure VOC?

*Rule 1113* of the SCAQMD has been extended past the September, 1985 deadline. This regulation sets the limit of 380 gms/L for non-flat architectural coatings. In September, 1985, the VOC maximum was to have been lowered to the 250 gms/L requirement, but the paint industry petitioned for a hearing to extend the old regulation since it could not meet the lower VOC values. U.S. EPA, Region #9, became aware of this and verbally stated it opposed the extension of time. No formal denial has been made through the *Federal Register* at this time. Is this the harbinger of EPA action throughout the U.S. in order to meet the VOC standards of the Clean Air Act?

*STT and STP*—Under Sub. D01.08, Environmental Concerns, ASTM's Special Technical Publication (STP) and Standard Technology Training (STT) are under study to expedite dissemination of VOC measurement methods to paint manufacturing personnel, users and government regulatory bodies and/or the training of their technical staffs to measure VOC in coatings.

ASTM's STT program, under Chairman K.C. Pearson, appears to be the fastest and most efficient approach. We are talking about setting up VOC workshops sponsored by either ASTM, U.S. EPA, regional Federation of Society for Coatings Technology, NPCA, the paint industry, and/or local government regulatory bodies, staffed by Sub. D01.21 members. The STT staff would set up the training programs and handle the details. If successful, D-1 could benefit from any profit accrued from these workshops.

*Microwave Oven*—J.C. Weaver reported on J. Avery's (CEM) suggested use of the microwave oven to determine nonvolatiles in coatings. The instrument has been evaluated by PPG and Inmont. The method is quick and precise, but the nonvolatile results differ from those obtained by using D 2369 (60 minutes @ 110°C). For repetitive nonvolatile determination of the same coatings, microwave might work. However, for variable miscellaneous coating products, the results would vary depending upon the type of paint tested.

*D01.21.22, Analysis of Electrocoat Bath Samples*, W.B. Van DerLinde, Chairman,

discussed results of recent round-robins (RR) tests on the Determination of Volatiles in Electrocoat Baths. Preliminary determinations show some weaknesses in the test methods. In summary, the procedure is to inject the specimen into a GC and the eluting components identified based on retention times in the resulting chromatogram. Three external calibration standards are used to determine the concentration vs response characteristics of the eluting components. A six (6) ft., 1/8 inch diameter stainless steel tubing, which is packed with 20% by weight of UCON® LB-550-X plus 6% by weight of potassium hydroxide on 60-80 mesh, Chromasorb G, AW, DMCS treated, or equivalent, is used.

After a lengthy discussion, it was agreed:

(1) An internal standard will be used. H.D. Swafford will provide the procedure and specify the internal standard to be used.

(2) Since the volatiles incorporated into the RR samples are commercial products, the task group requested the chairman to prepare and send to each of the collaborators, a known solvent mixture which will contain the standard volatiles and the internal standard.

(3) To improve reproducibility, the specimen will not be centrifuged prior to injection into the GC. The determination of anionic contaminants in electrocoat baths will be explored prior to the next meeting. To evaluate the performance of the different types of ionic chromatographic instruments, samples of spiked ultrafiltrate will be sent to H.D. Swafford, T. Benga, H. Fujimoto, and K.H. Leavell for analysis.

*D01.21.23, Thermoplastic Traffic Marking*, J.M. O'Brien, Sr., Chairman, held a brief discussion on precision data obtained from the round-robin for the determination of titanium and chromium in thermoplastic pavement markings. After Chairman O'Brien reviews this data, a final version of this proposed standard, "Analysis of Thermoplastic Traffic Markings," will be submitted to Sub. D01.21 ballot.

*D01.21.24, Revision of D 2697, "Volume Nonvolatile Matter in Clear and Pigmented Coatings,"* M.E. Sites, Chairman, reported that the revision passed the Sub. D01.21 ballot. Comments received will be incorporated and it will be submitted to Society ballot.

Based on the request of the U.S. EPA, via D. Crumpler, to develop a more reliable and less time-consuming method for the determination of VNV of coatings, this task group will remain active as "VNV-Method Development."

One of the most promising approaches to solve this problem is the use of the helium gas pycnometer. Both PPG and Inmont laboratories have generated data which

compares VNV obtained with D 2697 vs a gas pycnometer and which looks very promising. The results are comparable to those obtained using D 2697. The actual density measurement of the dried coating takes less than a minute.

There are three models available: (1) manually operated; (2) automated; and, (3) reduced measuring compartment (less sample). These types of instruments will be evaluated by K.H. Leavell and H. Fujimoto, before the next meeting, to ascertain if they are all applicable and give similar results.

Sample preparation was discussed. It was agreed a specified dried film thickness should be employed either by use of a drawdown blade or by spray. The technique used is to apply the coating on glass slides, bake, and then remove the paint film. The weighed dried film (about five grams) is put into a cup and placed in the pycnometer. The instrument measures the volume of helium displaced by the sample and calculates its density. Knowing the density of the wet film and dried film, the VNV can be calculated. An informal RR will be run by three laboratories which have the gas pycnometer prior to the next meeting.

A discussion ensued on the merit of the precision statement generated for D 2697 using both E 180 and D 3980. E 180 expresses precision as relative values, and D 3980 expresses it as absolute values. In order to eliminate confusion on these values, it was agreed that the precision statement will give the absolute values and then the relative values in parentheses.

*D01.21.46, X-Ray Analysis of Pigment*, A.M. Snider, Jr., Chairman, discussed the final draft of a proposed new standard, "Determination of Titanium Dioxide in Paint by X-Ray Fluorescence Spectroscopy." This method has been submitted for ballot.

Work will start immediately on a proposed new standard for the determination of pigment components in paints by use of x-ray diffraction. Five collaborators volunteered to participate in a RR. The material to be analyzed will be selected by the chairman and distributed along with the proposed method.

*D01.21.53, Trace Levels of Monomers in Paints*, J. Benga, Chairman, reported that a proposed new standard, "Determination of Free Monomer Content in Latexes Using Gas Chromatography," will be rebalotted after changes are made as a result of the negative and comments received on the recent ballot, all of which were approved.

The task group's next activity will be to concentrate on a procedure for the "Determination of Isocyanates in Resins by Derivatization and Separation by Use of a Liquid Chromatograph."

*D01.21.54, Revision of D 4017, Water (Karl Fischer Method)*, J.L. Abbamondi, Chairman, reviewed the results of the latest RR for water determination using Hydranal® reagents. Results from three participating laboratories were in good agreement, but one laboratory's results indicated difficulty in using the method. Particular problems are noted in dispersing the water-reducible coatings in the solvent(s) mixture selected.

It was agreed that a new RR should be initiated. Test samples will be selected to cover the same range of water content specified in the current method. Specific procedures will be rewritten and detailed to help overcome the previous problems encountered by the collaborators. Results from this RR should indicate whether the revision of D 4017 can include the use of the Hydranal reagents.

*D01.21.56, Revision of D 3960, VOC of Paints and Related Coatings*, M.E. Sites, Chairman, did not act on H.E. Ashton's negative on this method on the D0104(85-4) ballot because the changes made in the scope will satisfy the dissenting vote, and the edited method requires rebalancing. Other comments received on the ballot were discussed and acted upon. The revised scope (Para.1.3) reads as follows: "This practice includes all organic materials volatile under the test conditions. Allowances have been made for organic materials which are considered to have negligible photochemical reactivity for the purpose of brevity in this document, these materials are referred to as exempt."

It was agreed to include an addendum to D 3960 which will consist of a precision statement derived from the results of a RR conducted by General Motors and its paint suppliers. This data shows good precision when evaluated by E 180, which is surprising since it is much better than the precision obtained from the task group's RR on determining VOC in test coatings. In addition, this data will be re-evaluated by D 3980 and the precision expressed as % absolute. In the addendum, % relative values will be placed after % absolute in parentheses. The revised D3960 is ready for concurrent Sub. D01.21/D-1 ballot.

Sub. D01.21.54's revision of D 4017, "Water in Coatings by the Karl Fischer Method," shows promise based on the results obtained from an initial RR using Hydranal reagents. The task group plans to conduct another RR before the next meeting, which will involve more laboratories, and hopefully, a wider range of water-containing test samples.

The question was raised pertaining to the level of water necessary in a coating in order to calculate the VOC of coating as grams/liter of coating minus water. Connor stated any amount of water can be excluded from the VOC calculations. H. Fujimoto is

under the impression that the EPA requires a 5% water content in the coating before the water can be subtracted from the total VOC. He will check this out and report on his findings. He will also investigate EPA's RM 24 use of 8.33 as a conversion factor from grams/liter to pounds/gallon instead of 8.345 as used in D 3960.

*D01.21.80, Exploratory Analytical Research*, K. Leavell, Chairman, discussed a better way of determining volume nonvolatiles via Quantachrome's gas pycnometer. Problems exist around instrument availability and sample preparation. H. Fujimoto and K.H. Leavell have agreed to work with the new Sub. D01.21.24 to start a round-robin using this instrument. Five labs have agreed to participate in a RR (J. Benga, H.D. Swafford, K.H. Leavell, H. Fujimoto, H.E. Ashton).

A round-robin for the determination of anions in cationic electrocoat via ion chromatography was discussed. W. van der Linde will coordinate this effort with the five collaborators. As a preliminary, the focus will be on Cl, F, PO<sub>4</sub>, NO<sub>3</sub>, SO<sub>4</sub>, CrO<sub>4</sub> and CO<sub>3</sub> ions in the ultrafiltrate. Preliminary scouting will be done first for a method using a normal and a spiked sample (in <50 ppm range).

Also discussed was the development of a method for molecular weight measurement of resins using modern size exclusion chromatography and software. H.D. Swafford agreed to talk to T. Prover of Glidden about the feasibility and desirability of developing a general guideline for GPC analysis.

R.W. Scott reported that he had received a letter from T. Prover stating that particle size analysis equipment is available which can measure the particle size and distribution of paint products which are of general interest to the coatings industry (i.e., latices, pigments, etc.). The chairman will evaluate the information in the letter with the intention of developing a future test method.

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

**H.A. Wray, Chairman**

In the absence of the chairman, discussion was primarily on the "Proposed Guide for Precautionary Statements" used in D-1 Standards, and the balloted revision of D 3278, "Test Method for Flash Point of Liquids by Setaflash Closed-Cup Apparatus."

It was noted by the staff manager that any negatives on D-1 ballots, prior to the final approval of the D-1 policy, will be found not persuasive. The precautionary statement will be redrafted to include concerns of the COS Blue Book Subcom-

mittee, and it will be rebalanced by Sub. D01.22.

On the revision of D 3278, negatives were received by A. Richardson, E.R. Lewis, W.S. Engle and D.L. Lewis. It was voted by the members present that A. Richardson's negative was persuasive and W.S. Engle's comments will be added with the opinion that they cannot be made editorially. The method will be redrafted and reissued for a subcommittee ballot.

A draft, "Standard Test Method for the Determination of the Solvent Separated Layer from a Viscous Liquid," was circulated on behalf of the chair, and it was decided that this document should be issued for a subcommittee ballot.

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

**M.P. Morse, Chairman**

*D01.23.08 on Application of Uniform Films*, M.P. Morse, Chairman, is drafting revisions to D 823, "Preparation of Uniform Films," to include procedures for applying films to panels by hand spray and manual draw-downs.

*D01.23.10 on Adhesion*, H.E. Ashton, Chairman, reported that information has been received that the present Permacel 99 that is recommended for conducting D 3359, "Adhesion By Tape Test," may not meet the pull strength values specified in the method. This is being investigated and alternative suitable tapes will be sought.

Efforts are underway to extend D 3359 for use on thick films. Techniques involving modifications to the dimensions of cross-cut knives are being evaluated in round-robin tests.

*D01.23.14 on Hardness, Mar and Abrasion Resistance*, M.P. Morse, Chairman, reported that the revision of D 1474, "Indentation Hardness of Organic Coatings," has been approved by Society ballot.

Round-robin tests to establish the precision of Koenig and Persoz pendulum test procedures given in D 4366 have been completed. The test results have been analyzed and a precision statement has been drafted for letter ballot. The analysis shows that the Persoz pendulum exhibited considerably greater sensitivity in detecting hardness differences between baked finishes.

The group is investigating test procedures to measure mar resistance of coatings. A round-robin test is being undertaken to investigate the suitability and precision of the Balanced Beam Scrape Tester in measuring mar resistance. The



suitability of this tester will be determined by comparisons with erasure rub and fingernail and coin scratch tests.

*D01.23.15 on Slip Resistance*, M.P. Morse, Chairman, reported that a new method, "Measurement of Static Friction of Coatings," has been approved by Society ballot. A revision of this method to include a procedure for measuring static friction of rough coatings has been drafted and will be submitted to subcommittee ballot.

There is a need for a procedure to measure static friction of coatings wet with moisture. The procedures given in the new method are not suitable for performing such measurements. There is a good possibility that the Brungraber Tester may be suitable. It will be investigated.

*D01.23.16 on Water Vapor Permeability*, M.P. Morse, Chairman, reported that a major revision of D 1653, "Water Vapor Permeability of Organic Coating Films," has been approved by Society ballot. A round-robin test to establish the precision of this method is being initiated. Films to be tested are: exterior flat latex house paint, "top of the line" interior flat latex paint and alkyd semi-gloss enamel. The test conditions to be used are dry- and wet-cup procedures at 73° F and 50% R.H.

*D01.23.18 on Elongation*, M.P. Morse, Chairman, reported that minor revisions of D 522, "Elongation of Attached Organic Coatings with Conical Mandrel Apparatus," and D 1737, "Elongation of Attached Organic Coatings with Cylindrical Mandrel Apparatus," have been approved by Society ballot. A draft of a method combining these two test procedures is being prepared.

*D01.23.20 on Preparation of Free Films*, M.P. Morse, Chairman, has drafted a procedure for forming free films suitable for testing. It will be submitted to subcommittee ballot. Procedures given describe application of films to release paper, TFE surfaces, and dental foil, cutting specimens with a special knife and then stripping them from the substrate. Films are removed from the dental foil by amalgamation with mercury.

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

**C.K. Schoff**

*D01.24.19, Viscosity by Efflux Cups*, continued to discuss D 4212, "Viscosity by Dip Cups." It had been decided previously to break the method into two separate documents: (1) a revised D 4212 to

include only Zahn cups, and (2) a new method for Shell cups. The documents were recently rebalotted at the subcommittee level. A number of editorial comments were received and most will be incorporated into the methods. P. Gardner again cast negatives on the methods. Some of his suggestions will be included, but his negatives were unanimously rejected as being not persuasive or germane. Both methods will be submitted to D-1 ballot after editorial changes have been made.

It was felt that the present Zahn cup precision statement was questionable with regard to reproducibility (it appears that a single set of cups was circulated to the cooperators). Therefore, a new round-robin to collect precision data will be carried out before the next meeting of D-1. Number 2 Zahn cups will be used to test three paints, inks, and one or two standard oils. Cooperators will use their own cups, regardless of supplier.

*D01.24.20, Rotational Viscometers*, briefly considered the revision of D 2196, "Viscosity by Brookfield Viscometer." The document will be submitted to D-1 ballot. D 4287, "High Shear Viscosity by ICI Cone/Plate," also was discussed. It was pointed out that no provision is given in the method for zeroing the viscometer. The zeroing lever is mentioned, but in the context of adjusting the viscometer to the viscosity of a standard oil. It was felt that such adjustment caused more problems than it solved and should be removed from the method. Instead of adjusting the instrument to fit a given viscosity, a calibration curve should be developed with three standard oils. Corrections can be made from this curve. A revision of the Preparation of Apparatus section will be presented at the June meeting.

*D01.24.26, Electrical Properties of Liquid Paints and Paint Materials (Including Solvents)*, reviewed the latest draft of the proposed method on "Measurement of Electrical Resistance of Liquid Paints and Paint Materials." It was decided to limit the method to d.c. measurements, include procedures for both the Ransburg and Byk instruments, and address the questions of instrument, specimen preparation, temperature effects, etc. The method has a long way to go, but it is hoped to have a working procedure by the June meeting, so that interlaboratory testing on selected solvents can be set up.

*D01.24.27, Flocculation*, got a new lease on life as several people attended. It was decided to start with a very limited method that would mainly consist of a definition of flocculation and a list of methods with references.

*D01.24.28, Evaporation*, discussed D 3539, "Evaporation Rates of Volatile Liquids by Shell Thin-Film Evaporometer." It was decided to submit the current revision to Sub. D01.24 ballot after some additional editorial changes, including evaporometer (Quintel EV-1) and the non-availability of the manual recording Shell evaporometer (the automatic apparatus is available from FLC Instruments). Future work will involve consideration of a more general method to cover both the Quintel and Shell/FLC evaporometers.

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

**C.J. Sherman, Chairman**

*D01.26.02, Color Measurement*, C.S. McCamy, Chairman, reported that D 1535, "Method for Specifying Color by the Munsell System," is being revised. It was agreed that the Munsell Value table should be that accepted at the meeting in July, 1985. Some mention is to be made of the correspondence of the table to the Munsell Book of Color now being produced.

D 2244, "Method for Instrumental Evaluation of Color Differences of Opaque Materials," is being printed. The earlier concern about precision was resolved because this method is purely computational and doesn't require a precision statement.

D 3134, "Recommended Practice for Selecting Color and Gloss Tolerances," received four negatives on the last ballot. These were considered in detail. A new draft will be prepared by W.N. Hale based on these considerations and circulated to the task group members.

After a review of D 4086, "Practice for Visual Examination of Metamerism," a minor change was made. It will be submitted to concurrent Sub. D01.26/D-1 ballot.

D 1729, "Practice for Visual Evaluation of Color Differences of Opaque Materials," is being revised for balloting.

*D01.26.06, Hiding Power*, L. Schaeffer, Chairman, was informed that all necessary changes had been made in the text of the upcoming version of D 2805, "Test Method for Hiding Power of Paints," to respond to comments and previously resolved negatives. The finalized version is to be published in the next edition of the 06.01 Book of Standards.

The subcommittee chairman proposed that the two current D-1 hiding power methods, D 344 and D 2805, be circulated among D-1 members with a request for information as to what kind of methods are now actually being used in their laboratories. A similar request is to be insert-

ed in the JOURNAL OF COATINGS TECHNOLOGY, if this can be arranged.

Hopefully, this would also stimulate some new membership for both Sub. D01.26 and D-1. It was noted that the time for accomplishing these steps before the next meeting was ample, since Sub. D01.26.06 will skip the summer meeting entirely, because of a conflict with the ISCC meeting in Toronto in June.

*D01.26.11, Gloss and Goniophotometry*, M.P. Morse, Chairman, reported that a negative vote received from F. Billmeyer on the recent D-1 balloting for the reapproval (with small editorial change) of D 4039, "Reflection Haze of High Gloss Surfaces," was considered persuasive. The following editorial changes will be made before it is submitted to Society ballot:

(1) Paragraph 4.1 will be deleted. It confuses the meanings of haze and lack of clarity. Furthermore, it contributes little to the understanding of the significance and use of the method.

(2) Paragraph 3.2 will be added to state that this method is applicable to non-metallic specimens having a D 523, 60° specular gloss value greater than 60.

(3) Paragraph 4.3 will be modified to explain in more detail why haze-free organic coatings (usually having a surface refractive index of 1.53) will have a haze value of 5.

M.P. Morse presented some graphical plots illustrating relationships between a visual ratings scale and the D 523, 60° and 20° specular gloss scales. These plots are based on the data generated for a series of 20 gray painted panels by F. O'Donnell in his Ph.D. thesis work at Rensselaer Polytechnic Institute. The graphical plot for the D 523, 60° gloss ratings indicated that in the gloss range of 60 to 65, the instrumental measurements are insensitive to, and do not correlate well with, the visual ratings. A plot of visual ratings versus 60° gloss values, using a receptor aperture of approximately one-half the size of that specified in D 523, indicated the same lack of sensitivity and correlation occurs in the middle of the gloss scale.

Comments were made that perhaps the non-linear relationships between visual ratings and 60° and 20° D 523 gloss scales exhibited for these gray painted panels may not be representative of relationships that exist for other types of coated panels. Consideration is being given to assembling additional sets of panels and performing visual ratings and gloss measurements on them to gain additional information on this subject.

It was agreed that a statement should be inserted in D 523 to caution users that the gloss scales may be non-linear with respect to visual rating scales.

*D01.26.23, Reflectance of Horizontal Coatings*, N. Johnson, Chairman, reported

that the second draft revision of D 4061, "Test Method for Specific Luminance of Horizontal Coatings," was presented for balloting. Progress on the round-robin to explore closer controls on the laboratory test for retroreflectance of horizontal coatings was reported. W.N. Hale reported that J. Rennilson was working on the test panel preparation technique for beads on paint test panels and expected a progress review by the next meeting. The chairman described a report in preparation by CIE TC 4.04 on pavement marking materials.

*D01.26.24, Tinting Strength*, C.J. Sherman, Chairman, did not meet, but D 332, "Tinting Strength of White Pigments," and D 2745, "Instrumental Tinting Strength of White Pigments," have been revised to include the comments from the last balloting and will be submitted for concurrent Sub. D01.26/D-1 ballot. D 387, "Color and Strength of Color Pigments," will be balloted for reapproval.

*Publications*—Committee E-12's compilation of appearance standards does not contain all of the methods under Sub. D01.26 jurisdiction. Since the compilation is being revised and updated, it was the consensus of the group that the Sub. D01.26 chairman examine the standards not included and make recommendations of those to be included, or possibly that a separate section be prepared for coatings.

Revision of some chapters of STP 500, Paint Testing Manual, is progressing. It is intended that the chapters will be published as they are completed. H.K. Hammond III and F. Billmeyer have revised the chapter on Color, but it's been a while since that was done, so Hammond will examine those chapters again to determine if further revision is necessary.

Because of the conflict of D-1 and ISCC meetings, Sub. D01.26 and its task groups will not meet with D-1 in June in Louisville. One group, Sub. D01.26.02 on Color, will meet with E-12 at the ISCC meeting in Toronto.

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

**B.L. Williamson, Chairman**

*D01.27.02, Water Tests*, D. Grossman, Chairman, discussed comments received on the four water tests on the D0104(85-4) ballot. The group, and subsequently the subcommittee, agreed to a number of suggested editorial changes. A technical change regarding time between specimen removal from the test and evaluation was agreed upon.

The four practices (D 870, Water Immersion; D 1735, Water Fog; D 2247, 100% Relative Humidity, and a new

practice, D 4585, on controlled condensation) will be submitted to Society ballot. At the same time, the items which are being revised will be placed on D-1 ballot.

*D01.27.04, Light and Water Exposure Apparatus*, S.M. Totty, Chairman, discussed the comments and negatives received on the D0104(85-4) ballot on the extensively revised D 822 (Carbon Arc Type). The negatives were withdrawn based on the understanding that work begin immediately on updating D 822. The group agreed to do this as well as to start the development of a new practice for conducting exposures using Xenon-Arc apparatus.

*D01.27.09, Corrosion*, F. Lutze, Chairman, discussed the details of the round-robin which will be conducted prior to the next meeting and which will evaluate various cycle tests that are being used in industry to duplicate the scab-like corrosion which occurs in use.

*D01.27.10, Accelerated Outdoor Weathering*, M.P. Morse, Chairman, is currently involved with the evaluation of exposures of 12 automotive coatings (Series D01.27.10-84). The exposure study is designed to accomplish several things: (1) Comparison of the new EMMA-NTW (night-time-wetting) cycle at DSET Labs in Arizona to Florida black box exposure; (2) Comparison of black box exposure to backed and unbacked 5° S open rack exposure at South Florida Testing Service; and (3) Comparison of black box exposure at four Miami area exposure sites (DuPont, General Motors, South Florida Testing Service, and Sub-Tropical).

The chairman distributed tables showing the gloss loss and color change produced in these exposures after 12 months (DSET panels exposed for shorter periods). These results were discussed.

DSET Laboratories had run several variations of their EMMA-NTW cycle and agreed to conduct additional tests combining a couple of these variations which, based on results already obtained, should result in good correlation with Florida black box. It is planned to discuss the results of these DSET tests, as well as 18 months' Florida exposures, at the June meeting.

*D01.27.14, Filiform Corrosion*, B. Williamson, Chairman, discussed a proposed round-robin including outdoor testing as well as laboratory testing. It is anticipated that results from this round-robin testing will be available for discussion at the next meeting. The purpose of the round-robin is to determine if the development of filiform corrosion can be accelerated over the test currently specified in D 2803.

D01.27.17, *Evaluation of Weathering Effects*, A.S. Allen, Chairman, discussed the editorial comments received on the D0104(85-4) ballot on D 661 (Cracking), D 662 (Erosion) and D 772 (Flaking). Where it was felt appropriate, changes were made. The three items will be submitted to Society ballot. Since these are all standard methods, they should have a "Precision and Bias" section. Such a section for each of these methods will be submitted to D-1 ballot.

The completed extensive revision of D 660, "Evaluation of Checking," will also be placed on Sub. D01.27 ballot in the near future.

Discussion was started on an initial draft of a revision of D 661. The chairman will add the suggested changes. Discussion on them will continue at the next meeting.

*Additional Business*—D 1543, "Color Permanence," received no negatives. Comments were discussed and editorial changes made where thought appropriate. It will be sent to Society ballot. A question was asked whether there was any reference to the work that showed the color changes occurring in the test approximated those found in home atmospheres. (Note: If anyone has any thoughts on where this information might be, please contact the D01.27 chairman.)

D 1006, "Exterior Exposure Tests on Wood," received no negatives on the balloting for reapproval. It will be submitted to Society ballot.

D 2246, "Humidity-Thermal Cycle Cracking," received no negatives on the balloting for reapproval. However, there were several comments which the subcommittee agreed merited consideration. Since some of these were more than editorial in nature, the method has been reviewed in light of the comments. D.C. Gleason will review these and a task group will meet in June to finalize any proposed revision.

D 2933, "Corrosion Resistance of Coated Steel Specimens (Cyclic Method)," also received no negatives. However, based on comments from H.E. Ashton, which were primarily, but not entirely, editorial, it was agreed to draft a revision of the method for Sub. D01.27 ballot.

## **SUBCOMMITTEE D01.28 BIODETERIORATION**

### **D.L. Campbell, Chairman**

D.L. Campbell reported that Sub. G03.04, Biological Deterioration, plans to work on a proposed test method for soil burial. A similar procedure is in D 3083, "Specification for Flexible Poly (Vinyl Chloride) Plastic Sheeting for Pond, Canal, and Reservoir Lining."

The chairman reported that Chapter 6.4 of STP 500 was revised by Sub. D01.28. It appears that ASTM Headquarters is not planning to reprint the Gardner Manual (STP 500). It has been suggested that Sub. D01.28 propose a separate publication on Biodeterioration. It would include Chapter 6.4 of STP 500, results of 1973-74 exterior mildew exposures, test methods for deterioration, copies of papers presented 1/14/86, and other information on deterioration. Other ASTM committees will be contacted for in-pat.

D01.28.01, *Package Stability*, A.R. MacGillivray, Chairman, reported that materials are to be supplied to participating companies by the second quarter. Initial testing is designed to elicit suggestions based on the hands-on experience of performing the procedure as currently drafted. A mercurial preservative will be used as the control. The potential of grow-back and the measurement of this potential were discussed. In addition, since the procedure is based on serial dilution to failure, neutralizing media may not be required. These aspects of the test are to be investigated in the next year.

D01.28.02, *Rapid Determination of Enzyme Presence*, D.P. Leipold, Chairman, agreed that data from the December, 1985 round-robin showed that Carboxymethyl Cellulose (CMC) 7H3S was a suitable substrate for determination of cellulose enzyme activity. Reproducibility of both viscosity and viscosity loss among five cooperators was excellent.

Prior to the June meeting a round-robin will be conducted with six cooperating members. Samples of both vinyl-acrylic and acrylic paints will be supplied. These paints will be mixed with CMC 7H3S solutions and inoculated at four enzyme levels to test the viscosity loss response of the CMC-paint mixtures.

D01.28.04, *Resistance of Paint Films to Attack by Algae*, W.B. Woods, Chairman, reported that the unprotected control specimens are to consist of birch tongue depressors dipped in an exterior acrylic latex paint and allowed to air dry for 48 hours. Surface sanitization will be achieved by exposure to ultra-violet light. Five replicates will be tested. A revised method will be sent to interested parties. Companies participating in collaborative testing are to be one gallon of unprotected acrylic paint and culture of the test organism, *Oscillatoria*, within the next three months.

D01.28.05, *Recoating Mildewed Surfaces*, D.L. Campbell, Chairman, reviewed the sixth draft of the new "Standard Guide for Determining the Presence of and Removing Microbial (Fungal or Algal) Growth on Paint and Related Coatings." Discussion centered on the diminished disinfectant properties of aqueous sodium hypochlorite

solutions at pH 7 and above, versus the desirability of a single treatment with a blend of sodium hypochlorite solution and trisodium phosphate solution.

The sixth draft will be modified to offer two procedures for removal of fungal and algal growth:

(1) Wash with 5% trisodium phosphate in water or 2% non-ammonia-containing laundry detergent in water, rinse, wash with sodium hypochlorite solution, rinse.

(2) Wash with sodium hypochlorite solution and 2% laundry detergent solution combined 1:1 by volume, rinse.

The revised draft has been submitted for concurrent D01.28/D-1 ballot.

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## **DIVISION 30 PAINT MATERIALS**

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### **SUBCOMMITTEE D01.31 PIGMENT SPECIFICATIONS**

#### **C.W. Fuller, Chairman**

The subcommittee addressed three negatives from D0104(85-4). Two, concerning D 1199, "Specification for Calcium Carbonate Pigments," and D 1366, "Recommended Practice for Reporting Particle Size Characteristics of Pigments," were resolved by agreement that they were persuasive. The third, involving D 209, "Specification for Lampblack Pigment," which was to be dropped, was held over for more information. In the latter case, if commercially available materials can be identified, which conform to the specification, reinstatement will be considered.

There was a group of five late negatives, one of which was addressed in the time available. It concerned D 604, "Specification for Diatomaceous Silica Pigment," for which five points were presented. Two were considered not persuasive, two were considered editorial and one was persuasive enough to require a substantive change and rebaloting.

There were eleven other items to which editorial comments were submitted. Two were addressed, D 1199 and D 79, "Specification for Zinc Oxide." There was agreement with most of the comments with final details left to the ASTM Editorial staff.

The meeting closed with a discussion of Sub. D01.31 organization. At the time Chairman Fuller's occupational status was uncertain. It appears, subsequently, that he will be able to continue as chairman. W.J. Hart acted as temporary chairman.

## **SUBCOMMITTEE D01.32 DRYING OILS**

**P.C. Stievater, Chairman**

It was reported that of the 18 items appearing on the D01 (85-3) ballot, 11 received only editorial comments. These will go to Society ballot after the necessary changes are made. Five received technical comments. After revision, these will be resubmitted to D-1 ballot.

A negative received on D 12, "Standard Specification for Raw Tung Oil," from R. Martin was found to be persuasive. He objected to changes designed to make D 12 conform to the other specifications for tung oil and submitted strong justification for his position. This standard is being removed from the ballot and will be rebalotted at D-1 level with the objectionable changes removed, essentially taking it back to its original condition.

A negative on D 1983, "Test Method for Fatty Acid Composition by Gas-Liquid Chromatography of Methyl Esters," at subcommittee level, requesting many changes, some of which were technical in nature, was found to be persuasive. The method will be revised and resubmitted to D-1 ballot.

## **SUBCOMMITTEE D01.33 VARNISH & RESINS, INCLUDING SHELLAC**

**R.A. Orr, Chairman**

The major goal for 1986 will be the review/revision of the 34 standards requiring action and the assignment of method stewards. D 154, "Guide to Testing Varnishes," completed Society balloting successfully. Sub. D01.33.01 on Varnishes will become inactive until any new business is received.

D 3680, "Residual Vinyl Chloride Monomer Content of Poly (Vinyl Chloride) Resins, Compounds, and Copolymers by Solution Injection Technique," has not been transferred from D-20 to D-1 as requested by Sub. D01.33.25 Chairman, J.J. Brezinski. Chairman Orr will follow up.

*D01.33.23. Phenolic Resins*, H.D. Marshall, Chairman, discussed in-depth the methods for (1) Nonvolatile Matter, (2) Stroke Cure, and (3) pH of Water Insoluble Phenol-Formaldehyde resins. Although these methods passed Sub. D01.33 ballot, they were not submitted for D-1 ballot because some inconsistencies existed between prior change requirements and the balloted method. Revisions of the methods will be submitted for D-1 ballot.

A new method for incline plate flow differs from D 4242 only by the angle of incline. The combining of these two methods will be investigated. However, the new method will be routed to the task group for review.

*D01.33.24. Nitrogen Resins*, J.H. Smith, Chairman, discussed the third draft of a titration method for determination of free formaldehyde in amino resins. A round-robin will be conducted on a slightly modified version of the method. Changes to be made involve clarification of the procedure for reagent preparation and temperature control. Cooperators are needed for further progress on this method.

*D01.33.26. Emulsions*, R.M. Schiller, Chairman, discussed data from the six participants of the first round-robin for the method, "Filter Retained Solids Content of Latex Vehicles." Consistency of data was not indicated in any of the three samples as wide ranges were experienced between participants. Low confidence levels were reported in all cases. A new round-robin will be conducted using a modified sample preparation procedure and a time limit on analysis. It is felt that a better sample mixing technique and the prevention of skin formation over excess sample lifetime will improve these data. More input is required on the filter apparatus as some problems were experienced in reuse of the PVC fitting.

*D01.33.27. Epoxy Resins*, R.A. Orr, Chairman, discussed a method for determining easily hydrolyzable chlorides of epoxy resins via potentiometric titration using silver nitrate. Comparisons were made between this method and D 1726, the existing hydrochloric acid back-titration procedure. Since D 1726 is due for review, it was decided to add the new method to it as an addendum. Another method for determining total hydrolyzable chlorides was also discussed. The chairman will rewrite it in ASTM format and distribute it for task group approval. Dow Method RPM 900A, "Residual Epichlorohydrin via Head Space Analysis," has been submitted to Committee D-20 on Plastics. It will not be considered by this task group.

## **SUBCOMMITTEE D01.34 NAVAL STORES**

**C.M. Winchester, Chairman**

A meeting of the subcommittee was held in Atlanta in conjunction with the meeting of the Pulp Chemical Association on October 1, 1985.

Members of the former Committee D-17 expressed unanimous opposition to the disbanding of D-17 and its transfer to D-1 as a subcommittee. A drop in mem-

bership or at least poor attendance by most of the old members is expected.

It will be extremely difficult to get an old member of D-17 to review the existing standards. D.F. Zinkel of FPRS has revised D 3008, "Resin Acids in Rosin by GC," which was balloted last fall. Only three former D-17 members responded, one of which was this subcommittee's chairman.

The proposed program to develop methods for ink vehicles has received good response. Two new members each from Union Camp and Westvaco are expected. Hercules has added a man from the Hattiesburg plant who is involved in ink varnish production. Excellent response was obtained from the mailing to Sub. D01.56. Representatives from five ink producers are expected at the current meeting, plus three others, including the chairman and secretary of Sub. D01.56. Three more Sub. D01.56 members expressed interest in attending future meetings, but could not be in New Orleans.

## **SUBCOMMITTEE D01.35 SOLVENTS, PLASTICIZERS AND CHEMICAL INTERMEDIATES**

**L.R. Thurman, Chairman**

*D01.35.10. Solvents—Hydrocarbons and Ketones*, S.A. Yuhas, Jr., Chairman, had determined that the round-robin data presented at the last meeting would be used to restate the precision and bias of D 1364, "Test Method for Water in Volatile Solvents (Fischer Reagent Titration Method)."

A research report will be presented at the next meeting, with the revised precision and bias statement, for balloting.

*D01.35.20. Reactive Monomers—Vinyl Monomers and Aldehydes*, T.H. Golson, Chairman, held a discussion concerning the development of a method for Traces of Polymers in Reactive Monomers. There was no general support at this time to develop such an ASTM method. A visual method of polymer detection using a suitable solvent is generally used in industry.

Alternative methods for determining the end point in D 1613, "Test Method for Acidity in Volatile Solvents," were discussed. It was agreed that the method is for a phenolphthalein end point; however, it is possible to use alternative instrumentation when it is proven to give equivalent results; i.e., pH.

*D01.35.30. Chemical Intermediates*, J.R. Morrison, Chairman, reported that GC methods for determining the ethyl methyl pentanol content and purity of 2-



ethylhexanol were submitted and will be prepared in ASTM format for the next meeting.

The proposed GC test methods for propylene glycol monomethyl ether and dipropylene glycol monomethyl ether were combined with a similar method for propylene glycol monomethyl ether acetate and will be considered at the next meeting.

*D01.35.50, Liaison*, L.P. Field, Chairman, reported that response to letters regarding information on Sub. D01.35 referenced standards in other groups has been limited.

Further attempts to review standards revision at the lowest level possible will be continued.

A request will be made to Subcommittee E-15.53 on Alcohols and Polyalcohols that E 202, "Method for Analysis of Ethylene Glycols and Propylene Glycols," reference D 4052, "Test Method for Density and Relative Density of Liquids by Digital Density Meter," as an alternative method of specific gravity determination.

*New Business*: W.E. Whitlock will replace W.J. Frost as Sub. D01.35 secretary.

The property limits of D 235, "Specification for Mineral Spirits . . ." were discussed. Values as found in the NPCA Raw Materials Guide, List of Suppliers, were used as the basis for the property limits in Table I.

A survey will be made of ASTM standards for test methods that determine physical appearance of materials that are used in Sub. D01.35 standards.

A proposed instrument that would meet D 1209, "Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)," was discussed and evaluated.

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## DIVISION 40 PAINT PRODUCTS APPLIED ON SITE

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### SUBCOMMITTEE D01.41 PURCHASE OF PAINTS AND RELATED COATINGS

**S.L. Pitcher, Chairman pro tem**

*D01.41.02, D 3927, Standard Guide for State and Institutional Purchasing of Paint*, W.V. Moseley, Jr., Chairman, reported that the "Weighted Cost of Active

Ingredients" section of D 3927 has been revised extensively as a result of the July, 1985 meeting in San Diego and the special Sub. D01.41 meeting at the National Bureau of Standards on September 26, 1985.

It was moved to submit this revised D 3927 to concurrent Sub. D01.41/D-1 ballot.

*D01.41.05, Latex Flat Wall Paint Specification*, I.V. Bloom, Chairman, resolved five negatives received on the Sub. D01.41 ballot. The specification as amended will now be submitted to ballot.

*Old Business*—The subcommittee approved the following revised scope for submittal to Sub. D01.41 ballot:

"To develop purchasing standards for finished whole paint and related coatings, including trade sales and industrial products for government, industry, and private use."

*New Business*—Reports were made on the ASTM/DOD/SAE meeting in Williamsburg, VA on December 5-6, 1985 and on the GSA/NIGP-sponsored meeting on the new Multiple Award Schedule purchasing program held in Washington, DC on January 10, 1986.

## SUBCOMMITTEE D01.42 ON ARCHITECTURAL PAINTS

**R.H. Rowland, Chairman**

*D01.42.01, Soil and Stain Removal (Mechanical Test)*, T.J. Sliva, Chairman, is working on a method of testing removal of various household stains with a mechanical device (Scrub tester) in place of the hand cleaning method utilized by Sub. D01.42.02. It was decided that government specifications for various common household stains and cleaners would be reviewed. These stains would be applied and removed, closely following procedures outlined in D 3450, "Test Method for Washability Properties of Interior Architectural Paints." Color and reflectivity differences would be recorded on the soiled and unsoiled areas.

The response from the ballot on Draft 5 of the group's "Practical Soil and Stain Removal" test method was reviewed. Comments received were mostly editorial in nature, but sufficient in scope to warrant redrafting the test for Sub. D01.42 balloting. Draft 6 was prepared as directed by the subcommittee and forwarded for ballot. The task group chairman will develop terms to be used in conjunction with the method before the next meeting.

*D01.42.06, Hiding Change-Wet to Dry*, L. Schaeffer, Chairman, will draft a proposed method for the next meeting.

*D01.42.13, Brushability*, D.P. Leipold, Chairman, circulated for comment the first draft of a test method for ranking brush drag of flat latex paints. The major technical revisions are the addition of a section on brush conditioning, a specification that duplicate tests be run, and a clarification on the type of brush to be used. The editorial revisions include addition of a disclaimer that this method does not pertain to roller application, addition of a key word section, and a change in the "Significance and Use" section toward more emphasis on this as a "stand-alone" method for brush drag. Metric units must be inserted throughout the method and abbreviations converted to ASTM standard form.

The question was raised as to whether variations in Stormer viscosity could influence the brush drag rating. It was decided that this should be resolved through experimentation prior to the June meeting.

*D01.42.18, Block Resistance*, U. Jackson, Chairman, discussed the results of the first round-robin. It was stated that there were too many variables in the method, resulting in a wide spread of data. Plans for a new round-robin will have a reduction in variables. The group also discussed alternative methods of testing for block resistance. S. LeSota explained one such method, which will be used in the next round-robin. This method requires tester sensitivity and will be evaluated for reproducibility and discussed at the next meeting.

## SUBCOMMITTEE D01.45 MARINE COATINGS

**L.S. Birnbaum, Chairman**

*D01.45.06, Dynamic Testing*, D. Laster, Chairman, L. Birnbaum chaired the meeting in the absence of the chairman. The July 1985 Sub. D01.45 minutes as published in the JOURNAL OF COATINGS TECHNOLOGY were corrected as follows: (1) First paragraph, "D01.44" should read "D01.45" and, (2) Second paragraph, "these thicknesses" should read "film thickness measurements."

Results of the Sub. D01.45 ballot on the new "Standard Test Method for Marine Antifouling Coating Systems under Hydrodynamic Flow" were reviewed. There were three negatives. As a result of telephone contact with the negative voters, D. Laster reported that agreements were reached for the withdrawal of two of the three negatives, subject to agreed-upon changes in the draft. The changes were incorporated at the meeting.

The third negative was cast on the basis that there was no data to show correlation between ship performance and rotor per-

formance and that results could be misleading. This negative was not resolved prior to the meeting. It was pointed out that this point was covered in 4.4 of the draft. The negative was found to be not persuasive by a unanimous vote of the attendees.

T. Dowd reported on actions to date on the flow channel standard including a mailing of Draft #2 of the standard to all Sub. D01.45 members. After review and discussion of this draft, Dowd agreed to prepare a new draft, including addition of a method for measuring film thickness of AF coatings before and after testing, and addressing other items discussed at the meeting. He will also include appropriate comments received prior to April 30, 1986 in the revised draft for dissemination.

*D01.45.07, Antifouling Rating.* C. Perez, Chairman, distributed a final report (dated 1/13/86) covering work done since 1981 to improve on the existing rating scheme described in D 3623, "Method of Testing Antifouling Panels in Shallow Emergence." Objectives included collection of data for reapproval of D 3623 based on performance of the same antifouling (AF) paint formulation manufactured by six different suppliers at three different sites, plus some exploratory exposures of two AF paints on "dynamic apparatus." The latter effort was limited because of fiscal restraints.

Comparative photographic data for 18 months from two Florida test sites and for six months from the Hawaii test sites were collected and will be maintained and available for inspection at the Miami Research and Testing Station, Miami Beach, Florida. It was agreed that sufficient data had been accumulated to justify balloting for re-approval of the existing D 3623 rating scheme. It was also agreed that the accumulated data supported the addition of an alternative rating method to the standard. Chairman Perez offered to prepare a draft to include the alternative method for balloting.

*D01.45.08, Organo-Tin Release Rate.* L. Birnbaum, Acting Chairman. EPA representative, L. Vlier reported that EPA has initiated a review of antifouling paints containing organotin as an active ingredient (9 different tributyl tin compounds are being used by more than 50 paint companies in about 340 products). As part of this review, they are requesting all suppliers of AF paints containing organotin for release rates of tributyl tin. EPA is looking for an industry consensus standard for determining tributyl tin release rates for use as a protocol for a data "call-in" to be promulgated in February 1986 to serve as a means of categorizing the commercially registered paints in terms of tin release rates. Copies of the 1/8/86 announcement in the *Federal Register* on the Initiation of the Special

Review and a tributyl tin Support Document were distributed.

Birnbaum reported that a working group (consisting of members from industry, NBS and Navy) had held two meetings after the July meeting to develop Draft #3, dated 11/24/85, which was mailed out to all Sub. D01.45 members for review and discussion at this meeting.

The following issues concerning this draft were raised:

(1) It does not provide early tributyl tin release rates (30-day conditioning period prior to sampling);

(2) It addresses organotin rather than tributyl tin (lack of specification);

(3) It does not satisfactorily address a potential bio-slime problem (variable growth when conditioned in natural sea water);

(4) It does not address a potential mixing problem (lack of laminar flow over a flat panel in a cylindrical tank).

The following change was recommended to 1.1: Substitute "organotin expressed as tributyl tin" for "organotin" and "synthetic seawater" for "seawater".

The following resulted in an effort to resolve the issues raised:

(1) A consensus was developed that the test specimen be a cylindrical pipe of such dimensions so as to provide an appropriate exposure surface immersed in a sufficient quantity of synthetic seawater to yield meaningful data, possibly applying the coating by dipping. This pipe section would be rotated at a rate to simulate a water flow of about one knot (about 60 rpm for a 2½ diameter pipe). Mr. Longmore of Hempel agreed to work out details for task group consideration.

(2) It was agreed that the extraction method be modified by extracting with toluene, washing with NaOH and twice with distilled water to separate out tributyl tin; also to add an optional procedure for those who wished to speciate to a greater extent. It was felt that although the extraction procedure could still give high values for tributyl tin, it should be satisfactory for most formulations. In this connection, the question of tetrabutyl tin and triphenyl tin was raised. This may have to be addressed on a case basis.

(3) It was agreed that the elimination of natural seawater and use of synthetic seawater from initial immersion to the end of the test should eliminate the concerns on possible non-uniform formation of bio-slimes.

(4) It was agreed to modify the procedure to increase the number of early data points. EPA agreed to develop recommendations for consideration by the task group.

Birnbaum announced a tentative meeting on January 30th in Arlington, VA (Crystal City) to develop a Draft #4 incorporating the items discussed at this meeting and input from EPA (Vlier and Reiter), Long-

more (Hempel), Schatzberg (NSRDC), Beiter (M&T), Blair (NBS), Dowd (NAVSEA), Gibbons (International), Seligman (NOCS) and the acting chairman.

*D01.45 Meeting*—S. Bailey reported for F-25.94 on the Navy Documentation Program. This committee has 140 working documents in review. A small number, possibly 12 are paint related. Help is needed to review these methods.

R. J. Martell, SSPC liaison, reported that the Navy wants to replace the wash primer specification with comparable SSPC standards. This work is in progress and SSPC will issue Paint Standard 27.

T. Foster, Canadian Navy, has been appointed chairman of D01.45.05 on Algae Control and will pick up where D. Stevens left off.

## **SUBCOMMITTEE D01.46 INDUSTRIAL PROTECTIVE PAINTING**

**K.A. Trimber, Chairman**

*D01.46.02, Surface Preparation.* K.A. Trimber, Chairman, discussed a first draft of "Method for Conductometric Analysis of Water-Soluble Ionic Contamination on Blasting Abrasives." It was agreed there is a need for the development of a test method for determining the cleanliness of abrasives used for blast cleaning. Recommended changes will be incorporated into Draft 2, which will be circulated at the next meeting.

*D01.46.03, Repainting.* R. Wakefield, Chairman, reported that Draft 5 of "Standard Methods for Field Identification of Coatings" was included in the Fall, 1985 D-1 ballot. Two negatives (Smith and Wilson) were found to be persuasive. Both were withdrawn based on editorial changes. Comments were received from H.E. Ashton. Because both the negatives and the comments were minor and editorial, the revised document is being submitted to Society ballot.

Future work of this task group will include a standard method for evaluating coating repair materials through the application of test patches. Tentatively, the document will include the requirements for test patch applications and performance evaluations. Comments and suggestions for this standard are to be sent to R. Wakefield. A draft will be prepared prior to the next meeting.

Other future work may include a method for microscopic evaluation of coatings. Suggestions for other work are also solicited.

*D01.46.04, Pull-Off Adhesion.* A. Cunningham, Chairman, reported that the new "Standard Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers" has completed all balloting and has been designated D 4541-85.

The results of round-robin studies to establish a precision statement were reported. Two different coating systems (latex/alkyd and alkyd/alkyd) were circulated to laboratories for testing using both the Elcometer Model 106 and Fisher System 2000. It was believed that these systems would possess adhesion strengths less than 1000 psi. Four laboratories (six operators total) used the Elcometer and three laboratories (three operators) used the Fisher 2000. The data to date shows:

(1) The latex/alkyd system possessed values greater than 1000 psi (greater than the capacity of the Elcometer Tester) on some of the pulls. The alkyd/alkyd system was consistently less than 1000 psi as desired.

(2) The Fisher 2000 unit consistently showed values higher than the Elcometer (generally two to three times).

(3) A wide variation in test results occurred. It was reported that the data scatter was typical of that found in the past using laboratory tensile testers, such as Instron.

(4) No significant differences in results were found between scoring and not scoring around the test dolly. Therefore, scoring does not appear to be beneficial.

An Instron is to be used as a referee for the test panels. This work will be accomplished by H.E. Ashton and M.E. McKnight. The Instron result is to be submitted to A. Cunningham by March 1. Based on the data, a revised precision statement will be circulated for subcommittee comment in early April. The target for balloting the precision statement is fall, 1986.

*D01.46.07, Inspection*, R.J. Martell, Chairman, reported that Draft 8 of the proposed revision to D 3276, "Standard Guide for Painting Inspectors (Metal Substrates)," had been submitted to concurrent Sub. D01.46/D-1 ballot. Two negatives (Lewis and Smith) were received, found persuasive, and withdrawn based on editorial changes. Comments from Moore, Jenkins, and Ashton also resulted in editorial changes. The editorially revised document will be submitted to Society ballot.

*D01.46.10, Condition Assessment*, M.E. McKnight, Chairman, reviewed a proposed draft of a practice for assessing the condition of aged coatings on steel structures, and of the surface of the substrate. Recommended changes will be incorporated into a revised document (Draft 2) which will be circulated for subcommittee comments prior to the next meeting.

*D01.46.11, Zinc-Rich Coatings*, R.H. Wakefield, Chairman, reported that the results were affirmative on a Sub. D01.46 ballot of a proposed revision to D 2092, "Standard Practices for Preparation of Zinc-Coated (Galvanize) Steel Surfaces for Painting." Three negatives (Smith, Wilson, Applemen), along with comments,

will result in editorial changes. The revised copy (Draft 2) will be submitted to D-1 ballot.

Draft 2 of proposed "Test Method for Measuring the Degree of Cure of Ethyl Silicate (Inorganic) Zinc-Rich Primers by Solvent Rub" was discussed. Changes will be incorporated into Draft 3 and circulated for Sub. D01.46 comments.

## **SUBCOMMITTEE D01.47 MASONRY TREATMENTS**

### **R.S. Shane, Chairman**

The chairman announced that Prof. Norman Weiss was unavoidably detained in New York. He sent his regrets and asked for volunteers for his task group on "permeability of Masonry Walls". He plans to call a meeting in the late spring in New York or Philadelphia and make a progress report in June at the Louisville meeting. The present members of the task group are: N. Weiss (Chairman), F. Gale, J.W. Ribar, S. Sherwood, and R.S. Shane (Ex-officio).

Susan Sherwood distributed a recent publication, "A Glossary of Historic Masonry Deterioration Problems and Preservation Treatments." Additional copies are available from her at the U.S. National Park Service.

A request was made by D.H. Ruddick of the N.J. Zinc Co. and Dr. Victoria Scarborough for establishment of a task force to develop a standard "Composition for Laboratory Testing of Effect of Acid Rain on Masonry." Ruddick was named chairman. Other members are: F.G. Hale, J.W. Ribar, V. Scarborough, T.J. Sliva, H.R. Trechsel, and R.S. Shane (Ex-officio). This was approved. It was suggested that other contacts be made: James Gibson, Colorado State University, and Gary Stenslyod, Illinois Water Survey, Champaign, IL.

Another task group was authorized on Masonry Water Seals with the following members: T.J. Sliva (Chairman), J.W. Ribar, V. Scarborough, and R.S. Shane (Ex-officio). Others interested in joining should contact Shane or Sliva.

J.W. Ribar, Construction Technology Laboratories, Portland Cement Association, gave a "penetrating" analysis of water penetration of a brick and mortar wall. He made the following points:

(1) Two ASTM tests, E 514, "Test Method for Water Permeance of Masonry," and C 67, "Method of Sampling and Testing Brick and Structural Clay Tile," are used to measure penetration of mortar and of brick, respectively. The larger the sample, the more accurate the results.

(2) The work of the Masonry Research Foundation at Clemson University should be consulted.

(3) Leakage always occurs at interfaces.

(4) The best bond is usually in the center of the wall.

(5) The best mortar will always absorb some water.

(6) Soft bricks soak up water about as fast as mortar, but lose the water rapidly.

(7) Materials' test properties are much less important, relative to water permeation, than the skill of the mechanic who installed the wall.

(8) He would like to try the colloidal silica treatment described by R.S. Shane. He was promised a one-quart sample for evaluation.

(9) The masonry industry avoids coatings of any kind, except as a last resort.

(10) The masonry design must complement any coating.

H.E. Ashton noted that a major masonry problem is air leakage. He thought that the design should try to avoid this. Ashton and Ribar agreed that time before testing is a function of sample size. Old walls and new walls give quite different values.

In response to a question by S. Sherwood, Ribar stated that lime-cement mortar should not be evaluated in less than six months. Replacement mortar should be based on an analysis of the formulation of the inside mortar.

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## **DIVISION 50 PAINTS FOR FACTORY APPLICATION**

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### **SUBCOMMITTEE D01.52 FACTORY-COATED WOOD BUILDING PRODUCTS**

#### **S.B. Schroeder, Chairman**

The negative votes on withdrawal of D 3719, "Test Method for Dirt Collection on Coated Exterior Panels," and D 2921, "Method for Qualitative Tests for the Presence of Water Repellents and Preservatives in Wood," were resolved by removing them from balloting for further study.

Revisions and additions suggested to several other methods were accepted and approved for Sub. D01.52 ballot.

In accordance with a previous persuasive negative by H.E. Ashton, combination of D 2793, "Standard Practice for Block Resistance of Organic Coatings on Wood Substrates," with D 3003, "Standard Test Method for Pressure Mottling and Blocking Resistance of Organic Coatings on Metal Substrates," was dis-

cluded with Sub. D01.53 members. It was agreed that a rewritten D 3003, which incorporated language to include wood substrates will be mailed to members of both Sub. D01.52 and Sub. D01.53 for comment before balloting.

It was agreed that D 2830, "Standard Method for Durability and Compatibility of Factory-Primed Wood Products with Representative Finish Coats," will be discussed with technical committee members of the American Hardboard Association at their April meeting and their assistance requested for its review.

The chairman volunteered to review D 4144, "Standard Method for Estimating Package Stability of Coatings for Ultra-Violet Curing," with experts in this area within his company.

*D01.52B, Hardboard.* S.B. Schroeder, Chairman, discussed a tentative agenda and plans for a joint meeting of this task group and Sub. D01.52.13 with the technical committee of the American Hardboard Association at their annual meeting in Palm Springs in April. Renewed activity on new test methods for surfactant resistance of primed hardboard siding is anticipated.

*D01.52.13, Prefinished Siding.* K. Kruse, Chairman, This meeting continued the discussion of the planned joint meeting with AHA. Consideration of the DSET Lab "EMMAQUA" test and other accelerated weathering methods will resume at that time.

## **SUBCOMMITTEE D01.55 FACTORY-APPLIED COATINGS ON PREFORMED PRODUCTS**

### **J.M. Behrle, Acting Chairman**

Sub. D01.55.07 reported three negative votes on the recently balloted "Guide for Testing Industrial Water-Reducible Coatings." On motion, it was agreed to withdraw the guide from main balloting, resolve the negatives, and revise it accordingly for resubmission to Sub. D01.55 ballot.

Sub. D01.55 organizational changes were discussed. It was suggested that if Sub. D01.55 functions that applied to wood products were moved into Sub. D01.52, it might result in the strengthening of both subcommittees. Similarly, the incorporation of Sub. D01.55 functions pertaining to metal products into Sub. D01.53, might be mutually beneficial. Sub. D01.55 could then be disbanded.

A strategy was discussed to develop increased participation in Sub. D01.55 activities by both end users and coatings manufacturers. Specific contacts will be made regarding potential new member participation for the 1986 summer meeting.

## **SUBCOMMITTEE D01.56 PRINTING INKS**

### **J.M. Fetsko, Chairman**

*D01.56.01, Fineness of Grind.* J. Cichon, Chairman, conducted a review of the revised text for D 1316, "Test for Fineness of Grind of Printing Inks by the Production Grindometer." In accordance with current usage, the name of the grind gage will be changed to "NPIRI Grindometer." It was also suggested that end-points be read to the nearest 0.25 gage unit and mean values reported to the nearest 0.50 gage unit.

*D01.56.02, Lightfastness of Prints.* J. Daugherty, Chairman, distributed comparative results showing ratings of 16 test prints by indoor exposure in various laboratories. Analysis of results requires more information on the type of lighting and distance from the print. South Florida Testing Service offered to expose the prints to sun under glass in Florida and Arizona.

*D01.56.04, Viscosity of Paste Inks.* J.M. Fetsko, Chairman, discussed proposed changes in conjunction with the scheduled five-year review of D 4040, "Viscosity of Printing Inks by the Falling Rod Viscometer." Besides incorporating methods for obtaining various parameters of non-Newtonianism, it was suggested that the method give priority treatment to calculation rather than graphical solutions of viscosity.

*D01.56.06, Ink Tack.* J.M. Fetsko, Chairman Pro-tem, reviewed proposed revisions to "Apparent Tack of Printing Inks by the Inkometer." Task group members agreed to let experienced technicians in their laboratories gage the relative merits of the original and revised instructions. The group will review the comments prior to the next meeting.

*D01.56.09, Tinting Strength.* W. Rhodes, Chairman, reported that a few language revisions have been received for the proposed new test method, "Relative Color and Strength of Printing Inks by Visual and Instrumental Evaluation." After repeatability and reproducibility are calculated, the method will be submitted to Sub. D01.56 ballot. The instrumental procedure now permits reflectance readings with a complimentary filter. The utility of densitometer readings is being explored in a cooperative study between GATF and BASF.

*D01.56.10, Water Pickup of Litho Inks.* G. Bien, Chairman, reviewed the statistical analysis of round-robin data conducted by J. Cichon. A member pointed out that the single point test results of some inks are highly sensitive to quantity of water added prior to mixing.

A paragraph should be inserted into the method suggesting that the rate of water addition be based on preliminary testing.

*D01.56.11, Nonvolatile Content of Printing Inks.* B. Blom, Chairman, reported that the D-1 ballot on this method received a number of comments plus one negative vote because the precision statement was based on triplicate determinations. The negative will not be contested because a new round-robin has already been conducted according to the guidelines set forth in D 3980, "Practice for Interlaboratory Testing of Paint and Related Materials."

*D01.56.12, Viscosity of Liquid Inks.* J. Cichon, Chairman, reported that he has been collecting information on methods to improve reproducibility of the Zahn cup. J. Peters, chairman of Sub. D01.24.19 on Viscosity by Efflux Cups, was in attendance and suggested that joint sessions of the two task groups be scheduled at future meetings.

*D01.56.13, Ink Mileage.* B. Blom, Chairman, reported that a round-robin on ink mileage using pairs of unemulsified and emulsified inks exhibited poor between-lab reproducibility. Besides problems due to discrepancies between visual and densitometer-based matches, a recent technical article indicated that it was not possible to match a production print on a laboratory press with or without water. In this case, a mileage test must specify a reference ink instead of a reference print. The prints of the round-robin will be circulated to participants.

*D01.56.14, Setting of Heatset Inks.* A. Urdea, Chairman, distributed copies of a proposed procedure for evaluating relative setting of inks in the Silvaco Tester. J.M. Fetsko agreed to check out the instructions if provided with two inks. The cooperation of Sinclair & Valentine will be pursued.

*D01.56.17, Practice for Printing Inks.* A. Scarlatti, Chairman, distributed a list of ASTM methods that are applicable to printing inks. The task group members will make an independent search to ensure that all applicable methods are included.

## **SUBCOMMITTEE D01.57 ARTISTS' PAINTS**

### **J.T. Luke, Chairman**

*D01.57.01, Preparation of Samples for Colorimetric Determination.* T. Pamer, Chairman, has been working to develop a method for drawdowns of artists' oil and acrylic paste paints. In previous tests conducted by H.W. Levison, complete hiding was obtained in most oil color



drawdowns, but not in many of the acrylic drawdowns. Previous results will be reviewed and a round-robin will be conducted using templates previously passed out by I. Shack. Arylide yellow light acrylic and alizarin crimson oil color paints will be used in the round-robin. This proposed practice could possibly be used in the relative tinting strength test method presently being worked upon.

*D01.57.02, Lightfastness of Pigments*, reported that a task group consisting of P. Staples and J. Scott has been working to see if a recovery period in room light is needed after the exposed specimens have been shipped back to the lab before instrumental readings for color are made. A test was made and Staples shipped five alkyd specimens to Scott who measured them with a Minolta Color Difference Meter, then exposed them for 24 hours to a radiant energy source, measured them again, stored them in the dark, and then measured them periodically for two months to record the color change in the dark.

The test was criticized because (1) negative signs were left out of the report, making it difficult to tell whether the specimens were yellowing or bleaching; (2) since the first measurement was made immediately after unpacking the specimens following shipment from overseas, and all subsequent measurements compared to that first measurement, some of the first color change could be due to bleaching of the yellowing that occurred during shipment; then as storage in the dark proceeded there may have been gradual yellowing; (3) F. Billmeyer expressed concern that the erratic changes shown in the data may indicate that the Minolta is not precise enough over time to give reliable color differences throughout a two-month period. J. Scott volunteered to redo the test to answer the criticisms.

A number of editorial changes were approved to the revision of D 4303, "Test Methods for Relative Lightfastness of Pigments used in Artists' Paints." Two other revisions were also decided upon as follows:

8.4.5—Add: "For oil or alkyd paints an initial amount of 40 g of white paint is sufficient if specimens are read wet and recoverable paint is returned to the batch."

8.5.4.—Add: "Allow alkyd paints to dry for three days between coats and five days after the second coat before the first instrumental reading."

*D01.57.03, Tinting Strength*, T. Pamer, Chairman, distributed for comment the proposed "Standard Test Method for Relative Tinting Strength," which was written in ASTM format. The only correction recommended was on page 4—#8, in which the last sentence was

changed to read "Dry samples thoroughly using an overnight air dry." Pamer reported there still is a problem because the earth colors do not show a linear relationship between tinting strength and concentration. A factor which raises the K/S by some power may be necessary for the earth colors.

T. Pamer requested that the test method be tried on some strong colors such as cadmiums by people who have access to the necessary equipment and report back by the June meeting.

*D01.57.04, Specification for Artists' Paints*, A. Spizzo, Chairman, made several editorial changes in the revision of D 4302, "Specification for Artists' Oil and Acrylic Emulsion Paints," to include alkyd paints.

Suggestions were presented by H. Mueller and J.T. Luke for a section to be added to D 4302 covering resin-oil paints. Most paragraphs simply add mention of resin-oil paints to the paints covered in the standard. Proposed 6.7 would require that the nonvolatile content of an artists' resin-oil paint be not less than 90% of the average of a line.

It was decided to drop this requirement as not being as important as developing a re-solubility test. Since the re-solubility of the paint film when a top coating of varnish is removed is a major problem in conservation of paintings and since some resin-oil paints might be more soluble than oil paints, it was decided that a re-solubility test should be specified before the resin-oil section is added to D 4302. The chairman appointed a task group, including H. Mueller as chairman and J. Swency, to work on a test method to determine the re-solubility of dried resin-oil paint films.

It was agreed that earth colors, previously excluded from lightfastness testing, also be excluded in lightfastness testing of pigments in the resin-oil vehicle.

*D01.57.08, Labeling for Toxicity*, J.T. Luke, Chairman, reported that even though the subcommittee voted to leave Section 3.7 (present 3.6), Statement of Conformance, as it is, the task group has recommended that the following conformance statement be added to 3.7: "The purpose of the conformance statement is to inform the purchaser, at the time of purchase, of the product's compliance with the standard. To accomplish this purpose, the conformance statement should appear whenever practical, on the product, however, it shall also be acceptable to place the statement on one more of the following: (a) individual product package, (b) a display or sign at the point of purchase, (c) separate explanatory literature available on request at the point of purchase, and (d) a response to a formal request for bid or proposal.

There was discussion on Section 1.3 which states that this practice applies to

developing precautionary labeling for art materials intended for adult usage. E. Flax suggested that consideration be given to having D 4236 cover both adult and children's art materials. The chairman will consult with Dr. Stopford and the Art & Craft Materials Institute on this issue.

The task group then considered the request from Sub. C-21.08 on Standards of Ceramic Materials for Artists and Hobbyists to include "Guidelines for the Safe Use of Hobby Ceramic Art Materials" (C 1023-84) in the appendix of D 4236. This guideline was written by Sub. C-21.08 with advice from Dr. Stopford. It was the consensus of the task group that C 1023-84 should not be added to the Appendix.

There was a discussion as to whether additional precautionary statements are needed in A.2 of the Annexes. Such statements referring to children below certain grade school levels and unsupervised children below certain grade levels were discussed. The chairman will discuss these statements with Dr. Stopford, but it was decided not to add any phrases at this time.

*D01.57.09, Watercolors*, E.T. Vonderbrink, Chairman, requested at the last meeting that a list of proposed pigments that should be tested be submitted to E.T. Vonderbrink. A list of all the pigments received was circulated and a final request for additional pigments was made. The final list should be completed by February. Manufacturers will be contacted to supply samples for testing. Samples will be drawn down on filter paper at 40% reflectance at maximum absorption for testing.

*D01.57.10, Consumer Evaluation*, H. Brown, Chairman, reported the results of the vote on "Lightfastness Categories for the Blue Wool Standards Test." The categories receiving the most first choice votes were: lightfast, moderately lightfast, and fugitive. The number of people needed to evaluate the exposed specimens was three.

The draft document on the Blue Wool Test: Version I, was discussed. It was suggested that this document be called "Standard Practice for Visual Determination of Relative Lightfastness of Artists' Materials." It was suggested that definitions describing the kinds of products to be tested be eliminated from the draft. The background and work done so far in developing the proposed practice were discussed. H. Brown will prepare a second draft of the document and send it to the subcommittee members for review.

*D01.57*—Approval was given to sending I. Shack a letter of thanks from ASTM for his previous work on this subcommittee.

The subcommittee also took action on the recommendations made by the task groups relative to D 4236, D 4302, and D 4303. The detail will be found in the subcommittee minutes.

# Society Meetings

## BIRMINGHAM . . . . . JAN.

### "Electrostatic Powder Coating"

The technical presentation was given by J.F. Hughes, of the University of Southampton. Dr. Hughes addressed the topic of "ELECTROSTATIC POWDER COATING, AN ART OR A SCIENCE?"

A discussion on the history of electrostatics and the manipulation of solid particles commenced the program. Dr. Hughes explained that first evidence of electrostatics appears in ancient Greek history as mysterious effects were noted when contact was made between two different materials, such as cat fur standing on end when rubbed with an amber rod. Later evidence is found in the recording of a discharge of light from mast tops (corona discharge). Although the phenomenon was not understood, there is evidence of precautions being taken in wooden ships by those handling gunpowder, he explained.

Today, there are four methods of charging, stated Dr. Hughes. They include tribo, corona, induction, and radio active. Elaborating on the four types, the speaker noted that tribo and corona are the methods used in powder coating. Showing slides, he detailed their individual advantages and disadvantages.

Dr. Hughes included a discussion on the phenomenon of self limiting as it pertains to the behavior of positive and negative ions at the point of application. The problems of back ionization are being studied at Southampton, he stated.

In conclusion, Dr. Hughes listed other applications of basically the same process as powder coatings, including: dots on the mail, pollination of palms, confectionary, and photographic prints. Referring to the prints of photographs, he suggested that the scientific knowledge and working technology of electrostatic deposition has produced a work of art.

DAVID M. HEATH, *Secretary*

## BIRMINGHAM . . . . . FEB.

### "Titanium Dioxide"

Club President-Elect Graham Fowkes reported that the 1986-87 program has been completed except for details of lecture titles. He noted that several companies have offered to provide lectures for next year.

Technical Officer John N. Hitchin, of W. Canning Materials Ltd., reported that two out of the four lecturers scheduled for the

Club's Symposium "Miracle 86" to be held May 22, 1986, had offered synopses. The draft program will soon be available for printing, he stated.

Certificates of Merit, to be presented to the original members of the CAT Sub-Committee, had been received from Federation Headquarters, reported Mr. Hitchin. These will be presented by Sub-Committee Chairman Brian J. Addenbrook, Croda Paints Ltd., at the March meeting:

Mr. Hitchin also reported that no nominations had yet been received for the 1986-87 Technical Committee Chairman post. He stated that every member of the Committee was asked to consider names for the position.

Secretary David M. Heath, of Holden Surface Coatings Ltd., tabled a number of collated amendments and suggested amendments to the By-Laws, so that all necessary amendments could be incorporated into the next edition of the By-Laws booklet. The amendments concern: Article V—Election to Membership; Article VI—Subscriptions; Article IX—Organization; and Article XI—Duties of Officers and Committees.

Club President J. Ron Jukes, of Croda Paints Ltd., suggested that consideration be given to an internally distributed and audited questionnaire asking members attending meetings for their views on the standard of lectures and the meeting facilities.

"TITANIUM DIOXIDE—WORLD SUPPLY AND DEMAND IN THE 80'S" was the title of the evening's technical presentation given by G. Deighton, of SCM Chemicals.

Mr. Deighton began his presentation showing slides to illustrate where  $TiO_2$  is distributed and who makes it. The world market is 2.5 million tons, he explained. In 1970, world demand was 1.6 million tons and is expected to reach 2.8 million tons by 1990. He noted that a significant drop in demand occurred during 1973-75 due to the oil crisis and the resulting down-turn in industrial activity. Another down-turn in demand was measured in 1979-80, and again was due to the oil crisis. Since 1982, the  $TiO_2$  market has sustained recovery growth. In 1983, demand was up 14%.

The speaker closed with a discussion on reasons for the present position of difficult supply and steeply rising prices. Environmental problems plus a lack of profitability during the 1970's had caused a reduction in overall capacity, he concluded.

DAVID M. HEATH, *Secretary*

## CDIC . . . . . FEB.

### "Water-Borne Coatings"

Al Heitkamp, of Cargill, Inc., delivered an informative talk on "CORROSION RESISTANT WATER-BORNE COATINGS."

Mr. Heitkamp elaborated on selection of both pigments and resins, anti-corrosive additives, and dryers. He stressed that these were the major areas to consider when formulating water-borne coatings.

The second presentation of the evening was given by two representatives of Wright Patterson Air Force Base. The speakers provided an interesting debate on "STRENGTH AND TECHNOLOGICAL ADVANCES OF U.S. AND SOVIET AIR FORCES."

SAM KRATZER, *Secretary*

## CLEVELAND . . . . . FEB.

### "Polymer Science Lab Tour"

A special tour of several polymer science laboratories at Case Western Reserve University served as the program of the February meeting. Participants viewed the university's labs and received a detailed presentation on the facilities and equipment

## FSCT Membership Anniversaries

### 50-YEAR MEMBERS

#### Chicago

Carroll M. Scholle, Retired  
A.L. Jordan, Jordan Paint  
Manufacturing Co.

### 25-YEAR MEMBERS

#### Birmingham

Cyril V. Lawton, Evode Limited

#### Piedmont

Ernest L. Broome, Prillaman Co.  
Robert K. Kitson, Blue Ridge Talc Co.  
John Nielsen, Premium Coatings, Inc.  
Ralph A. Williams, Consultant.  
Cecil H. Wilson, Burks, Inc.

available. The Major Analytical Instruments Facility (MAIF), which houses state-of-the-art instrumentation in nuclear magnetic resonance, electron paramagnetic resonance, vibrational spectroscopy, and ion bombardment spectroscopy was toured, as was the Solid State Electronics Laboratory, and Polymer Micro-device Laboratory. The services of MAIF are available to private concerns.

Also included on the tour was the Macromolecular Science Building where those attending viewed the Newpher Composite Processing Laboratory, Electron Microscopy Laboratory, Instron Laboratory, Molecular Spectroscopy Laboratory, Research Computing Laboratory, and the X-Ray Structure Analysis Laboratory.

RICHARD R. ELEY, *Secretary*

## KANSAS CITY . . . . . JAN.

### "Extender Pigment Blends"

Society Treasurer Roger Haines, of Tnemec Co., Inc., reported for the Technical Committee. Hardboard panels were due in for a five year check, he stated. The Committee had originally planned on presenting a paper at the 1986 Annual Meeting, however, due to insufficient data, the paper will not be ready at that time. A five year follow-up report may be presented at the joint meeting with St. Louis, noted Mr. Haines.

It was announced that the joint meeting with St. Louis Society would be held June 7, 1986. Proposals for joint sponsorship of an annual joint meeting have been placed before the Executive Committee of both Societies.

Using a slide presentation, Dan Dixon, of Englehard Corp., detailed "A NEW APPROACH TO THE EVALUATION OF VARIOUS EXTENDER PIGMENT BLENDS."

STEVEN D. JOHNSON, *Secretary*

## LOS ANGELES . . . . . FEB.

### "High Occupancy Vehicle Lanes"

Society Treasurer Ray DiMaio, of Koppers Co., reported on the activities of the Society's Manufacturing Committee. The annual seminar will be held April 9, 1986, and focuses on "Plant Costs Control." The seminar will encompass employee motivation, OSHA training, Right-to-Know Law, safety and worker's compensation, inventory control, and warehousing and material safety.

W.T. Maloney, of California Department of Transportation, spoke on "HIGH OCCUPANCY VEHICLE LANES."

Traffic control, speed enforcement, and car pooling were some of the topics covered in Mr. Maloney's presentation.

MELINDA K. RUTLEDGE, *Secretary*

## LOUISVILLE . . . . . FEB.

### "Waste Management"

Southern Society member Mel Penn, of Kerr-McGee Chemical Corp., distributed notices of the Society's Annual Meeting, slated for April 2-4, 1986, in Savannah, GA.

Technical Committee Chairman Louis Holzknecht, of Devco Marine Coatings, reported on the group's projects. Rick Irizarry of Reliance Universal, Inc., has completed the literature research on the project "Effect of Catalysts on Urethanes," stated Mr. Holzknecht. He also told of the Federation's project on Mildew Research where Stan Harshfield, of Devco Reynolds, will evaluate the panels at Celanese Specialty Resins Co.

Joe Durezynski, of Systech Corporation, provided the technical talk of the evening. Mr. Durezynski discussed "WASTE MANAGEMENT."

Services provided by waste management firms for the disposal of combustible waste using cement kilns and disposal facilities were described by the speaker. He noted that EPA licensed facilities to burn these wastes at temperatures of 2800 to 3000°F, providing for safe and environmentally sound disposal medium.

LARRY FITCHFORD, *Secretary*

## NEW ENGLAND . . . . . FEB.

### "EB and UV Curing"

Program Committee Chairperson, Maureen Lein, of Davidson Rubber Co., reported that the program for the upcoming symposium "Launching the New Revolution — Compliance for the 21st Century," on May 15-16, was ready and is being mailed to the membership.

Society Representative, Dan Toombs, of DN Lukens Co., reported on the discussions of the Federation Executive Committee meeting, which he attended in Philadelphia.

It was announced that Kevin Mulkern, of Samuel Cabot, Inc., and Gail Pollano, of Polyvinyl Chemical Ind., would be attending the Federation's Spring Week in Pittsburgh.

Alice Pincus, of Pincus Associates, provided a technical talk on "EB AND UV CURING — HOW, WHAT, AND WHERE?"

A definition of radiation curing started the presentation. Ms. Pincus described it as the curing or crosslinking of specially formulated materials onto various substrates by use of a radiation source, such as UV lamps or electron beams (EB). She noted that it is a specialty process and requires special equipment.

Next, Ms. Pincus discussed the types of equipment used in radiation curing, including scanning type and curtain type. For UV curing systems she mentioned five pieces of equipment: UV lamps, lamp housings, voltage stabilizers, a control console, and safety hardware.

The chemistry of radiation was the next area explored by the speaker. The formulation consists of monomers or reactive diluents, oligomers, pigments, photoinitiators (required for UV, not EB curing systems), and other additives. She added that UV curing systems cure by free radical polymerization. Further, she explained that multifunctional monomers, crosslinking types, usually contain two or more reactive sites and the diluent types are monofunctional and are used to decrease viscosity. The oligomers are low to medium molecular weight and provide adhesion and toughness and other properties. Additives must be carefully selected so that they do not prevent the penetration of UV rays, noted the speaker.

Ms. Pincus then identified applications of radiation curing. In the coatings field, radiation curing is described by the substrate that is being coated, such as wood, metal, or plastic. In the ink area, radiation curing is described by the process such as offset, screen, flexo, or gravure. In the adhesive area, it is described by the function, such as pressure sensitive.

Her presentation concluded with a slide show identifying some products where UV and EB curing was used. For UV curing, she included bumper stickers, yogurt cups, signature strips on credit cards, and certain beer cans. For EB, she identified tanning blankets, floppy discs, release coatings mainly in the medical field, and high quality shopping bags.

*Q. What are the EPA requirements for producing a coating for EB curing?*

A. Specifically, handling of raw materials. Most are skin sensitizers. Some monomers of the acrylate type require the use of gloves and goggles.

GAIL POLLANO, *Secretary*

## PHILADELPHIA . . . . . FEB.

### "Titanium Dioxide"

Society President Phil Reitano, of Inolex Chemical Co., reported that Robert Ziegler has been appointed Executive Secretary to the Federation.

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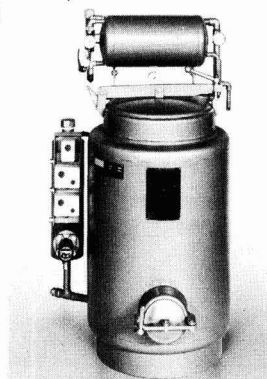
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Mr. Reitano also noted that EPA office has videotapes on solid and hazardous waste. They may be ordered in small quantities from the EPA at 1-800-368-5888.

Technical Committee Chairman William Fabiny, of Sermatech International, Inc., confirmed that the keynote speaker for the Society spring technical seminar will be Honorable James Florio D-NJ. "The Impact of Environmental Regulations and the Right-to-Know on the Coatings Industry" is the title of the seminar, he added, and it will be held May 12, 1986, at the Airport Hilton, in Philadelphia, PA.

Bob Sonntag, of Superior Varnish & Drier Co., reported that the Technical Committee project on HLB Surfactants can be ready for the Annual Meeting provided the membership participate.

The technical speaker was R.I. Ensminger, of NL Chemicals/NL Industries, Inc. He discussed "TITANIUM DIOXIDE—WHY SO MANY GRADES?"

Mr. Ensminger began by dividing titanium dioxide into three broad categories used by the coatings industry: rutile, of which there are 50 grades; anatase, offered in 16 grades; and rutile slurries, available in 12 grades. He mentioned that, for industries such as plastics, paper, synthetic fibers, ceramics, and food-drug/cosmetics, other grades are available.

Next, the speaker discussed the two processes used to manufacture titanium-dioxide pigments. They are sulfate and chloride. He noted that the sulfate process is the oldest. He explained that the actual dioxide content (purity) is not the only factor influencing performance.

An explanation of the inorganic treatment of titanium dioxide followed. Mr. Ensminger divided the inorganic treatment into three classes: untreated, no treatment intentionally added which includes grades for ceramics, glass, and paper; alumina treated, general purpose grades used for paint and plastics; and alumina and silica treated, used for chalk resistant and latex flat paint grades.

In addition to inorganic treatments, many manufacturers will add organic treatments to alter the surface of the pigment and make it more compatible with the organic liquid. He then enumerated on the types of organic treatments which include polymeric hydrocarbons, vegetable oil acids, naphthenates, phthalates, silicon polymers, and amines.

With respect to performance, Mr. Ensminger stated that, for the user, the organic treatment should provide better wetting and dispersing. He also explained that, as the PVC is increased, the optical efficiency of titanium dioxide is reduced. Maximum hiding with titanium dioxide as a single pigment is 30% PVC, he stated. To space

the TiO<sub>2</sub>, extenders are used. This process is used in latex flat paint, he noted. Building a spacer on the pigment particle is another way of improving the titanium dioxide efficiency. Chalking resistance can be improved by treatment on the pigment surface. He explained that this prevents TiO<sub>2</sub> from acting as a photochemical catalyst which would normally degrade the binder.

In conclusion, Mr. Ensminger said that the question of "why so many grades of titanium dioxide?" is answered by the fact that titanium dioxide is used in a wide variety of end use applications. Each application has its own specific requirements."

THOMAS L. PETA, *Secretary*

to hazard communications, he explained that the Occupational Safety and Health Administration sets the rules for communicating these hazards to customers and employees.

Mr. Elliott concluded by stating that future formulations may change due to government relations, but hopefully these changes will be made by the coatings chemist based on knowledge of what is going on in the country today.

MARK D. TROUTMAN, *Secretary*

## PITTSBURGH . . . . . FEB.

### "Cationic Electrodeposition Primers"

Reporting on behalf of the Society Education Committee, Clifford Schoff, of PPG Industries, Inc., told of a presentation to be made at Saint Vincent College on February 7, 1986. He noted that Indiana University of PA, Washington & Jefferson College, and Chatham College are all interested in having presentations in the spring. Indiana is also interested in the possibility of a Society-sponsored graduate or undergraduate project. He explained that the Committee had also been notified from the Federation that monies used to assist a project of this type could possibly be matched with Federation funding.

Society President Joe Mascia, of Campbell Chemical Co., reported that there was information available on 1986 short courses being offered at the University of Missouri-Rolla. He also stated that the Federation has video tapes available on the requirements for small generators of hazardous wastes.

Ray Uhlig, of PPG Industries, Inc., chairing the committee handling Spring Week, informed members that all receptions, tours, and other events have been finalized. In addition, he reported that both the restaurant guide and souvenir photo to be distributed to participants, are either completed or near completion.

Clifford Schoff, of PPG Industries, Inc., delivered the technical presentation. Dr. Schoff described "CATIONIC ELECTRODEPOSITION PRIMERS OVER ZINC COATED STEEL."

Two kinds of electrodeposition were defined by the speaker: cathodic and anodic. He explained that many different polymers are used in electrodeposition. In cathodic, the three main types are epoxy, acrylic, and vinyl. These are made cationic by modification with amines. The most common amines are quaternary ammonium, quaternary phosphonium, and ternary sulfonium. He explained that the amines used with the resin are neutralized with an acid to form a salt.

## PITTSBURGH . . . . . JAN.

### "Government Relation of Paint Chemists"

Society President Joseph Mascia, of Campbell Chemical Co., announced that the new Society Representative is Ray Uhlig, of PPG Industries, Inc. Mr. Mascia then conveyed his appreciation for the fine contribution to the membership made by outgoing Representative Ed Vandevort, of PPG Industries.

James N. Elliott, of National Paint and Coatings Association, provided the technical talk on "THE GOVERNMENT RELATION OF PAINT CHEMISTS."

Mr. Elliott began by listing three areas of government relations. They are: influencing legislation (acting as an industry voice to legislators); influencing regulations (contacting the regulating agencies to be sure they are fair and enforceable); and interpreting the regulation (helping industry companies comply with the regulations).

Next, present issues which are in the foreground of the paint and coating industry's concerns were listed. Mr. Elliott included "SuperFund," Right-to-Know laws, product liability, solvent toxicity, methylene chloride, and hazards communications, in his list.

Elaborating on each item, Mr. Elliott stated that Congress is working on a bill to try and reauthorize the "SuperFund" law that was passed about five years ago. A law providing for uniform rules throughout the country on community Right-to-Know laws is also before Congress. Bills that could result in a federal product liability law are also being considered in Congress.

Further, stated Mr. Elliott, our government seems to be getting more and more interested in the areas of solvent toxicity to learn what might be the effect of chronic exposure to organic solvents, and methylene chloride when it was found to possibly cause cancer in animals. With respect

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## Constituent Society Meetings and Secretaries

**BALTIMORE** (Third Thursday—Martin's Market Square, Towson, MD). ED COUNTRYMAN, Bruning Paint Co., 601 S. Haven St., Baltimore, MD 21224. Virginia Section—Fourth Wednesday, Ramada Inn-East, Williamsburg, VA.

**BIRMINGHAM** (First Thursday—Strathallan Hotel, Birmingham, England). D.M. HEATH, Holden Surface Ctg. Ltd., Bordesley Green Rd., Birmingham B9 4TQ England

**CHICAGO** (First Monday—meeting sites vary). RAYMOND CZICZO, Reliance Universal, Inc., 1915 Industrial Ave., Zion, IL 60099.

**CDIC** (Second Monday—Sept., Jan., Apr., June in Columbus; Oct., Dec., Mar., May in Cincinnati; and Nov., Feb. in Dayton). SAMUEL KRATZER, Potter Paint Co., P.O. Box 265, Cambridge City, IN 47327.

**CLEVELAND** (Third Tuesday—meeting sites vary). RICHARD ELEY, Glidden Coatings & Resins, Div. of SCM Corp., D.P. Joyce Research Center, P.O. Box 8827, Strongsville, OH 44136.

**DALLAS** (Thursday following second Wednesday—Executive Inn, near Lovefield Airport). FREDERICK T. BEARD, Glidden Coating & Resins, Div. of SCM Corp., 1900 North Josey Ln., Carrollton, TX 75006.

**DETROIT** (Fourth Tuesday—meeting sites vary). JOANNE CEDERNA, Inmont Corp., 26701 Telegraph Rd., Southfield, MI 48086.

**GOLDEN GATE** (Monday before third Wednesday—Alternate between Sabela's Restaurant on Fisherman's Wharf and Francesco's in Oakland, CA). KARL SAUER, Pfizer, Inc., MPM Div., 776 Rosemont Rd., Oakland, CA 94610.

**HOUSTON** (Second Wednesday—Sonny Look's, Houston, TX). JAMES A. HARRELL, Buckman Laboratories, 5127 Wightman Ct., Houston, TX 77069.

**KANSAS CITY** (Second Thursday—Cascone's Restaurant, Kansas City, MO). STEVEN JOHNSON, Cook Paint & Varnish Co., P.O. Box 389, Kansas City, MO 64141.

**LOS ANGELES** (Second Wednesday—Steven's Steak House, Commerce, CA). MELINDA RUTLEDGE, Allo Chemical Co., P.O. Box 443, Ontario, CA 91761.

**LOUISVILLE** (Third Wednesday—Breckinridge Inn, Louisville, KY). LARRY F. PITCHFORD, Reynolds Metals Co., P.O. Box 3530, Plant III, Louisville, KY 40232.

**MEXICO** (Fourth Thursday—meeting sites vary).

**MONTREAL** (First Wednesday—Bill Wong's Restaurant). W WILDE, Hoechst Canada, Inc., 4045 Cote Vertu, Montreal, Que., Canada H4R 1R6.

**NEW ENGLAND** (Third Thursday—LeChateau Restaurant, Waltham, MA). GAIL POLLANO, Polyvinyl Chemical Industries, Inc., 730 Main St., Wilmington, MA 01887.

**NEW YORK** (Second Tuesday—Landmark II, East Rutherford, NJ). JOHN W. BURLAGE, Pacific Anchor Chemical, 14 Ridgedale Ave., Cedar Knolls, NJ 07927.

**NORTHWESTERN** (Tuesday after first Monday—Jax Cafe, Minneapolis, MN). JOAN B. LAMBERG, Horton-Earl Co., 750 S. Plaza Dr., St. Paul, MN 55120.

**PACIFIC NORTHWEST** (Portland Section—Tuesday following second Wednesday; Seattle Section—the day after Portland; British Columbia Section—the day after Seattle). YVON POITRAS, General Paint Co., 950 Raymur Ave., Vancouver, B.C., V6A 3L5, Canada.

**PHILADELPHIA** (Second Thursday—Dugan's Restaurant, Philadelphia, PA). THOMAS L. PETA, J.C. Osborne Chemicals, Inc., P.O. Box 1310, Merchantville, NJ 08109.

**PIEDMONT** (Third Wednesday—Howard Johnson's, Brentwood Exit of I-85, High Point, NC). CHARLES HOWARD, DeSoto, Inc., P.O. Box 22105, Greensboro, NC 27420.

**PITTSBURGH** (First Monday—Montemurro's, Sharpsburg, PA). MARK TROUTMAN, Bradley Paint Co., 608 W. Crawford St., Connellsville, PA 15425.

**ROCKY MOUNTAIN** (Monday following first Wednesday—Bernard's, Arvada, CO). MARCY S. BAUGH, Hutson Industries, 60 Tejon St., Denver, CO 80223.

**ST. LOUIS** (Third Tuesday—Engineers Club). JAMES N. McDERBY, F.R. Hall & Co., 6300 Bartmer Ind. Dr., St. Louis, MO 63130.

**SOUTHERN** (Gulf Coast Section—Third Thursday; Central Florida Section—Third Thursday after first Monday; Atlanta Section—Third Thursday; Memphis Section bi-monthly on Second Tuesday; Miami Section—Tuesday prior to Central Florida Section—R. SCOTT MCKENZIE, Southern Coatings & Chemicals, P.O. Box 2688 Sumter, SC 29150.

**TORONTO** (Second Monday—Cambridge Motor Hotel). HANS WITTMAN, BASF Canada Ltd., 10 Constellation Ct., Rexdale, Ont., Canada M9W 1K1.

**WESTERN NEW YORK** (Third Tuesday—meeting sites vary). JEAN L. LUCK, Pratt & Lambert Inc., Powder Coatings Div., P.O. Box 22, Buffalo, NY 14240.

Next, Dr. Schoff focused on the actual deposition which is a physical agglomeration. He explained that, at the cathode, the pH of the bath is about 6.0 and the cathode itself has a pH of 12 or 13. The coating is actually destabilized and kicks out, and it is this kick-out that forms the coating. With the pH of the cathode being that high, problems can exist, he noted. An area is currently under study to find a means whereby the pH can be lowered while still producing the desired results.

Galvanized and other zinc coated steels were then discussed. Dr. Schoff explained that today there are zinc-iron alloys, iron-zinc, zinc nickel, and other zinc coated steels. In the U.S., the most common method is hot-dipped galvanized. In Europe, electrogalvanized metal is commercial. It will not be available in the U.S. until 1987.

The discussion then moved to Dr. Schoff's latest research which was fueled by the problems of cratering and pinholing at high voltages on some of the zinc coated steels. He stated that these problems can occur at voltages as low as 200 volts, a common voltage for entry into some electrodeposition tanks. His research has found that the cause of craters could be an electrical discharge or sparking in the bath. Sparking creates a problem since it causes a cure in the immediate area of the spark, he explained.

Dr. Schoff's research also shows that sparking may be due to gassing caused by the steam or heat coming off at the point of the spark.

The final area explored by Dr. Schoff was ripple effect. This is a direct current which has a tremendous amount of spikes in it caused when an alternating current is changed to direct current. To combat this problem, chokes or filters must be installed to change from a high ripple voltage to a low ripple voltage, he stated.

MARK D. TROUTMAN, *Secretary*

## WESTERN NEW YORK . . . . FEB.

### "Extender Pigment Blends"

Society President Charles C. Tabbi, of Spencer Kellogg Prod./NL Chemicals, reminded members of upcoming meetings. The May Society meeting is to be held at the Saratoga Restaurant on May 20, 1986, and will feature a visit from the Federation Officers. Richard Kemmerer, Celanese Chemical Co., will provide a talk on "Recent Advances in Radiation Coatings Technology."

Dan Dixon, of Englehard Corp., provided a technical talk on "A NEW APPROACH TO THE EVALUATION OF VARIOUS EXTENDER PIGMENT BLENDS."

JEAN L. LUCK, *Secretary*

# Elections

## BIRMINGHAM

### Active

PHIPPS, KENNETH J.—SCM Chemicals Ltd.,  
NR Brigg, S. Humberside, UK.

## C-D-I-C

### Active

FYRNY, EDWARD L.—Naval Avionics Cent.,  
Indianapolis, IN.

INDIHAR, JOSEPH F.—D&L Paint Co., Liberty,  
IN.

PATTERSON, JOSEPH R.—U.S.I. Chemicals Co.,  
Cincinnati, OH.

WELLS, BRUCE—Hanna Chemical Coatings  
Co., Columbus, OH.

### Associate

APPLGATE, TERRY—Midwest Color Techno,  
Columbus, OH.

MANDAT, MICHAEL J.—Man-Gill Chemical  
Co., Bedford.

MAYS, RUSSELL A.—Sherwin-Williams Co.,  
Cleveland.

MEYERS, RAYMOND E.—SCM Corp., Glidden  
Div., Strongsville.

MILLER, ROBERT—Harrison Paint Corp.,  
Canton.

MOSS, JOHN M.—SCM Corp., Glidden Div.,  
Strongsville.

MUSE, DON—Sherwin-Williams Co.,  
Cleveland.

OWEN, JAMES E.—Obron Corp., Painesville.

OWENS, PHILLIP M.—SCM Corp., Glidden  
Div., Strongsville.

PEKAREK, GEORGE E.—SCM Corp., Glidden  
Div., Strongsville.

REZABEK, JAMES J.—Sherwin-Williams Co.,  
Cleveland.

RICE, MARY R.—Harshaw/Filtrol, Cleveland.

SAFRAN, RUSSEL S.—Man-Gill Chemical Co.,  
Bedford.

SIMECEK, GARY—Q-Panel Co., Westlake.

SIRENO, ROBERT C.—Man-Gill Chemical Co.,  
Bedford.

SWEITZER, GERALD M.—Sherwin-Williams  
Co., Cleveland.

WALTZ, HAROLD W.—Harrison Paint Corp.,  
Canton.

WELLS, PAMELA L.—Standard Oil of Ohio,  
Warrensville, OH.

### Associate

PASSEN, FRANK J.—Obron Corp., Painesville,  
OH.

ROBBINS, JAMES H.—B.F. Goodrich Chemical  
Co., Cleveland, OH.

SEMPLER, JANE F.—Nordson Corp., Amherst,  
OH.

SPERELAKIS, JAMES P.—Johnson Wax,  
Lakewood, OH.

### Educator/Student

ERICKSON, RAY A.—Case Western Reserve  
University, Cleveland, OH.

KULKARNI, VAMAN G.—University of Akron,  
Akron, OH.

SCHURR, DONALD A.—Case Western Reserve  
University, Cleveland.

## CLEVELAND

### Active

ANWARI, FREIDUN—CRGI, Cleveland, OH.

BLAHA, DAVID A.—Man-Gill Chemical Co.,  
Bedford, OH.

BODE, DANIEL—SCM Corp., Glidden Div.,  
Strongsville, OH.

BRENNAN, Patrick J.—Q-Panel Co., Westlake,  
OH.

CARR, BRENDA L.—Coatings Development,  
Painesville, OH.

CSUMITTA, RICHARD A.—Sherwin-Williams  
Co., Cleveland.

DAMKO, Stephen J.—CRGI, Cleveland.

DIETSCH, THOMAS N. JR.—The Thermoclad  
Co., Erie, PA.

FEDOR, CAROL—Q-Panel Co., Westlake.

FELICE, RITA M.—Sherwin-Williams Co.,  
Cleveland.

FITZWATER, JOHN E.—B.F. Goodrich Chemical  
Co., Cleveland.

HAHN, KENNETH G. SR.—SCM Corp.,  
Strongsville.

HOSBACH, MARILYN J.—Jamestown Paint &  
Varnish Co., Jamestown, PA.

HOUCK, PAUL J.—Tremco Corp., Cleveland.

HSU, SHUI-JEN R.—Sherwin-Williams Co.,  
Cleveland.

JARUFE, JUAN E.—Sherwin-Williams Co.,  
Cleveland.

JEWELL, THOMAS—Jamestown Paint & Varnish  
Co., Jamestown.

KATT, RONALD A.—Harrison Paint Corp.,  
Canton, OH.

KRAFCIK, RANDOLPH B.—Sherwin-Williams  
Co., Cleveland.

LIS, ANN MARIE—Sherwin-Williams Co.,  
Cleveland.

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## KANSAS CITY

*Active*

RALSTON, RON—Farmland Industries, Inc.,  
N. Kansas City, MO.

*Retired*

KEYSER, MORRIS R.—Kansas City, MO.

## LOS ANGELES

*Active*

ALAM, M.—Durrall Plastics, Inc., Anaheim,  
CA.

BERNARD, DAN A.—Deft, Inc., Irvine, CA.

BESWICK, JAY—Textured Coatings, Los  
Angeles, CA.

DOOLITTLE, WIN—Durrall Plastics, Inc.,  
Anaheim.

JARA, NORMA D.—Whittaker Corp./Bauer  
Coatings Div., Los Angeles.

KELLY, W. ERIC—Engravers Ink Co., Brea,  
CA.

LAMOREAUX, JUSTIN F.—Engravers Ink Co.,  
Brea.

MITRA, ARUP R.—Guardsman Chemicals, Inc.,  
South Gate, CA.

MULDOWN, MICHAEL J.—Mar-Lak Products  
Co., Hawaiian Gardens, CA.

NIKOU, KAREEM A.—Beecham Home  
Improvement Products, Inc., Santa Fe  
Springs, CA.

TASHJIAN, YOUSIF D.—Western Specialty Co.,  
Los Angeles.

THOMPSON, CARL V.—Ellis Paint Co., Los  
Angeles.

*Associate*

ADDINGTON, WADE—Pacific Coast Chem.,  
Vernon, CA.

ANSLEY, STEVE—Ashland Chemicals, Santa Fe  
Springs, CA.

BROOKS, GARY L.—Oil & Solvent Process,  
Azusa, CA.

HAGEMIER, RICK—Borden Chemical Co.,  
Compton, CA.

HAMLIN, GARETH—Western Star Color &  
Chemical Co., Redondo Beach, CA.

HENDERSON, PAUL T.—Celanese Chemical Co.,  
Placentia, CA.

JOHNSON, RON L.—John K. Bice Co., Inc., Los  
Angeles, CA.

MARS, CHRISTOPHER E.—Rho Chem,  
Inglewood, CA.

MARTING, LOU—Dow Chemical Co., Mission  
Viejo, CA.

PANARIELLO, JAY R.—United States Can Co.,  
Anaheim, CA.

REED, ROBERT M.—Montgomery Brothers,  
Commerce, CA.

RICHARDS, DALES R.—Rho Chem, Inglewood.

*Retired*

BARBER, WALTER—Santa Ana, CA.

## MONTREAL

*Associate*

LAPIERRE, CLAUDE—Ashland Chemicals,  
Boucherville, Quebec.

SHEFFORD, ROSS S.—GAF Canada, St.  
Laurent, Quebec.

## NEW YORK

*Associate*

O'BRIEN, GEORGE J.—R.T. Vanderbilt Co.,  
Forked River, NJ.

## NORTHWESTERN

*Active*

FANSLAW, JAMES E.—Cargill, Inc.,  
Minneapolis, MN.

FARNUM, BRUCE W.—Certified Tech Corp.,  
Minneapolis.

HAUGAN, BRUCE D.—Arcograph, Inc.,  
Montrose, MN.

*Associate*

LOOMIS, WILLIAM W.—Zeelan Industries, St.  
Paul, MN.

## PHILADELPHIA

*Active*

PLATT, EDWARD—Laurence McFadden,  
Philadelphia, PA.

*Associate*

SMITH, KENNETH B.—Tate Engineering Inc.,  
Aston, PA.

*Educator/Student*

WHITE, MALCOLM L.—Lehigh University,  
Bethlehem, PA.

## PITTSBURGH

*Active*

GREENWALD, JOHN R.—Aluminum Co. of  
America, Alcoa Center, PA.

*Associate*

BAUDENDISTEL, CRAIG—NL Chemicals,  
Tonawanda, NY.

BOAZ, T. WILLIAM—DuPont Co., Wexford,  
PA.

HELBLING, CYNTHIA—Mobay Chemical Co.,  
Pittsburgh, PA.

VIGNETTI, ANNA M.—Neville Chemical Co.,  
Pittsburgh.

## ROCKY MOUNTAIN

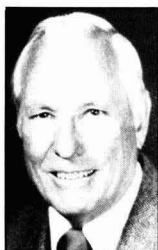
*Active*

WOLMA, KENNETH M.—Protex Industries,  
Denver, CO.

*Associate*

GEERDES, J. PATRICK—Union Chemicals,  
Denver, CO.

The McCloskey Corp., Philadelphia, PA, has named **Joseph E. King** to Senior Vice-President, Specialty Polymers Marketing. Based in Philadelphia, he will oversee sales and marketing efforts for the firm's specialty polymers division. Mr. King is a member of the Los Angeles Society and the Southern California Paint and Coatings Association.



J.E. King



W.R. Near



P. Onyskiw



M. Rapaport

DeSoto, Inc., Des Plaines, IL, has appointed **William R. Near** to Market Development Manager, New Ventures Group. Mr. Near began his employment with DeSoto in 1974, serving in technical project management and field sales positions.

University of Missouri-Rolla, Rolla, MO, has announced the addition of **Michael Van De Mark** to the Chemistry faculty. Dr. Van De Mark will serve as an Associate Professor of Chemistry. His responsibility includes research efforts in paint and polymer science as well as instructing in the paint short course program.

**Audrey LeNoble** has joined the sales staff of EMCO Chemical Distributors, Inc., N. Chicago, IL. Ms. LeNoble is a member of the Chicago Society.

Eastman Chemical Products, Inc., New York, NY, has named two new directors. **Brian D. Dillon** becomes Director, Market Management, Chemicals, and **John W. Edmisten** becomes Director Product Management, Chemicals.

**George Aman** has been named Executive Vice-President of Duron, Inc., Beltsville, MD. Mr. Aman joined Duron in 1971.

In addition, Duron has named **Jerry Berke** to Director of Field Services. Mr. Berke previously served as the firm's Sales Manager.

**John C. Miller** has become the new Vice-President of Research and Development at Angus Chemical Co., Northbrook, IL. Dr. Miller brings over eight years of chemical research and management experience to his post.

Akzo Coatings America, Inc., headquartered in Troy, MI, has promoted **Peter Onyskiw** to Manager of Process Engineering. A member of the Detroit Society, Mr. Onyskiw began his employment with Akzo in 1977.

In addition, Akzo has promoted **Peter T. Maxwell** to Vice-President Coil Products. Mr. Maxwell will be in charge of developing new markets for pre-coated metal.

**Larry M. Wlodar** has advanced to Manager of Manufacturing—Atlanta at Akzo. Mr. Wlodar will be responsible for developing new manufacturing techniques and expanding existing ones.

LanChem, a division of Akzo, has named **James W. Schweikart** to Plant Manager. Mr. Schweikart will head the firm's synthetic resin facility in E. St. Louis, IL. Mr. Schweikart is a member of the St. Louis Society.

In addition, LanChem has appointed **James B. Lindsley** to the position of Sales Manager for coatings resins and **Kees Riphagen** to Marketing Manager of printing ink resins.

**Karen Ray** has been promoted to Customer Service Manager, Northeast Polymer District, for Unocal Chemical Div., Schaumburg, IL.

**Mike Coulston** has joined MegaChem, Inc., High Point, NC, as Product Manager for pigments and chemicals. Mr. Coulston brings 20 years of experience to the position.

**Monroe Rapaport**, of Pryolac Corp., Hawthorne, NJ, has volunteered to provide assistance to an industrial coatings and alkyd manufacturer in Sri Lanka through a project sponsored by the International Executive Service Corps. He will work to upgrade the management skills, improve basic technologies, and increase productivity of businesses in this country. Mr. Rapaport is a member of the 50 Year Club of NPCA.

Glidden Coatings & Resins, Div. of SCM Corp., has appointed **Martin Hastings** to Field Sales Manager, Coil and Wood Coatings. He is responsible for planning and controlling regional sales and marketing efforts.

Southern Society member **Art Benton**, of McCullough & Benton, Inc., Atlanta, GA, has been elected to a two-year term as President of Materials Marketing Associates, Inc., Hartford, CT.

Also elected to MMA officer positions were: Treasurer—**Eugene P. Diagle**, of Mehaffey and Daigle, Inc., a member of the Southern Society, and Vice-President—**Tom R. Brandt**, of George C. Brandt Co., a member of the Chicago Society.

Two new members elected to the MMA Board are **Jack Boorman**, of Van Horn, Metz & Co., and **Jack McMahon**, of Majemac Enterprises. Mr. Boorman is a member of the Philadelphia Society.

## Max Saltzman Is Chosen Recipient Of 1986 ISCC-Macbeth Award

Max Saltzman, a long-time member of the Federation and its New York and Los Angeles Societies, and an Honorary Member of the Inter-Society Color Council, has been chosen as the recipient of the ISCC-Macbeth Award for 1986.

Mr. Saltzman is a distinguished scholar and scientist in the field of color technology and a recognized authority on the history and technology of ancient dyestuffs. Since 1973, he has been Head of the Laboratory for Historical Colorants in the Institute of Geophysics and Planetary Physics at the University of California, Los Angeles.

Mr. Saltzman has been associated with the dye and pigment industry for many years. He retired as Manager of Color Technology of Allied Chemical Corp.'s Specialty Chemical Div. in 1973. From 1965-82, he was an Adjunct Professor of Chemistry (Color Science) at Rensselaer Polytechnic Institute.

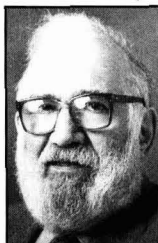
His many activities in industry and education over the years have included: Chairman of the Technical Committee of the Dry Color Manufacturers Association; Co-author (with F.W. Billmeyer, Jr.) of "Principles of Color Technology"; authorship of 12 technical publications; and many lectures on the subject of color measurement, appropriate use of colorants, chemistry, and technology of chromatic pigments, dyes, and pigments of historical interest.

He received the Federation's Bruning

Award, for outstanding contributions to the science of color in the field of coatings technology, in 1969.

Other organizations in which he holds membership are: American Chemical Society, American Association of Textile Chemists and Colorists, Society of Plastics Engineers, Society of Dyers and Colourists, The Colour Group, Optical Society of America, International Institute for Conservation, and American Institute for Conservation.

The Inter-Society Color Council is a non-profit organization of societies and creative individuals—artists, designers, industrialists, educators and scientists—actively interested in the study of color as it



applies to Art, Science and Industry. The Council acts as a forum for the interchange of ideas and programs among the varied disciplines represented by its member bodies and individual members. (The Federation is a member).

The ISCC-Macbeth Award, first presented in 1967, is awarded every two years in recognition of recent important contributions in the field of color, preferably made within the last ten years. The Award was established by the late Norman Macbeth, a past Director and long time Treasurer of the Inter-Society Color Council, to honor the memory of his father Norman Macbeth, Sr. and his pioneering contributions to the art and science of color and illumination.

**Ronald S. Bauer**, of Shell Development Co., Houston, is the current Chairman of the ACS Div. of Polymeric Materials Science and Engineering. Other officers are: Chairman-Elect—**John H. Lupinski**, of General Electric Co., Schenectady, NY; Vice-Chairman—**Ray Dickie**, of Ford Motor Co., Dearborn, MI; Secretary—**Theodore Davidson**, of Ethicon, Inc., Somerville, NJ; and Treasurer—**Theodore Provder**, of Glidden Coatings & Resins, Strongsville, OH. Mr. Dickie is a member of the Detroit Society and Mr. Provder is a member of the Cleveland Society.

**Owen L. Quantz** has become the Sales Manager for Morton Thiokol, Inc., Morton Chemical Div., headquartered in Chicago, IL. Based in Chicago, Mr. Quantz will be responsible for the marketing of specialty polymers products.

The Morton Chemical Div. has also promoted **Roger L. Isbister** to Aqueous Polymers Product Manager and **Bill McCune** to Technical Sales Representative. Mr. McCune will be based in Woodland Hills, CA.

The Coatings Division of Ferro Corp., Cleveland, OH, has promoted **Harvey C. Johnson** to Automotive Market Manager, Powder Coatings. Mr. Johnson joined Ferro in 1980 and most recently served as Northern Regional Sales Manager.

**Peter W. Kopf**, formerly a Group Leader at Union Carbide Corp., has joined Arthur D. Little, Inc., Cambridge, MA, as a Senior Consultant in the firm's Product Technology section. He brings more than 15 years of experience in the development of polymer and plastics coatings to his new post.

**Stan LaBelle** has been named Technical Service Manager for the Protective Coatings Group of H.B. Fuller Co., St. Paul, MN. He will supervise product development and technical service for the group.

**Robert J. Purcell, Jr.**, has joined Witco Corp., New York, NY, as a Sales Representative for the organics division. He will be based in Perth Amboy, NJ.

**Richard J. Di Marcantonio** has joined the A.B. Kohl Sales Co., Towson, MD, as a Technical Sales Representative. He is a member of the Baltimore Society and will be responsible for accounts in Virginia, and portions of North Carolina and Maryland.

Sun Chemical Corp., Cincinnati, OH, has appointed two new sales representatives. **Dave Scheick** will be based in the southeast region, and **Tim Polgar** will serve the Cleveland area.

CasChem, Inc., Bayonne, NJ, has named **Jerry J. Farro** to President and Chief Operating Officer. Prior to the appointment, Mr. Farro served as the company's Executive Vice-President and Chief Operating Officer.

## Obituary

**Joseph A. Limardo**, President of Burks, Inc., died on January 25. Born in New York, Mr. Limardo moved to Atlanta in 1959 and joined Charles L. Burks & Co. in 1961. He was a member of the Southern Society for Coatings Technology. As President of Burks, Inc., Mr. Limardo was instrumental in building the agency-distributorship serving the southeast region.

New York Society member **Milton Klein** recently passed away. After serving as Technical Director with Hoboken White Lead Co. and Pergament Home Centers, Mr. Klein retired in 1974 to become a consultant to the coatings industry.

## "Coatings and Films" Program Among Topics Scheduled for 1986 Gordon Research Conferences

The 1986 Gordon Research Conferences, designed to foster and promote education and science by organizing informal meetings of research scientists with common interests in the fields of chemistry and related sciences, have been scheduled in various academic locations in New Hampshire from June 9 to August 22.

Among the varied topics to be considered is "Physics and Chemistry of Coatings and Films," to be held August 11-15 at the Plymouth State College (S), Plymouth, NH. Chaired by Dr. Irvin M. Krieger, of Case-Western Reserve University, the sessions will include the following presentations:

### AUGUST 11

"Theories of Flocculation by Water-Soluble Polymers"—Peter R. Sperry.

"Structural Features Promoting Association in Thickened Latex/TiO<sub>2</sub> Slurries"—J. Edward Glass.

"Correlation of Chemical Structure and Properties of Materials of Interest to the Coatings Industry"—Vincent D. McGinness.

### AUGUST 12

"Mathematical Models of Coatings and Films"—Robert Mullen.

"Physics of the Flow of Coatings"—L.E. Scriven.

"Film Formation from Latex Polymers"—Paul van Rheenen.

### AUGUST 13

"Rheology of Concentrated Dispersions"—James Goodwin.

"Effect of Particle Interactions on Low-Shear Rheology of Model Coatings Systems"—Richard Buscall.

"Elongational Viscosity"—James Ferguson.

"Surface Elasticity and Foam Stability of Water-Borne Polymers and Coatings"—Richard Eley.

### AUGUST 14

"Photodegradation, Photostabilization, and Accelerated Tests"—David Bauer.

"Early Detection of Photodegradation Processes by Physical Methods"—Siegfried Storp.

"Use of SIMS Techniques for Surface and Interface Studies of Polymers and Polymeric Coatings"—Wim van Ooij.

### AUGUST 15

"Characterization of Metallic Orientation"—G. Dubbeldam.

"Adhesive Strength of Coatings"—Abdelsamie Moet.

For more information on these and other topics to be presented, contact Dr. Alexander M. Cruickshank, Director, Gordon Research Conferences, Gordon Research Center, University of Rhode Island, Kingston, RI 02881.

## Washington Paint Technical Group Conducts 26th Annual Symposium

"The Government/Industry Paint Puzzle" was the theme of the Washington Paint Technical Group's 26th Annual Symposium held April 7-8, 1986, at the Twin Bridges Marriott Motor Hotel, Washington, D.C.

Opening remarks by WPTG President Kenneth L. Zacharias and Program Chairman Lloyd Smith started the two-day event. The keynote address followed and was delivered by Lou DeProspero, Gene Moore, and Bob Bearden, of the General Services Administration/Federal Supply Service.

The symposium offered 12 presentations. Topics and speakers included the following:

### MONDAY, APRIL 7

"Review of Pesticide Products Containing Tributyltins"—Speaker from the Environmental Protection Agency, Office of Pesticide Programs.

"Purchasing Paint by Weighted Cost of Active Ingredients"—William V. Moseley, Jr., Virginia Dept. of Highways and Transportation.

"Supplying Paint to the Government—A Small Company Perspective"—J.E. Spector, Everseal Manufacturing Co., Inc.

"Battle Against Corrosion—Electrocoating for the Military"—Peter Beucher, PPG Industries, Inc.

"Specifications—Unraveling the Coatings Mystery"—Barry Saylor, High Industries, Inc.

"NAVSEA Quality Assurance Program for Coatings"—Henry Bleile, Naval Sea Systems Command.

### TUESDAY, APRIL 8

"Coordination of Standardization Efforts"—Tom McAndrews, GSA Center.

"Corrosion Control Program in Air Force Facilities"—Jesse R. Neal, Jr., HQ Air Force, Tyndall AFB.

"Navy Acquisition of Painting Materials and Services"—Richard Drisco and Peter Hearst, Naval Civil Engineering Laboratory.

"Compliance with Government Paint Specifications"—Alfred Beitelman, U.S. Army Construction Engineering Research Laboratory.

"Specification Needs for the Federal Facility Engineer"—Bruce Flowers, Naval Facility Engineering Command.

"DOD Painting Programs and VOC"—Art Vance, Defense Materials Specifications and Standards Office.





# Kent State University Offers Dispersion Course, June 2-6

Kent State University, Kent, OH, has scheduled a short course on "Dispersion of Pigments and Resins in Fluid Media," for June 2-6, 1986. The course will be conducted at the university and offers an overview on the fundamental applied information related to the practice of dispersion science.

The program is targeted to those in research, development, and production positions in the coatings, adhesives, inks, polymers, and related materials industries. Topics and speakers are as follows:

## MONDAY, JUNE 2

"Fundamentals of Surface and Colloid Chemistry I"—Richard J. Ruch, Kent State University.

"Dispersant Selection Based on Pigment Functionality"—Robert F. Conley, Mineral and Resource Technology.

"Fundamentals of Surface and Colloid Chemistry II"—Richard J. Ruch.

"Mineral Pigment Processing Parameters and Their Influence on Polymer Properties"—Robert F. Conley.

## TUESDAY, JUNE 3

"White Pigments—Processing and Rheology"—Robert F. Conley.

"Pigment Dispersion and Rheology"—Walter K. Asbeck, Consultant.

"Particle Size Analysis of Coatings Systems"—Theodore Provder, Glidden Coatings & Resins Division, SCM Corp.

"Dispersion of Organic Pigments"—Theodore Vernardakis, Sun Chemical Corp.

## WEDNESDAY, JUNE 4

"The Use of Rheological and Sedimentation Techniques in Colloid Science"—

Mel Croucher, Xerox Research Centre of Canada.

"Formulation of Solvent-Based Mill Bases"—James E. McNutt, Du Pont Co., Coatings Division.

"Dispersion in Oil-Soluble Coatings Systems: The Inseparable Roles of Art and Science"—George R. Pilcher, Hanna Chemical Coatings Corp.

"Preparation Techniques of Magnetic Dispersions"—Frederick D. Rotar, Netzsch, Inc.

## THURSDAY, JUNE 5

"Small Media Milling and Dispersion Technology"—Leo Dombrowski, Chicago Boiler Co.

"High Viscosity Dispersion"—James W. White, White Chemical Equipment Co.

"Characterization of Grinding and Dispersion Media"—Walter W. Perkins, Consultant.

"Principles of Ball and Pebble Milling"—John M. Rahter, Paul O. Abbe, Inc.

"Attritor Grinding and Dispersing Equipment"—Arno Szegvari, Union Process, Inc.

## FRIDAY, JUNE 6

"Dispersing Fine Particle Materials"—David G. Bosse, Magnet-Projects.

Additional details can be obtained from Carl J. Knauss, Chemistry Dept., Kent State University, Kent, OH 44242.

## Applied Color Systems Sponsors Color Technology Seminars

A series of two- and three-day seminars on industrial color technology and its impact on business will be conducted in various locations in the northeastern portion of the U.S. by Applied Color Systems, Inc., Princeton, NJ. Under the direction of Ralph Stanziola, Color Consultant and President of Industrial Color Technology, the seminars are designed to provide managers with an overall understanding of color control techniques for reducing waste and improving product quality and productivity.

Technical personnel will be updated on the latest developments in color technology, equipment, and systems. Topics to be explored in the seminars include: colorimetry and factors affecting color; spectrophotometry and metamerism; colorant

characteristics; color differences; and application of color theory.

A complete listing of locations and dates, and registration details are available from Ms. Bobbie Deel, Applied Color Systems, Inc., 2848 M Carolina Center, I-85 S., Charlotte, NC 28208.

## AESF Announces SUR/FIN '86 Program

The American Electroplaters and Surface Finishers Society (AESF) has announced technical program plans for SUR/FIN '86, its 73rd Annual Technical Conference and 21st Exhibit of Surface Finishing, to be held June 23-26, 1986, at the Civic Center and Wyndham Franklin Plaza Hotel, in Philadelphia, PA.

Designed to involve all aspects of the electroplating/surface finishing industry, the technical program features 12 sessions comprised of 55 paper presentations. Session topics include: organic finishing, vacuum coating, environmental concerns, electroforming, general interest, research, analytical methods, electronics, and light metal finishing.

SUR/FIN also features a plant tour to SPS Technologies to full registrants. Other highlights include the William Blum Lecture, awards presentations, AESF Annual Business meeting, and election of AESF Officers. Special programs for spouses/guests and youths are also offered.

Registration information can be obtained from Mary Lou Dowdell, AESF, 12644 Research Parkway, Orlando, FL 32826.

## CALL FOR PAPERS

### 6th International Meeting On Radiation Processing

May 31-June 5, 1987  
Ottawa, Ontario, Canada

The sixth meeting of this biennial series will be conducted at the Skyline and Holiday Inn Hotels in Ottawa.

The program will consist of invited lectures and contributed papers. Those interested in presenting contributed papers are invited to submit abstracts (no more than 300 words) on state of the art technology related to industrial uses of radiation processing, including thin film coatings and adhesives. Abstracts should be forwarded by June 1, 1986, to Mrs. E. Golding, 6th International Meeting on Radiation Processing, P.O. Box 13533, Kanata, Ontario, Canada K2K 1X6.

## Colorant Dispenser

A recently released product bulletin describes a compact, automatic colorant dispenser for paint dealers. Included are features and specifications of the dispenser, including computer controlled stirring and recirculation of colorants, automatic platform adjustment for varying can sizes, and complete computer control of all mechanical components. For more information, contact Applied Color Systems, Inc., P.O. Box 5800, Princeton, NJ 08543.

## Anti-Corrosive Primer

Newly published information focuses on the characteristics of an anti-corrosion primer for sheet metal parts. Designed to provide excellent rustproofing properties and to cover more parts per gallon than traditional anti-corrosive primers, this new product was developed as an economical alternative to cathodic electrocoat. For more information, contact Akzo Coatings America Inc., 650 Stephenson Hwy., Troy, MI 48083.

## Test Centers

A comprehensive test center which provides solutions for a broadened range of dry processing problems is described in recently released literature. The center provides simulated laboratory, pilot, and production runs to customer specifications, including complete analysis of test results. For complete details, write: Alpine American Corporation, Mechanical Processing Div., 5 Michigan Dr., Natick, MA 01760.

## Cartridge Filters

The features of a line of wound and pleated cartridge filters are described in a new 30-page guide. Designed to assist the cartridge filter user in making the best selection for an application, the booklet covers the design of the different types of cartridges, fluid compatibility and its effect on the performance of the different types of cartridge filters, non-destructive testing and other quality assurance programs used by the cartridge filter manufacturer to maintain quality and consistency of product, as well as guidelines to system design. This guide is available from Tom Murphy, Marketing Manager, Filterite, 2033 Greenspring Dr., Timonium, MD 21093.

## Precision Balances

A recently released product bulletin introduces a line of precision balances which feature an exclusive multifunctional display. For more information, contact Mettler Instrument Corp., Laboratory Div., Box 71, Hightstown, NJ 08520.

## Epoxy Hardeners

A six-page, four-color brochure describes the advantages of extra-fast setting, low-temperature-cure epoxy hardeners. The products are designed to offer faster cures in mass and thin film, cures at lower temperatures and shorter induction time of epoxy/polyamide coatings with no loss of gloss. For copies of the brochure on Capcure<sup>®</sup> mercaptan and amine curing agents, write to Diamond Shamrock Chemicals Co., 350 Mt. Kemble Ave., CN-1931, Morristown, NJ 07960-1931.

## Mineral Extender

A calcined aluminum silicate extender has been introduced in literature. Designed as an ultra high brightness, very white, high bulking functional extender for the development of excellent opacity, high reflectance and low sheen, the product can be used in a variety of latex and oil-based paint systems. For additional information, contact Engelhard Corp., 70 Wood Ave. South, Iselin, NJ 08830.

## Laboratory Dispenser

Information has been released which details the features of an explosion-proof laboratory dispenser which may be used with any of five interchangeable mixing heads. For a product bulletin on the Explosion-proof, Variable Speed Laboratory Dispenser, contact Premier Mill Corp., 220 East 23rd St., New York, NY 10010.

## Polyurethane Coating

A new technical bulletin details a conductive, silver-filled, two-part polyurethane finish. This finish is specifically formulated to meet the requirements of electromagnetic and radio frequency interference shielding for flexible coatings applications. For more information, contact Carroll Coatings Co., 217 Chapman St., Providence, RI 02905.

## Solvent

A six-page brochure characterizing a new high performance/low toxicity propylene glycol monotertiary butyl ether has been issued. Information summarizing typical physical properties and illustrating typical coating and cleaner formulations is presented. Copies of the brochure, "Introducing ARCOSOLV PTB Solvent," are available from the Marketing Communications Dept., ARCO Chemical Co., 1500 Market St., Philadelphia, PA 19101.

## Air Preparation

Literature which describes a new air preparation system is now available. The system is designed specifically for use with ionizing air systems which neutralize static charge in a wide range of manufacturing operations. For further information about the 3M Model 913 air preparation system, contact 3M, Dept. ST85-26A, P.O. Box 2963, Austin, TX 78769-2963.

## Analytical Services

Analysis and testing capabilities for the polymer and coatings industries are highlighted in a new brochure. The information describes the use of instrumental techniques of analysis as they apply to problem-solving in manufacturing, formulation, and quality control. Request copies from Vance Laboratories, Inc., 5728 W. 71st St., Indianapolis, IN 46278.

## Precision Batching

A new line of industrial scales for precision batching and check weighing has been introduced in literature. Attached, post-mounted, remote, or dual display models are available. For further information, write Sartorius Scale Div., 11 Kripes Rd., East Granby, CT 06026.

## Color Analysis System

Information is now available on a new color analysis system intended to perform high-volume QA/QC testing on incoming, in-progress, and finished products. Complete details are available from Milton Roy Co., Analytical Products Div., 820 Linden Ave., Rochester, NY 14625.

## Viscosity-Temperature Sensor

A viscosity-temperature sensor which uses a new electromagnetic measurement principle is the subject of literature. Developed for computer monitoring and control applications, the sensor is suited for industrial environments. For more information, write Cambridge Applied Systems, Inc., 23 East St., Cambridge, MA 02141.

## Lab & Technology Resources

The second edition of the *Directory of Federal Laboratory and Technology Resources—A Guide to Services, Facilities, and Expertise*, has been published. The work offers readers the possibility of working with special labs, research centers, testing facilities, and special technology information centers. Detailed indexes cover subject, location by state, organization name, and Federal agency. To order this 288-page, softcover book, send \$29, plus \$3 for shipping and handling, to National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22161.

## Silicone Antifoams

A fold-out guide to silicone antifoams and their applications has been issued. Contained in the folder is a tabular selection guide that matches silicone antifoam products with their applications in a wide range of industries. The industries covered include food, chemical, petroleum and gas processing, textiles, coatings, adhesives and inks, chemical specialties, and paper. Copies of the silicone antifoam guide, designated SUI-552, are available from Union Carbide Corp., Specialty Chemicals Div., Dept. L-3493, 39 Old Ridgebury Rd., Danbury, CT 06817-0001.

## Electrochemical Corrosion Measurement

A new system for automated electrochemical corrosion measurement is the focus of recently issued literature. For complete information on Model 342-1, write: EG&G Princeton Applied Research, Electrochemical Instruments Div., CN 5206, Princeton, NJ 08540.

## Product Data Sheets

Up-dated product data sheets on a complete line of propylene glycol ethers and acetates have been issued recently. These products are designed for use in reformulation of paints, cleaners, and inks which formerly used ethylene-based solvents. Copies are available from the Marketing Communications Dept., ARCO Chemical Co., 1500 Market St., Philadelphia, PA 19101.

## Chemical Resistance

A new chemical resistance guide for polyvinyl chloride Type 1, Grade 1 users is now available. Compiled from various literature sources, the brochure lists nearly 700 chemical materials and categorizes their resistance to PVC Type 1, Grade 1 products. For a copy of "PVC Chemical Resistance Guide," contact Air Products and Chemicals Inc., Plastics Div., Box 538, Allentown, PA 18105.

## UV Energy Controller

An electro-optic instrument which controls the amount of UV energy impinging on a workpiece in a conveyORIZED UV curing system is the focus of technical literature. The instrument permits the operator to choose a switch selectable energy level known to provide suitable curing, and it automatically adjusts conveyor speed to obtain the selected energy level. Additional details may be obtained from Bob Radcliff, EIT, Inc., 108 Carpenter Dr., Sterling, VA 22170.

## Inspection Instrumentation

A recently released data sheet details the features of a test kit designed to identify and quantify water-soluble chemical contamination on surfaces. The kit contains water demineralizer, chloride ion indicator strips, ferrous ion indicator strips, pH paper, necessary equipment for sample collection, a procedure booklet, and a carrying case. For more information, contact KTA-Tator, Inc., 115 Technology Dr., Pittsburgh, PA 15275.

## Solvents

An eight-page booklet describing a solvent for industrial coatings has been published. Use of the new ether ester solvent in high solids and electro-statically sprayed coatings, as well as in conventional enamels and lacquers is covered in the booklet. For copies of "UCAR Ester EEP," designated F-060101, contact Union Carbide Corp., Solvents & Coatings Materials Div., K3442, 39 Old Ridgebury Rd., Danbury, CT 06817-0001.



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**AUDIO/VISUAL PRESENTATIONS**

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**HIGH SPEED DISPERSION**

*Produced by the Manufacturing Committee, Montreal Society for Coatings Technology*

High speed dispersion is widely used in the manufacture of protective coatings. The show covers theoretical and practical techniques used for dispersion in paint plants. Color slides show laboratory test equipment and plant scale manufacturing procedures. 20 Minutes (60 Slides) ..... \$65

**INTRODUCTION TO RESIN OPERATIONS**

*Produced by the Manufacturing Committee, Toronto Society for Coatings Technology*

This presentation has been developed to assist in the selection and training of resin plant operators, and focuses on basic concepts of manufacture and the role of a resin operator. 12 minutes (58 slides) ..... \$65

**OPERATION OF A VERTICAL SAND MILL**

*Produced by the Manufacturing Committee, Kansas City Society for Coatings Technology*

This presentation focuses on the basics of operating a vertical sandmill, and has been developed to assist in training plant personnel in the use of this equipment. 14 minutes (73 slides) ..... \$75

**A BATCH OPERATED MINI-MEDIA MILL**

*Produced by the Manufacturing Committee, New York Society for Coatings Technology*

This presentation describes the design and operation of a batch operated mini-media mill, and was developed to assist in the training of plant personnel to operate such equipment. 8½ minutes (51 slides) ..... \$60

**TRAINING SERIES ON TEST METHODS**

**Volume II (3 Lessons)** Lessons vary from 7 to 11 minutes (79 slides) ..... \$70

- (1) A Simple Method to Determine Microbiological Activity—Philadelphia Society;
- (2) Salt Spray (Fog) Testing Cabinet—Golden Gate Society;
- (3) Wet Film Thickness Gages—Golden Gate Society.

**For additional titles contact:**

**FEDERATION OF SOCIETIES  
FOR COATINGS TECHNOLOGY**  
1315 Walnut Street • Philadelphia, PA 19107  
215-545-1506

# Coming Events

## FEDERATION MEETINGS

For information on FSCT meetings, contact FSCT, 1315 Walnut St., Philadelphia, PA 19107 (215-545-1506).

1986

(June 3-6)—Symposium on Automotive Color Control (SACC). Sponsored jointly by FSCT, Detroit Society, Detroit Colour Council, and Manufacturers Council on Color and Appearance. Michigan Inn, Southfield, MI.

(Nov. 5-7)—64th Annual Meeting and 51st Paint Industries' Show. World Congress Center, Atlanta, GA.

1987

(Apr. 28-May 2)—Combined Federation Spring Week and Pacific Northwest Society Symposium. FSCT Society Officers Meeting on April 28; FSCT Board of Directors Meeting on April 29; FSCT Spring Seminar (and PNW Society Technical Sessions) on April 30 and May 1. PNW Society activities on Saturday, May 2, will conclude the week.

(Oct. 5-7)—65th Annual Meeting and 52nd Paint Industries' Show. Convention Center, Dallas, TX.

## SPECIAL SOCIETY MEETINGS

1986

(May 22)—Birmingham Club. Symposium: "Miracle '86." Strathallan Hotel, Birmingham, England. (David Heath, Holden Surface Coatings Ltd., Bordesley Green Rd., Bordesley Green, Birmingham B9 4TQ, England).

(June 6-7)—Joint meeting of Kansas City and St. Louis Societies. Holiday Inn, Lake of the Ozarks, MO. (Steve Bussjaeger, Davis Paint Co., 1311 Iron St., N. Kansas City, MO 64116).

(June 16)—Golden Gate Society Manufacturing Committee Conference, "Demonstration/Determination of VOC in Solvent- and Water-Borne Coatings." Holiday Inn, S. San Francisco, CA. (E. "Bud" Harmon, Borden Chemical Co., 41100 Boyce Rd., Fremont, CA 94538).

1987

(Feb. 23-25)—Western Coatings Societies' Symposium and Show, Monterey Convention Center, Monterey, CA. (Barry Adler, Royell, Inc., 1150 Hamilton Ct., Menlo Park, CA 94025).

(Apr. 1-3)—Southern Society. Annual Meeting. Dutch Inn, Lake Buena Vista, FL. (C. Lewis Davis, 802 Black Duck Dr., Port Orange, FL 32019).

(Apr. 28-May 2)—Combined Federation Spring Week and Pacific Northwest Society Symposium. FSCT Society Officers Meeting on April 28; FSCT Board of Directors Meeting on April 29; FSCT Spring Seminar (and PNW Society Technical Sessions) on April 30 and May 1. PNW Society activities on Saturday, May 2, will conclude the week.

## OTHER ORGANIZATIONS

1986

(May 19-22)—"Basic Microcomputer Programs for Coatings" Short Course sponsored by the University of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, University of Missouri-Rolla, Rolla, MO 65401).

(May 19-23)—"Basic Microcomputer Programming for Coatings" Short Course. Univ. of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, Univ. of Missouri-Rolla, Rolla, MO 65401).

(May 21-23)—"Advances in the Stabilization and Controlled Degradation of Polymers" Eighth International Conference. Luzern, Switzerland. (Dr. A.V. Patsis, Institute in Materials Science, State University of New York, New Paltz, NY 12561).

(June 2-6)—"Dispersion of Pigments and Resins in Fluid Media" Short Course sponsored by Kent State University, Kent, OH. (Carl J. Knauss, Kent State University, Chemistry Dept., Kent, OH 44242).

(June 2-6)—"Advances in Emulsion Polymerization and Latex Technology" Short Course sponsored by Lehigh University, Bethlehem, PA. (Dr. Mohamed S. El Aasser, Dept. of Chemical Engineering, Sinclair Lab #7, Lehigh University Bethlehem, PA 18015).

(June 10-11)—Industrial Color Technology Seminar sponsored by Applied Color Systems, Inc. Cleveland, OH. (Ms. Bobbie Deel, ACS, 2848 M Carolina Center, I-85 S., Charlotte, NC 28208).

(June 10-16)—CHINAPLAS 86. International Exhibition Centre, Beijing, China. (Kallman Associates, 5 Maple Court, Ridgewood, NJ 07450).

(June 11-12)—"Coatings for Wood" Seminar sponsored by the Paint Research Association. Forum Hotel, London, England. (Dip Dasgupta, Head of Information, Paint Research Association, Waldegrave Rd., Teddington, Middlesex TW11 8LD England).

(June 15-18)—60th Colloid and Surface Symposium. Georgia Institute of Technology, Atlanta, GA. (Symposium Chairman, M.J. Matteson, School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332).

(June 15-18)—1986 Annual Meeting of Inter-Society Color Council. Ryerson Polytechnical Institute, Toronto, Ont. (Dr. Peter Kaiser, Dept. of Psychology, York University, 4700 Keele St., N. York, Ont. M3J 1P3).

(June 17-19)—"Industrial Painting Processes" clinic sponsored by AFP/SME. Toronto, Ont., Canada. (Diane Korona, SME Special Programs Div., SME Dr., P.O. Box 930, Dearborn, MI 48121).

(June 18-19)—"Care and Maintenance of Exterior Timber" Symposium co-sponsored by Paint Research Institute and Prince Research Laboratory, Crest Hotel, London, Heathrow, England. (Dip Dasgupta, PRA, Waldegrave Rd., Teddington, Middlesex TW11 8LD England).

(June 23-26)—SUR/FIN '86-Annual Technical Conference of American Electroplaters and Surface Finishers Society. Civic Center and Wyndham Franklin Plaza Hotel, Philadelphia, PA. (Mary Lou Dowdell, AESF, 12644 Research Pkwy., Orlando, FL 32826).

(July 1-3)—Industrial Color Technology Seminar sponsored by Applied Color Systems, Inc. Myrtle Beach, SC. (Ms. Bobbie Deel, ACS, 2848 M Carolina Center, I-85 S., Charlotte, NC 28208).

(July 3-6)—Oil and Colour Chemists' Association Australia. 28th Annual Convention. The Estate, McLaren Vale, South Australia. (OCCAA, 6 Wilson Ave., Felixstow, South Australia 5090, Australia).

(July 7-11)—"Organic Coatings Science and Technology" Twelfth International Conference. Athens, Greece. (Dr. A.V. Patsis, Institute in Materials Science, State University of New York, New Paltz, NY 12561).

(Aug. 11-15)—"Physics and Chemistry of Coatings & Films" Program. Gordon Research Conference. Plymouth State College (S), Plymouth, NH. (Dr. Alexander Cruickshank, Director, Gordon Research Center, University of Rhode Island, Kingston, RI 02881).

(Sept. 3-4)—Industrial Color Technology Seminar sponsored by Applied Color Systems, Inc. Lancaster, PA. (Ms. Bobbie Deel, ACS, 2848 M Carolina Center, I-85 S., Charlotte, NC 28208).

(Sept. 3-5)—"Estimating for Painting Contractors and Maintenance Engineers" Short Course. Univ. of Missouri-Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, Univ. of Missouri-Rolla, Rolla, MO 65401).

(Sept. 7-12)—Symposium on High Solids Coatings. Sponsored by the ACS Div. of Polymeric Materials: Science and Engineering. Anaheim, CA. (George R. Pilcher, Hanna Chemical Coatings Corp., P.O. Box 147, Columbus, OH 43216).

(Sept. 7-12)—ACS, Div. of Polymeric Materials: Science and Engineering. Anaheim, CA. (T. Davidson, Ethicon, Inc., Route 22, Somerville, NJ 08876).

(Sept. 8-13)—190th National Meeting. American Chemical Society. Chicago, IL. (ACS, A.T. Winstead, 1155 16th St. N.W., Washington, D.C. 20036).

(Sept. 9-11)—RadCure '86—Association for Finishing Processes of the Society of Manufacturing Engineers Conference and Exposition. Baltimore Convention Center, Baltimore, MD. (AFP/SME Public Relations, Society of Manufacturing Engineers, One SME Dr., Dearborn, MI 48121).

(Sept. 15-17)—13th International Naval Stores Meeting. Waldorf-Astoria, New York, NY. (Douglas E. Campbell, Executive Director, Pulp Chemicals Assn., 60 E. 42nd St., New York, NY).

(Sept. 15-19)—53rd Introductory Short Course on "The Basic Composition of Coatings." Univ. of Missouri-Rolla, MO. (Prof. James

O. Stoffer, Dept. of Chemistry, Univ. of Missouri-Rolla, Rolla, MO 65401).

(Sept. 21-23)—Canadian Paint and Coatings Association. 74th Annual Convention. Hilton Hotel, Quebec City, Que., Canada. (CPCA, 515 St. Catherine St. W, Montreal, Que., Canada H3B 1B4).

(Sept. 21-26)—XVIIIth Congress of FATIPEC. (Federation of Associations of Technicians in the Paint, Varnish, and Printing Ink Industries of Continental Europe). Venice, Italy. (C. Bourger, Secretary General of FATIPEC, 76 Blvd. Pereire, 75017 Paris, France-or-Amleto Poluzzi, AITVA, Piazzale R. Morandi 2, 20121 Milano, Italy).

(Sept. 22-25)—"Your Chosen Finish." FINSTRAT Conference and Exposition sponsored by the Association for Finishing Processes of the Society of Manufacturing Engineers. Long Beach, CA. (Gerri Andrews, SME, Public Relations Dept., One SME Dr., P.O. Box 930, Dearborn, MI 48121).

(Sept. 23-25)—"Industrial Painting Processes" clinic sponsored by AFP/SME. Indianapolis, IN. (Diane Korona, SME Special Programs Div., SEM Dr., P.O. Box 930, Dearborn, MI 48121).

(Sept. 29-Oct. 3)—13th Introductory Short Course on "Paint Formulation." Univ. of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, Univ. of Missouri-Rolla, Rolla, MO 65401).

(Oct. 1-2)—Industrial Color Technology Seminar sponsored by Applied Color Systems, Inc. Minneapolis, MN. (Ms. Bobbie Deel, ACS, 2848 M Carolina Center, I-85 S., Charlotte, NC 28208).

(Oct. 8-10)—SPI/SPE Plastics Show and Conference—South. Georgia World Congress Center, Atlanta, GA. (Jeffrey A. Forger, Conference and Programs Director, SPE, 14 Fairfield Dr., Brookfield Center, CT 06805-0403).

(Oct. 13-17)—"Physical Testing of Paints & Coatings" Short Course. Univ. of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, Univ. of Missouri-Rolla, Rolla, MO 65401).

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(Oct. 27-28)—Industrial Color Technology Seminar sponsored by Applied Color Systems, Inc. Dalton, GA. (Ms. Bobbie Deel, ACS, 2848 M Carolina Center, I-85 S., Charlotte, NC 28208).

(Oct. 27-31)—"Modern Instrumentation for the Polymer & Coatings Industry" Short Course. Univ. of Missouri-Rolla, Rolla, MO. (Prof. James O. Stoffer, Dept. of Chemistry, Univ. of Missouri-Rolla, Rolla, MO 65401).

(Nov. 3-4)—Industrial Color Technology Seminar sponsored by Applied Color Systems, Inc. Atlanta, GA. (Ms. Bobbie Deel, ACS, 2848 M Carolina Center, I-85 S., Charlotte, NC 28208).

(Nov. 3-5)—Paint Research Association. Sixth International Conference, Sheraton Hotel, Brussels, Belgium. (D. Dasgupta, PRA, 8 Waldegrave Rd., Teddington, Middlesex TW11 8LD, England).

(Nov. 3-5)—Annual Meeting of National Paint and Coatings Association. Hilton Hotel, Atlanta, GA. (Karen Bradley, NPCA, 1500 Rhode Island Ave., Washington, D.C. 20005).

(Nov. 21-23)—39th Annual Show and Convention of National Decorating Products Association. Cervantes Convention Center, St. Louis, MO. (Lillian Smysor, NDPA, 1050 N. Lindbergh Blvd., St. Louis, MO 63132).

(Dec. 3-5)—Industrial Color Technology Seminar sponsored by Applied Color Systems, Inc. Orlando, FL. (Ms. Bobbie Deel, ACS, 2848 M Carolina Center, I-85 S., Charlotte, NC 28208).

(Apr. 5-10)—ACS, Div. of Polymeric Materials: Science & Engineering, Anaheim, CA. (T. Davidson, Ethican, Inc., Route 22, Somersville, NJ 08876).

(May 31-June 5)—Sixth International Meeting on Radiation Processing Skyline and Holiday Inn Hotels, Ottawa, Ont., Canada. (Mrs. E. Golding International Meeting on Radiation Processing, P.O. Box 13533, Kanata, Ont., Canada K2K 1X6).

(June 17-19)—"Chemically Modified Surfaces" Conference co-sponsored by Colorado State University and Dow Corning Corp. Holiday Inn, Fort Collins, CO. (Ward T. Collins, Mail Stop C41C00, Dow Corning Corp., Midland, MI 48686-0994).

(June 17-20)—Oil and Colour Chemists' Association Biennial Conference. Eastbourne, England. (Mr. R.H. Hamblin, Director & Secretary, OCCA, Priory House, 967 Harrow Rd., Wembley, Middlesex, England HA0 2SF).

(Sept. 15-18)—XVIIth Congress of AFTPV (French Association of Paint and Varnish Technicians) and Eurocoat. Nice, France. (J. Roire, 5, Rue Etex, 75018 Paris, France).

(Nov. 7-11)—10th International Congress on Metallic Corrosion sponsored by Central Electrochemical Research Institute on behalf of International Corrosion Council. Madras, India. (Dr. V.I. Vasu, Chairman, ICMC Organizing Committee, Director CERI, Karaikudi 623006, Tamil Nadu, India).

1987

(Mar. 17-19)—Powder Coatings '87. G-MEX Exhibition Center, Manchester, England. (Mervyn W.K. Little, Specialist Exhibitions Ltd., Grantleigh House, 14-32 High St., Croydon, CRO 1YA Surrey, England).

1988

(Oct. 18-21)—12th World Congress on Metal Finishing, INTERFINISH 88. Palais des Congres, Paris, France. (SEPIC INTERFINISH), 17 rue d'Uzes, 75002 Paris, France).

## Advertisers Index

ALLIED CORP., A-C POLYETHYLENE .....	96
ARCO CHEMICAL CO. ....	31
CABOT CORP., SPECIAL BLACKS DIV. ....	90
CUNO INDUSTRIAL PRODUCTS .....	132
DISTI, INC. ....	121
E.J. GAISSER, INC. ....	126
GEORGIA MARBLE CO. ....	22
HEUBACH INC. ....	17
J.M. HUBER CORP. ....	125
INOLEX CHEMICAL CO. ....	Cover 3
S.C. JOHNSON & SON .....	52
KING INDUSTRIES, INC. ....	134

MOBAY CHEMICAL CO. ....	9-11, 13
MONSANTO CHEMICAL CO. ....	4-5
NL CHEMICALS/NL INDUSTRIES, INC. ....	15
PFIZER, INC., MPM DIV. ....	70
POLYVINYL CHEMICALS INC. ....	21
ROHM AND HAAS CO. ....	8, 19
SHAMROCK CHEMICALS CORP. ....	Cover 4
SYLVACHEM CORP. ....	51
UNION CARBIDE CORP. ....	Cover 2-1, 123
UNOCAL CORP., UNOCAL CHEMICALS DIV. ....	89
R.T. VANDERBILT CO. ....	2

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## 'Humbug' from Hillman

Just when everything seemed to be going right and the promise of spring lightened one's spirits, along came a package from Dr. Tom Miranda containing a load of refuse—otherwise known as "Robert Ahlf's Words of Doubtful Wisdom." Among other things, "Humbug from Hillman" is dedicated to harassing the JCT readers who are incautious enough to reach this last page. Bob Ahlf has been most helpful in this project. This month's harassments are:

- Sitting on your hands may not be the best way to cover your butt.
- If you and I stick together, we'll never be alone.
- Self pity is better than none.
- Life teaches you many things, some of which you didn't want to learn.
- In the winter, when you need daylight, the government takes away daylight savings time.
- Lots of businesses are like show business.
- Don't trust people who always lie to you.
- Don't worry about your job while you're gone, we'll have a girl from the cafeteria do it during her lunch hour.
- There ought to be a law against crime.
- You look good, but I wouldn't invest much in lifetime guarantees.

Not to be outdone (obviously, no great difficulty), Henry Pritula contributed a line of frozen refuse from his perch in Canada. We'll annoy you with some of his collection from time to time. This month, Henry adds to the list of "How You Can Tell It's Going To Be A Rotten Day" with:

- You wake up face down on the pavement.
- You put on your bra backwards and it fits better.
- You want to put on the clothes you wore home from the party and there aren't any.
- You wake up and discover your waterbed broke and then realize you don't have a waterbed.
- Your income tax check bounces.
- You put both contact lenses in the same eye.
- You walk to work and find your dress is stuck in the back of your panty hose.
- You're past fifty and the rabbit dies.

Henry also found the Murphy of Murphy's Law was an optimist when he philosophized that "Anything that can go wrong, will." Henry added the following observations without identifying from whence they came. We apologize for that and for repeating them.

- No good deal goes unpunished.
- Leakproof seals, will.
- Self starters, will not.
- All warranties expire on the date of invoice.
- Interchangeable parts, won't.
- The repairman will never have seen a model like yours before.

- Never play leapfrog with a unicorn.
- If you try to please everybody, nobody will like it.

Since things are going so badly for you this month, you might as well suffer through this gem contributed by Frank Borrelle from "Association Trends."

A visitor to one of the Aleutian Islands off Alaska broke his glasses. He was told by his guide that he couldn't get the glasses repaired until he returned to Anchorage.

"You mean there aren't any optometrists on the island?" said the man.

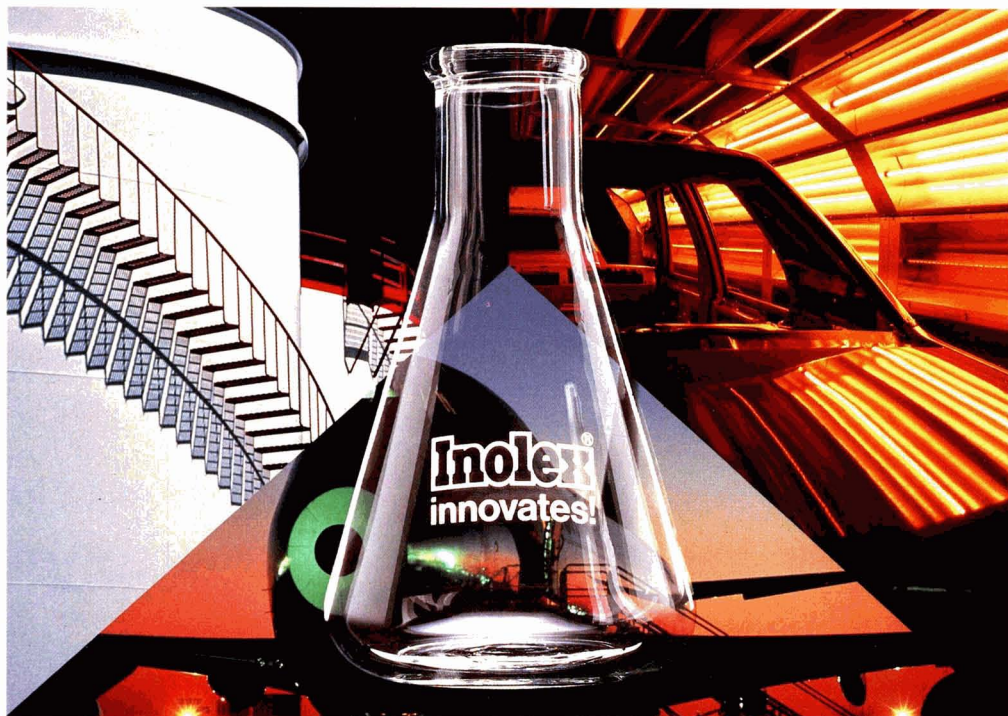
"If you see one," replied the guide, "it will be an optical Aleutian!"

Lou Eromenok, a devoted and active New York Society member of many years has, nonetheless, been foolish enough to be devoted and dedicated to reading "Humbug." Now, Lou has compounded this folly by sending me some choice nonsense from the Flemington, NJ "Democrat" (Reagan supporters, please copy), as prepared by Dereck Williamson.

- Tim's truth—"If you want to catch fish, you gotta fish."
- Nobody is ever ready in ten minutes.
- If a public building has two glass doors, one will be locked.
- You always push the locked door first.
- Shelley's warning—"Never participate in a sport that has an ambulance standing by."
- If you tell a plumber there's no hurry, you'll get no plumber.
- The tool to unjamb the kitchen drawer is in the drawer.
- If you take just one picture, something will be wrong with it. If you take several, they'll all come out.
- A wasp will fly into a car, but not out.
- You need glasses to find your glasses.
- In every group picture, somebody will have closed eyes.

Warning! Coming attraction: Excerpts from "How to Paint" by F.S. Gardner, 1875 . . . as dug up by Sal Sanfilippo.

—Herb Hillman  
Humbug's Nest  
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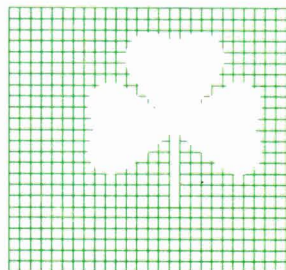
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